

NON-PROVISIONAL UTILITY PATENT APPLICATION

5

For

TOBACCO FILLER OF LOW NITROGEN CONTENT

by

10 JOHN-PAUL MUA, a United States Citizen, residing at 234 Chadwick Circle, Macon, GA
31210

Attorney Docket No. R-03-13

15

CROSS-REFERENCE TO RELATED APPLICATIONS

This international patent application is a Paris Convention filing of and claims priority
to and benefit from, currently pending, U.S. Patent Application serial number 10/855,925,
filed on 27 May 2004.

20

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR
DEVELOPMENT

Not applicable.
FIELD OF INVENTION

25

This invention relates generally to tobacco and reconstituted tobacco smoking
materials and methods of making same. More particularly, the present invention relates to
the materials and methods that provide smoking materials with low nitrogen content.

BACKGROUND

Tobacco material contains various nitrogenous compounds that can adversely affect
30 its smoke quality. Among these nitrogenous compounds are proteins, amino acids,
heterocyclic aromatic amines, tobacco specific nitrosamines (TSNA), as well as other
compounds formed by pyrolysis or transfer of these nitrogenous compounds. It has been

determined that tobacco quality is improved by reducing its nitrogen content. It has been found to be relatively easy to remove protein from uncured tobacco leaf, however there are disadvantages to removing protein before curing. The protein broken down during curing form flavor compounds that are important contributors to the organoleptic properties of the smoke. Another disadvantage is that efficient extraction of green leaf usually necessitates tobacco structural changes which make it difficult to produce shredded tobacco suitable for use as a cigarette filler. Tobacco processing sometimes includes steps in which the nitrogen content of the tobacco is reduced, so as to improve the smokability of the tobacco. However, nitrogenous compounds, especially proteins, are difficult to extract from cured tobacco lamina, stem, and fiber cell walls.

Plant proteins are divided into four major classes: albumins; globulins; prolamins (also known as gliadins); and, glutelins. Albumins are soluble in water, whereas globulins are soluble in dilute salt solutions. Prolamins are soluble in dilute acid or alkali solutions, while glutelins are soluble in alcohol solutions. Some proteins, however, overlap into two of these four classifications, thereby increasing the difficulty of accurately predicting the appropriate diluent. Insoluble proteins make up 85% to 95% of the total proteins found in cured tobacco. These insoluble proteins are globular in conformation, and are bound to lipoidal organocellular membranes of fiber or cellulose cell walls. Solubilization and extraction of these insoluble proteins have proven difficult when using water or solvents under moderate digestion conditions (i.e. less than 100° C at 65-70 psig) and with shredded tobacco of size suitable for cigarette manufacture. Partial removal of protein from cured tobacco can be accomplished by extraction with water, with the efficiency of the extraction improving as the particle size is reduced. However, for shredded tobacco of the size normally used for cigarette manufacture, most of the protein cannot be extracted by water

alone. This difficulty is due in large part to the compact and rigid structure of fiber cells. Penetration of rigid cell wall structure by solvents has proven feasible only after thermal and/or mechanical sample treatment. However, the common result of such sample treatment is poor recovery of the solvated particulate material. Moreover, cell wall penetration does not
5 necessarily lead to protein solubilization, since plant proteins differ in their conformity and solubilization patterns.

Many of the current processes used to reduce nitrogen content in tobacco material employ enzymatic compounds and microbial agents to break down the proteins and other nitrogen-containing compounds within the tobacco. Several inventors have found that
10 proteolytic enzymes will break down tobacco protein into readily soluble fragments and that strip or cut tobacco can be treated by such enzymes. Gaisch et al. (U.S. Pat. No. 4,407,307) described the removal of protein from tobacco strips in an aqueous solution of a proteolytic enzyme whereby insoluble proteins are decomposed into soluble fragments. The extract is separated from the tobacco and inoculated with a yeast culture, which, as it grows,
15 removes the soluble protein fragments in the extract by metabolic assimilation. After removal of the yeast, the protein-free extract is concentrated and added back to the tobacco strips. Bernasek et al. (U.S. Pat. No. 4,887,618) describes a process in which tobacco is first extracted with water. The tobacco residue remaining after extraction is separated from the solution, mixed with water and treated with a proteolytic enzyme. The protein-reduced
20 tobacco is separated from the enzyme solution, rinsed and dried. The water extract is concentrated and added back to the protein reduced tobacco. The advantage described by Bernasek et al. for this process is that the water soluble flavor components of tobacco and the nicotine can be retained in the final product.

DeGrandpre et al. (U.S. Pat. No. 5,311,886) teaches a process where cut tobacco is extracted in an aqueous solution having a surfactant and proteolytic enzyme. The proteolytic enzyme, if used, is chosen from the group comprising the bacterial and fungal enzymes. The enzymes used commercially in the food and detergent industries (ie. Savinase*, Neutrase*, 5 Enzobake* or Alcalase*) available from Novo Inc. were found to have been effective for protein removal from tobacco. These enzymes were added to the solution in the concentration range 0.1% to 5% w/w of the tobacco material.

*Trade-mark

10 The above described processes primarily rely on enzymes to remove protein from tobacco material. Disadvantages arise from the use of such enzymatic compounds and agents. In particular, enzymes are expensive, pH sensitive, and degrade proteins into amino acids which tend to remain with the tobacco material. It is also thought that enzymatic compounds leave residues on tobacco material after processing. Additionally, special 15 handling requirements and the additional process step of inactivating the enzymes (i.e. steam, autoclave, salt wash) cause the process to incur additional expenses. Furthermore, microbial agents used in treating tobacco tend to cause unwanted reactions that generate undesirable by-products.

Another current processes used to reduce nitrogen content in tobacco material 20 employs the use of an alkali or caustic solution. Poulose et al. (U.S. Pat. No. 4,716,911) also realized the disadvantage using protease enzymes and proposed using either an alkali or a combination of a protease and a non-protease depolymerase to effect protein removal in an overall processing scheme similar to that of Gaisch et al. However, it was found that alkaline solutions at the strengths quoted by Poulose et al. may have a deleterious effect on the

physical structure of the tobacco. Moreover, the use of a protease combined with a depolymerase may not be an economical approach to protein removal.

Mua, et al. (U.S. Pat. No. 6,508,254) relates to a method for providing a reconstituted tobacco material having a reduced nitrogenous content without having the
5 problems associated with the use of enzymes. In Mua, et al. the tobacco material in the form of flue cured and burley whole leaf, stems, fines, lamina or scraps, and/or burley stems was first contacted with an aqueous solvent. The resulting liquid extract was then separated from the tobacco fiber portion. The tobacco fiber portion was then contacted with a solution containing sodium acetate and/or sodium hydroxide and/or potassium hydroxide. This
10 solution was also separated from the tobacco fiber portion. The tobacco fiber portion may then have been washed, refined and processed into reconstituted tobacco sheets. The liquid extract from the aqueous solvent extraction may have been concentrated and added back to the sheets.

There is a need to provide an economically competitive process by which the nitrogen
15 content of smoking material may be reduced without leaving residues or undesirable by-products and remains in a form that can be processed into paper or bandcast. This process must provide for an efficient and effective reduction of proteins and other nitrogenous compounds. It is desirable to provide a technique for protein reduction in smoking materials which does not cause a physical degradation of the tobacco structure and is economical and
20 efficient.

BRIEF SUMMARY OF THE INVENTION

The present invention relates to a method for providing a reconstituted tobacco containing material having a reduced nitrogenous content. The process starts with flue cured and / or burley tobacco in the form of whole leaf, stems, fines, lamina or scraps. The tobacco

material is first contacted with an aqueous solvent. The resulting liquid extract is separated from the tobacco fiber portion. The tobacco fiber portion is then processed according to the present invention. The weak extract liquid (WEL) may be discarded, processed and/or concentrated and added back to the processed fiber portion.

5 The tobacco fiber portion is then refined and optionally digested with an alkali solution. The resulting tobacco material is then mixed with cellulose fibers and optionally inert materials. The inert materials may be inorganic or organic. The inorganic inert material that may be mixed with the tobacco fibers and cellulose includes CaCO_3 , MgO , MgCO_3 and combinations thereof. The organic inert materials that may be mixed with the tobacco fibers
10 and cellulose include chitosan, liposan and combinations thereof. Additionally, both the organic and inorganic inert materials may be mixed together with the tobacco and cellulose material. These combined materials may then be cast into sheet via a papermaking process or a binder and a humectant may be added and the material cast into sheet via bandcast sheet processing. The inert materials may include up to 20% of the finished sheet produced by the
15 present invention. The tobacco fiber portion may make up to 50% of the finished sheet produced by the present invention. Advantageously, the finished sheet includes between approximately 10% to 50% tobacco.

 The liquid extract or WEL from the aqueous solvent extraction may be discarded or added back to the fiber portion. If the WEL is to be added back to the fiber portion, it may be
20 processed and/or concentrated. If the smoking material is to be cast into sheet by a paper making process, then the WEL is added back directly to the formed sheet. If the smoking material is to be cast into sheet by a bandcast process, then the WEL is added back just prior to the bandcast sheet processing. These finished sheets may then be used in smoking articles, such as cigarettes. The reduction of nitrogenous compounds in the smoking material

provides for improved smokability and a reduction in nitrogen containing pyrolytic products emitted from smoking articles which contain the tobacco material.

It is an object of the present invention to provide a reconstituted tobacco material with reduced levels of nitrogenous compounds.

5 It is another object of the present invention to provide a method of making a reconstituted tobacco material with reduced levels of nitrogenous compounds.

It is a further object of the present invention to provide a reconstituted tobacco material paper with a cured tobacco leaf-like texture.

More particularly, the present invention is directed to a method for reducing the
10 nitrogenous content of a smoking material. A better understanding of the present invention will be realized from the hereafter processes and the Examples following such description.

DESCRIPTION OF DRAWINGS

FIG. 1 is a schematic of the process steps representative of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

15 In a preferred method of carrying out the nitrogen reduction process of the present invention, tobacco materials 10 in the form of flue cured and/or burley stems, scraps, fines, and cut lamina are contacted at process station 11 with an aqueous solvent, such as water, at a temperature of about 60° C to 90° C for about 0.25 to 1 hour. The contacting of the tobacco with an aqueous solvent, process station 11, may be conducted in a tank or similar mixing
20 vessel in which the solvent and tobacco are heated and may be agitated or stirred. The resulting aqueous tobacco extract or weak extract liquid (WEL), containing flavor compounds, is separated from the tobacco fiber portion at process station 12. The tobacco/solvent mixture formed at process station 11 is then pumped into a centrifuge from the mixing vessel and centrifugally separated or alternatively the mixture may be filtered to

remove the tobacco fiber portion 17 from the WEL 13 at process station 12. The WEL 13 may be reserved for reapplication to the fiber portion 17 with or without separate processing 14. In one embodiment, the WEL 13 may be deproteinated by contacting it with a solid phase adsorbent at process station 14. A solid phase adsorbent, such as such as a bentonite and carbon containing adsorbent, is mixed with the WEL 13 in a vessel and then separated 5 therefrom by centrifugation, or a similar separation process well known in the art. The WEL 13, whether treated at process station 14 or untreated, containing flavor compounds may then be concentrated at process station 16 by vacuum evaporation.

The tobacco fiber portion 17 may undergo several alternative processes. The tobacco 10 fiber portion 17 may be refined and digested at process station 18. The refining process at process station 18 consists of fiberizing or breaking down the tobacco into smaller fibers. The digestion process at process station 18 consists of adding one part of the refined tobacco to about eight parts of an alkali solution having about 1.25 % to 12 % NaOH or KOH. The tobacco is digested in the alkali solution at about 170° F to 190° F for up to 3 hours.

15 Alternatively, digestion may be accomplished by autoclaving the tobacco at about 120° C for about 15 to 30 minutes at about 17 to 27 psig. The resulting tobacco material is then mixed with cellulose fibers and optionally inert materials at process station 19. Tobacco may make up to 50% of the sheet formed at process station 21. Advantageously, tobacco will make up between about 10% to 50% of the sheet formed at process station 21. The optional inert 20 materials at process station 19 may be inorganic or organic. The inorganic inert materials that may be mixed with the tobacco fibers and cellulose at process station 19 include CaCO_3 , MgO , MgCO_3 and combinations thereof. The organic inert materials that may be mixed with the tobacco fibers and cellulose at process station 19 include chitosan, liposan and combinations thereof. Additionally, both the organic and inorganic inert materials may be

mixed together with the tobacco and cellulose material at process station 19. The inert materials added at process station 19 may include up to 20% of the sheet formed at process station 21. These combined materials may then be mixed with the WEL 13, whether treated 14, untreated 13 and/or concentrated 16. At process station 20 a binder and humectant is
5 added to the mixture. The mixture resulting from process station 20 is then cast into sheets via a bandcast process at process station 21. The sheets resulting from process station 21 is then cut and added to tobacco producing a tobacco blend at process station 34.

Alternatively, the tobacco fiber portion may be refined at process station 22 and mixed with cellulose fibers and optionally inert materials at process station 23. The optional
10 inert materials at process station 23 may be inorganic or organic. The inorganic inert materials that may be mixed with the tobacco fibers and cellulose at process station 23 include CaCO_3 , MgO , MgCO_3 and combinations thereof. The organic inert materials that may be mixed with the tobacco fibers and cellulose at process station 23 include chitosan, liposan, and combinations thereof. Additionally, both the organic and inorganic inert
15 materials may be mixed together with the tobacco and cellulose material at process station 23. The inert materials may include up to 20% of the bandcast sheet formed at process station 25. The tobacco fiber portion may make up to 50% of the bandcast sheet formed at process station 25. These combined materials may optionally be mixed with the WEL 13, whether treated 14, untreated 13 and/or concentrated 16. At process station 24 a binder and
20 humectant is added to the mixture. The mixture resulting from process station 24 is then cast into sheets via a bandcast process at process station 25. The sheets resulting from process station 25 are then cut and added to tobacco producing a tobacco blend at process station 34.

In addition to the embodiments incorporating a bandcast step in the present invention the process of the present invention may produce sheet via a paper making process. Two

embodiments of the present invention are shown in Figure 1 to incorporate a paper making process station.

In one embodiment the tobacco fiber portion 17 is refined and digested at process station 26. The refined and digested tobacco fiber is then mixed with cellulose fibers and optionally inert materials at process station 27. The optional inert materials at process station 27 may be inorganic or organic. The inorganic inert materials that may be mixed with the tobacco fibers and cellulose at process station 27 include CaCO_3 , MgO , MgCO_3 and combinations thereof. The organic inert materials that may be mixed with the tobacco fibers and cellulose at process station 27 include chitosan, liposan, and combinations thereof.

Additionally, both the organic and inorganic inert materials may be mixed together with the tobacco and cellulose material at process station 27. The inert materials may include up to 20% of the sheet formed at process station 28. The tobacco fiber portion may make up to 50% of the sheet formed at process station 28. These combined materials resulting from process station 27 are then made into sheets via a paper making process at process station 28.

The paper sheet resulting from process station 28 may then have the WEL 13, whether treated 14, untreated 13 and/or concentrated 16 added back at process station 29. The paper sheets resulting from process station 29 are then cut and added to tobacco producing a tobacco blend at process station 34.

In another embodiment incorporating a paper making process the tobacco fiber portion 17 is first refined at process station 30. The refined tobacco fiber is then mixed with cellulose fibers and optionally inert materials at process station 31. The optional inert materials at process station 31 may be inorganic or organic. The inorganic inert materials that may be mixed with the tobacco fibers and cellulose at process station 31 include CaCO_3 , MgO , MgCO_3 and combinations thereof. The organic inert materials that may be mixed

with the tobacco fibers and cellulose at process station 31 include chitosan, liposan, and combinations thereof. Additionally, both the organic and inorganic inert materials may be mixed together with the tobacco and cellulose material at process station 31. The inert materials may include up to 20% of the sheet formed at process station 32. The tobacco fiber portion may make up to 50% of the sheet formed at process station 32. These combined materials resulting from process station 31 is then made into sheets via a paper making process at process station 32. The paper sheet resulting from process station 32 may then have the WEL 13, whether treated 14, untreated 13 and/or concentrated 16 added back at process station 33. The paper sheets resulting from process station 33 is then cut and added to tobacco producing a tobacco blend at process station 34.

The following Examples are incorporated herein to illustrate the present invention with no intention of being unduly limited thereby.

EXAMPLES 1-4

Example 1:

Burley tobacco in the form of whole leaf, stems, fines, lamina and scraps was first extracted with water. This was accomplished by placing approximately 1 part of tobacco material into a vessel having approximately 11 parts of water. The tobacco and water were contacted at about 160° F for about 30 minutes. The tobacco and water was periodically mixed during this extraction. The tobacco and extract were then separated by basket centrifuge forming a WEL and an extracted tobacco fiber. The WEL was discarded, while the fiber was refined or fiberized.

A portion of the extracted burley tobacco fiber was formed into sheet with a MKS sheet former and analyzed to provide control data. Another portion of the fiber was treated with Savinase* in accordance with the process in DeGrandpre et al. (U.S. Pat. No.

5,311,886). This was accomplished by placing approximately 3.8 to 4.8 kg of extracted tobacco fiber into solution having approximately 200 ml. Savinase*, 40 g NaOH and 114 L water. The tobacco fiber was digested in the solution at about 57° C for about 30 minutes while the solution was periodically stirred. The Savinase* solution was then separated from the tobacco fiber by basket centrifuge and discarded. The Savinase* treated burley tobacco fiber was rinsed three times with a salt solution having 1.84 kg NaCl in 80 L water. The rinsed burley tobacco fiber was autoclaved (heated to 121°C and held for 15 min, at 17 psig) to inactivate any remaining enzymes and formed into sheet with a MKS sheet former and analyzed for Hoffmann analytes.

10 A separate portion of the burley tobacco fiber was treated with alkali in accordance with Mua et al. This was accomplished by adding 1 part of tobacco fiber having about 60% moisture to about 8 parts of an alkali solution having about 2.5 % NaOH. The tobacco was digested in the alkali solution at about 190° F for about 2 hours. The tobacco fiber was then separated by basket centrifuge and the alkali solution was discarded. Water was then added to the digested tobacco fiber after which it was refined and formed into sheet with a MKS sheet former and analyzed for Hoffmann analytes.

20 Yet another portion of the burley tobacco fiber was processed by the method claimed in the present invention. This was accomplished by adding cellulose pulp and CaO₃ filler to the extracted tobacco fiber forming a tobacco containing material having approximately 25% tobacco. This tobacco containing material was then formed into sheets with a MKS sheet former and the resulting sheets were analyzed for Hoffmann analytes. The analytical results of this example are tabulated below.

**Protein (nitrogen) and Other Hoffmann Analyte Precursor Removal / Reduction from
Burley Base Sheet**

Reconstitution process	Total nitrogen (% dwb)	Protein (% dwb)	Total TSNAs (ppm)	Polyphenols (% dwb)			Alkaloids (% dwb)	Nitrate (% dwb)
				Chlorogenic acid	Rutin	Scopoletin		
Washed burley fiber- Control	3.57	20.44	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Burley fiber- Savinase* treated	1.63	9.38	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Burley fiber- 2.5% alkali treated	2.49	14.38	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Burley- with 25% tobacco fiber + cellulose and inert filler	0.97	1.33	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

5

The present invention was shown to substantially reduce the total nitrogen and protein content in the smoking material. Neither the Savinase* nor the alkali treated tobacco lowered the nitrogen and protein content of the smoking material near the 73% and 93% reductions as obtained in this example.

10 **Example 2:**

A mixture of tobacco having flue and burley tobacco in a ratio of about one to one was processed in accordance with the procedures in Example 1 to obtain a washed flue / burley control, Savinase* treated, and alkali treated tobacco sheets. The flue/burley tobacco was processed in accordance with the present invention where a finished base sheet was

15 produced having 15% tobacco and separately 50% tobacco. These base sheets were analyzed for Hoffmann analytes and the results are as shown below.

**Protein (nitrogen) and other Hoffmann analyte precursor removal/reduction
from Flue/Burley Base Sheet**

Reconstitution process	Total nitrogen (% dwb)	Protein (% dwb)	Total TSNAs (ppm)	Polyphenols (% dwb)			Alkaloids (% dwb)	Nitrate (% dwb)
				Chlorogenic acid	Rutin	Scopoletin		
Washed flue/burley- Control	1.48	8.50	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Flue/burley – Savinase* treated	0.56	3.38	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Flue/burley- alkali treated	0.76	4.06	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Flue/burley- with 15% tobacco fiber + wood pulp + inert filler	0.21	1.06	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Flue/burley- with 50% tobacco fiber + wood pulp + inert filler	0.75	4.56	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

5

The present invention was again shown to substantially reduce the total nitrogen and protein content in the smoking material. Neither the Savinase* nor the alkali treated tobacco lowered the nitrogen and protein content of the smoking material near the 86% and 88% reductions as obtained in this example having 15% tobacco fiber.

10 Example 3:

Burley tobacco was processed in accordance with the procedures in Example 1 to obtain a washed burley control, Savinase* treated, and alkali treated tobacco sheets.

However, in this example the WEL was treated with an adsorbent having bentonite and carbon, to remove nitrogenous compounds, and the treated WEL was added back to the

15 tobacco sheets. The burley tobacco containing sheets were processed in accordance with the present invention where a finished sheet was produced having 50% tobacco (15% tobacco

fiber + 35% concentrated extract). These finished sheets were analyzed for Hoffmann analytes and the results are as shown below.

5 Protein (nitrogen) and other Hoffmann analyte precursor removal/reduction from Burley Finish Sheet

Reconstitution process	Total nitrogen (% dwb)	Protein (% dwb)	Total TSNAs (ppm)	Polyphenols (% dwb)			Alkaloids (% dwb)	Nitrate (% dwb)
				Chlorogenic acid	Rutin	Scopoletin		
Washed burley-Control + regular burley extract	4.27	13.94	7.01	0.18	>0.5	0.02	1.13	1.41
Burley fiber-Savinase* treated + burley treated extract	2.03	6.38	1.55	<0.1	<0.1	<0.1	0.88	1.23
Burley fiber-2.5% alkali treated + burley treated extract	3.19	7.38	1.64	<0.1	<0.1	<0.1	0.90	1.43
Burley- with 20% tobacco fiber + burley treated extract	1.23	1.75	1.65	<0.1	<0.1	<0.1	0.89	1.41

The present invention was again shown to substantially reduce the total nitrogen and protein content in the smoking material. Neither the Savinase* nor the alkali treated tobacco

10 lowered the nitrogen and protein content of the smoking material near the 71% and 87% reductions as obtained in this example having 50% tobacco.

Example 4:

A mixture of tobacco having flue and burley tobacco in a ratio of about one to one

15 was processed in accordance with the procedures in Example 3 to obtain a washed burley control, Savinase* treated, and alkali treated tobacco sheets. The flue/burley tobacco containing sheets were processed in accordance with the present invention where a finished

sheet was produced having 65% and 45% tobacco. These finished sheets were analyzed for Hoffmann analytes and the results are as shown below.

5 Protein (nitrogen) and other Hoffmann analyte precursor removal/reduction from Flue/Burley Finish Sheet

Reconstitution process	Total nitrogen (% dwb)	Protein (% dwb)	Total TSNAs (ppm)	Polyphenols (% dwb)			Alkaloids (% dwb)	Nitrate (% dwb)
				Chlorogenic acid	Rutin	Scopoletin		
Flue/burley-Control + regular burley extract	4.66	5.56	17.80	0.03	0.02	0.01	3.4	2.10
Flue/burley-Savinase* + treated burley extract	2.29	2.38	3.24	0.00	0.00	0.00	1.81	1.95
Flue/burley -alkali + treated burley extract	3.01	2.56	3.41	0.00	0.00	0.00	1.58	1.53
Flue/Burley-with 50% tobacco fiber + burley treated extract	3.22	3.00	2.98	0.00	0.00	0.00	1.76	1.63
Flue/Burley-with 15% tobacco fiber + burley treated extract	1.09	1.38	3.04	0.00	0.00	0.00	1.79	1.53

The process of the present invention substantially reduced the total nitrogen and protein content in the smoking material. Neither the Savinase* nor the alkali treated tobacco lowered the nitrogen and protein content of the smoking material near the 77% and 75% reductions as obtained in this example having 45% tobacco.

The aforementioned examples show that the present invention substantially reduces total nitrogen and protein content of smoking materials. Additionally, the problems associated with the prior art of having enzyme residues in the smoking material and having problems in the paper making process have been resolved in the present invention.

We Claim:

1. A method of making a smoking material with reduced levels of nitrogenous compounds comprising:
 - (a) contacting a tobacco material with an aqueous solvent to provide an aqueous tobacco
5 extract and a tobacco fiber portion;
 - (b) separating said aqueous tobacco extract from said tobacco fiber portion; and
 - (c) mixing said tobacco fiber portion with a cellulose containing material.
2. The method of Claim 1, further comprising:
 - (d) mixing said tobacco fiber portion and said cellulose containing material with an inert
10 material.
3. The method of Claim 2 wherein said inert material is selected from the group consisting of CaCO_3 , MgO , MgCO_3 , chitosan, liposan, and combinations thereof.
4. The method of Claim 2 wherein said inert material comprises up to about 20% of said smoking material.
- 15 5. The method of Claim 1 wherein said smoking material has up to 70% tobacco.
6. The method of Claim 1 wherein said cellulose containing material is selected from the group consisting of wood pulp, bleached wood pulp, flax, any plant cellulosic material, and combinations thereof.
7. The method of Claim 1, wherein said tobacco material is contacted with said aqueous
20 solvent at a concentration of approximately 1 part of said tobacco material for every 11 to 15 parts of said aqueous solvent.
8. The method of Claim 1 wherein said separation of said aqueous tobacco extract from said tobacco fiber portion is accomplished by centrifugation.
9. The method of Claim 1 wherein said separation of said aqueous tobacco extract from said
25 tobacco fiber portion is accomplished by filtration.

10. The method of Claim 1, wherein said tobacco material is contacted with said aqueous solvent at a temperature range of about 60° C to 90° C for about 0.25 to 1 hour.
11. The method of Claim 1 wherein said tobacco material includes flue cured tobacco.
12. The method of Claim 1 wherein said tobacco material includes burley tobacco.
- 5 13. The method of Claim 1 wherein said tobacco material includes burley tobacco and flue cured tobacco in a ratio of about 1 to 1.
14. The method of Claim 1, further comprising:
- (e) processing said tobacco fiber portion and said cellulose containing material into a reconstituted tobacco sheet.
- 10 15. A method of making a smoking material with reduced levels of nitrogenous compounds comprising:
- (a) contacting a tobacco material with an aqueous solvent to provide an aqueous tobacco extract and a tobacco fiber portion;
- (b) separating said aqueous tobacco extract from said tobacco fiber portion;
- 15 (c) refining said tobacco fiber portion forming a refined tobacco material; and
- (d) mixing said refined tobacco material with a cellulose containing material.
16. The method of Claim 15 wherein said refining of said tobacco fiber portion is followed by a step of digesting said refined tobacco material.
17. The method of Claim 16, wherein said digesting of said refined tobacco material
- 20 comprises contacting said refined tobacco material with a solution containing about 1.25% to 12.0% sodium hydroxide.
18. The method of Claim 17, wherein said refined tobacco material and said sodium hydroxide solution are contacted at a ratio of about 1 part of refined tobacco material for each part of sodium hydroxide solution.

19. The method of Claim 18, wherein said digesting of said refined tobacco material is at a temperature of from about 170° F to about 190° F for about 30 min to 4 hours.
20. The method of Claim 16, wherein said digesting of said refined tobacco material comprises contacting said refined tobacco material with a solution containing about 1.25% to
5 12.0% potassium hydroxide.
21. The method of Claim 20, wherein said refined tobacco material and said potassium hydroxide solution are contacted at a ratio of about 1 part of refined tobacco material for each part of potassium hydroxide solution.
22. The method of Claim 21, wherein said digesting of said refined tobacco fiber portion is
10 at a temperature of from about 170° F to about 190° F for about 30 min to 4 hours.
23. The method of Claim 16, wherein said digesting of said refined tobacco fiber portion comprises autoclaving said refined tobacco fiber portion at a temperature of about 120° C and at a pressure of about 17 to 27 psig for about 15 to 30 minutes.
24. The method of Claim 15, further comprising:
- 15 (e) mixing said refined tobacco material and said cellulose containing material with an inert material.
25. The method of Claim 24 wherein said inert material is selected from the group consisting of CaCO₃, MgO, MgCO₃, chitosan, liposan, and combinations thereof.
26. The method of Claim 24 wherein said inert material comprises up to about 20% of said
20 smoking material.
27. The method of Claim 15 wherein said smoking material has up to 70% tobacco.
28. The method of Claim 15 wherein said cellulose containing material is selected from the group consisting of wood pulp, bleached wood pulp, flax, any plant cellulosic material, and combinations thereof.

29. The method of Claim 15, further comprising:

(e) processing said refined tobacco material and said cellulose containing material into a reconstituted tobacco sheet.

30. The method of Claim 15, further comprising:

5 (e) contacting said refined tobacco material and said cellulose containing material after said mixing with a selected portion of said tobacco extract.

31. A method of making a tobacco containing material with reduced levels of nitrogenous compounds comprising:

(a) contacting a tobacco material with a first aqueous solvent to provide an aqueous tobacco
10 extract and a tobacco fiber portion;

(b) separating said aqueous tobacco extract from said tobacco fiber portion;

(c) contacting at a temperature from about 170° F to 190° F said tobacco fiber portion with a solution containing a compound selected from the group consisting of sodium hydroxide and potassium hydroxide, wherein said solution contains said compound in a concentration about
15 1.25% to 12% (w/v) of said solution;

(d) separating said solution from said tobacco fiber portion; and

(e) mixing said tobacco fiber portion with cellulose fibers.

32. The method of Claim 31, further comprising:

(f) contacting said tobacco fiber portion with a portion of said aqueous tobacco extract.

20 33. The method of Claim 31, further comprising:

(f) treating a portion of said aqueous tobacco extract with an adsorbent; and

(g) contacting said tobacco fiber portion with a portion of said treated aqueous tobacco extract.

34. The method of Claim 31, further comprising:

(f) mixing said tobacco fiber portion and said cellulose fibers with an inert material.

35. The method of Claim 34 wherein said inert material is selected from the group consisting of CaCO_3 , MgO , MgCO_3 , chitosan, liposan, and combinations thereof.

5 36. The method of Claim 34 wherein said inert material comprises up to about 20% of said tobacco containing material.

37. The method of Claim 31 wherein said tobacco containing material has up to 70% tobacco.

38. The method of Claim 31 further comprising:

10 (f) mixing said tobacco fiber portion and said cellulose fibers with a binder;

(g) mixing said tobacco fiber portion, cellulose fibers, and binder with a humectant; and

(h) making sheets via a bandcast process.

39. The method of Claim 31 further comprising:

(f) making sheets via a paper making process.