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ETHER DERIVATIVES OF TOBACCO AND TOBACCO PRODUCTS

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This invention relates to new compositions of matter and tobacco smoking products. More particularly this invention relates to ether derivatives of tobacco and mixtures of these derivatives and straight tobacco as sheet forming material useful in the preparation of smoking products.

One of the constituent parts of most cigars marketed today is a binder sheet, composed of comminuted tobacco, a cellulose derivative as a binding agent and a plasticizer. This type of synthetic sheet is adapted to the machine processing of cigars and is designed to resemble tobacco in smoking quality. The cellulose derivative component of the binder sheet has certain objectionable features; for example, since the material is not tobacco, it modifies the smoking taste of the mixture and it is a hotter burning material than the tobacco component.

It is an object of the present invention to provide ether derivatives of tobacco which are useful for the preparation of smoking products having improved smoking properties.

It is another object of this invention to provide a manufactured leaf or sheet of tobacco containing an ether derivative of tobacco.

It is a further object of this invention to provide reconstituted tobacco containing an ether derivative of tobacco as a binding agent of improved strength.

It is still another object of this invention to provide reconstituted tobacco sheets having excellent strength and machineability and which are economically manufactured.

It is still another object of this invention to provide a cigar having a wrapper or binder sheet of reconstituted tobacco containing an ether derivative of tobacco.

These and other objects are accomplished in accordance with this invention which broadly stated is a new composition of matter comprising an ether derivative of tobacco. In addition, the invention includes a mixture of tobacco and an ether derivative of tobacco. Both the derivative and mixture are readily formed into reconstituted tobacco sheets, for example, by mixing water slurries thereof, spreading and drying. These sheets are then preferably used as wrappers or binder sheets for cigars or cigar-like smoking products. They are also shredded and used as fillers for smoking products. Sheets of reconstituted tobacco, in accordance with this invention, are formed from any proportion of comminuted tobacco of any variety or origin, at least 5% and up to 100% by weight of an ether derivative of tobacco, and usually up to 20% by weight of a plasticizer, for example, glycerine. For additional strength cross-linking agents can be incorporated in the tobacco composition.

Additives, to improve smoking or burning properties, may be included in the reconstituted tobacco of this invention. These additives include, for example, siliceous materials such as acid-treated clays, heat-treated montmorillonite, bentonite, and natural and synthetic silicates containing mobile hydrogen atoms; colloidal alumina, activated bauxite, light alumina hydrate, desulfated aluminum sulfate, etc.

Ether derivatives of tobacco are prepared using well known etherifying procedures on tobacco. For example,

the etherifying process can be broadly described as first treating the tobacco with an alkaline catalyst or activator and then reacting the alkaline treated tobacco with an organic etherifying agent. Etherifying agents preferably include, for example, alkylene oxides or epoxide compounds containing from 2 to 8 carbon atoms, for example, ethylene oxide, propylene oxide, butylene oxide, 3,4-epoxy-1-butene, 2-ethylhexyl oxide, epoxypropyl amines; organic halides containing from 2 to 8 carbon atoms, for example, methyl chloride, ethyl chloride, propyl chloride, 2-ethylhexyl chloride, ethyl bromide, ethyl iodide, ethylene chloride, propylene chloride, butylene chloride, ethylene chlorohydrin, propylene chlorohydrin, epichlorohydrin, monohalo fatty acids such as monochloro acetic acid, monochloro propionic acid, monobromo acetic acid, etc.; dialkyl sulfates such as diethyl sulfate and dimethyl sulfate.

Alkaline catalysts or activators include, for example, alkali and alkaline earth metal hydroxides and carbonates, basic quaternary ammonium hydroxide derivatives, ammonia, and organic amines including triethyl amine and tetraethylene diamine. Sodium hydroxide is a preferred alkaline medium.

Any tobacco is useful for the etherified material of this invention without limitations as to quality or origin. It may be an individual type or a blend. There is no requirement as to the size of the tobacco particles for the etherification reaction and they may range from dust-like material to leaf size. It is preferred, however, for ease of handling that the tobacco be comminuted to obtain particle sizes which will pass through sieves having a mesh ranging from about 50 to about 500.

The etherification can be carried out with or without the presence of organic solvents or diluents including, for example, carbon tetrachloride, ethyl ether, chloroform, heptane and benzene. The reaction will take place under increased pressure or at atmospheric and at elevated or room temperature, but it is preferred for obtaining the desired product within a reasonable time, that a raised temperature of at least 50° C. be used to heat the reaction mass which is maintained in a closed pressure reaction vessel.

It is believed that the etherification process and modifications thereof are so well known to those skilled in the art that no further description except for the following examples is necessary.

EXAMPLE I

An amount of 50 grams of cigar tobacco ground to pass a 100 mesh sieve and be retained by 200 mesh sieve was combined with 18.5 grams of sodium hydroxide dissolved in 50 grams of water by mixing by mechanical agitation for 15 minutes. The mixture was treated with 100 grams of propylene oxide, the whole introduced into a pressure reactor, 100 grams of dioxane added, and the closed vessel heated for four hours in a water bath at 50° C. The product mixture was then neutralized with dilute acetic acid, centrifuged, and the residue washed with water and an aqueous solution of acetone.

The residue was spread on a glass plate and allowed to dry in air. The dark brown, leathery product when removed from the glass plate was glassine on the side which had been in contact with the glass plate. The following table is set forth to demonstrate the excellent physical properties of the sheet prepared in accordance with the foregoing example as compared to conventional reconstituted tobacco sheets.

Table

Sample identification	Ream Wt. (lbs.)	Breaking Length (Meters)	Elongation, percent	Thickness (mils)
Control I [1]	50.4	557	30.6	3.3
Control II [2]	46.1	608	14.8	3.2
Sheet of Example I	31.2	1,310	5.4	2.7

[1] Reconstituted tobacco sheet comprising 20% carboxymethyl cellulose, 25% calcium carbonate, 20% glycerine and a remainder of tobacco.
[2] Reconstituted tobacco sheet comprising 20% carboxymethyl cellulose, 20% glycerine and a remainder of tobacco.

It will be obvious to one skilled in this art on reading the results set forth in the above table that the sheet of Example I demonstrates improvement in all properties listed in comparison to the conventional reconstituted tobacco sheets. These improved physical properties provide improved machineability in the manufacture of smoking products as well as excellent smoking properties.

EXAMPLE II

A water slurry of 9.5% of the product of Example 1, 1.5% glycerine and 89% water was prepared. The slurry was cast on a glass plate with a doctor blade set at 0.015 inch. The film was allowed to dry in air and then was easily peeled from the plate to form a film 0.004 inch thick. The film was characterized as thin, strong, pliable, slightly elastic with an appearance of natural thin tobacco leaf and having a slight tobacco odor.

EXAMPLE III

Ground tobacco was treated similarly with the same amounts and proportions of water and sodium hydroxide as disclosed in Example I. Then the mixture was treated with an amount of monochloro acetic acid equal to the weight of tobacco. No dioxane was used. The mass was heated at 50° C. for four hours in a closed pressure reaction vessel. The product mixture was neutralized with dilute acetic acid, centrifuged, and the mixture was washed in an aqueous solution of acetone.

A film of the reaction product was prepared by spreading a mixture thereof, water and glycerine as the plasticizer, on a glass plate with a doctor blade set at 0.015 inch. The carboxymethyl film when dried was similar in all physical characteristics to the hydroxypropyl derivative sheet described in Example II.

EXAMPLE IV

A water slurry of 8% of the product of Example I, 18% untreated ground tobacco, 1.2% glycerine and 72.8% water was prepared and cast on a glass plate with a doctor blade set at 0.015 inch. The film was allowed to dry in air and was easily peeled from the plate in the form of a film 0.014 inch thick. The sheet had the same appearance and odor of a commercial reconstituted tobacco sheet.

EXAMPLE V

A smoking product was prepared by wrapping shredded parts of the sheet produced in Example II in a wrapper consisting of the sheet produced in Example IV. The product was smoked and, except for the very mild taste of the totally synthetic cigarette, no deleterious taste or burning qualities were observed. The ash of the product was gray.

A cigarette was prepared by wrapping a whole cigarette in the sheet of Example II. Another cigarette was prepared by wrapping cigarette tobacco in the sheet of Example II. Both of these cigarettes were smoked. No deleterious taste or burning qualities were observed.

The method of the present invention is advantageously employed with attrited tobacco formed by the process set forth in copending application Serial No. 140,194, filed September 25, 1961, by O. A. Battista and R. J. Semeraro. The copending application concerns hydrolyzed, attrited tobacco and a method for the preparation of reconstituted

tobacco sheets. Generally, it is disclosed that tobacco, particularly whole leaf tobacco containing stems and veins, is subjected to a mild hydrolysis treatment to break down or loosen the amorphous areas present therein. The hydrolyzed tobacco is then neutralized and mechanically attrited to obtain colloid-forming size particles. Hydrolysis may be carried out with any mineral acid capable of degrading the tobacco. It is preferred for plant scale operation that the tobacco be subjected to from about 0.1 to about 1.0% aqueous solution of hydrochloric acid for about ½ to 2 hours at 200 to 300° F. in a pressure vessel.

The following example is set forth to demonstrate the preparation of a reconstituted sheet utilizing hydrolyzed attrited tobacco.

EXAMPLE VI

36.4 grams of whole leaf, coarse Wisconsin tobacco, 1.75 cc. of concentrated hydrochloric acid and 149 cc. of water were placed in a pressure bomb reactor which was closed and placed in an autoclave. The above mixture gave a 0.5% acid solution which is the equivalent of 4.8% hydrochloric acid based on the weight of the tobacco. The temperature of the autoclave was raised to 240° F. for 95 minutes and then the reactor was allowed to cool to room temperature. Thereafter the acid was neutralized with sodium hydroxide to a pH of 7.0. After neutralization the residue was diluted to a solids content of 15% and then attrited in a Waring Blendor for 45 minutes. This attrition completely broke down the particle size of the tobacco and gelation resulted. The entire mixture, including retained tobacco juices, was spray dried by introducing it into the hopper of a positive displacement pump which fed the slurry at the controlled rate of about 3 lbs. per hour to a high speed spiraling air stream having an input temperature of about 680° F. The dried product was collected in a cyclone collector and had a moisture content of about 5%. Microscopic examination determined that no fibrous bundles were present; all had been completely broken down into individual fine, fibrillar particles.

About 10 grams of the finely divided tobacco prepared as described above is combined with about 4 grams of sodium hydroxide dissolved in 10 cc. of water by mixing with mechanical agitation for 15 minutes. The mixture is treated with 20 grams of propylene oxide, the whole introduced into a pressure reactor, 20 grams of dioxane added, and the closed vessel heated for 4 hours in a water bath at 50° C. The product mixture is then neutralized with dilute acetic acid, centrifuged and the residue washed with water and an aqueous solution of acetone.

5 grams of the etherified tobacco is mixed with 20 grams of the finely divided hydrolyzed tobacco prepared as described above and 25 cc. of water. 5 grams of glycerine is added to the slurry with mixing and the mixture is spread on a glass plate and allowed to dry in air. The resulting sheet has excellent physical properties and contains no cellulosic binder to degrade smoking properties.

The above examples demonstrate the utility of the products of this invention as components for tobacco smoking products, and in particular, as binder sheets or, if embossed to assume the appearance of natural tobacco leaf, as an outer wrapper in the manufacture of cigars.

Various changes and modifications may be made in practicing the invention without departing from the spirit and scope thereof and, therefore, the invention is not to be limited except as defined in the appended claims.

I claim:

1. A composition of matter comprising an ether derivative of tobacco.

2. A composition of matter comprising a mixture of tobacco and an ether derivative of tobacco.

3. An article of manufacture comprising a self-supporting sheet of an ether derivative of tobacco.

4. The article of claim 3 wherein the ether derivative is the hydroxypropyl ether derivative.

5. An article of manufacture comprising a self-supporting sheet of a mixture of tobacco and an ether derivative of tobacco.

6. A tobacco smoking product comprising a shredded material containing an ether derivative of tobacco.

7. A tobacco smoking product comprising a shredded material selected from the group consisting of ether derivatives of tobacco and mixtures of a tobacco and an ether derivative of tobacco.

8. A tobacco smoking product comprising shredded material selected from the group consisting of tobacco, an ether derivative of tobacco and mixtures thereof, and a wrapper enveloping said shredded material containing an ether derivative of tobacco.

9. The tobacco smoking product of claim 8 wherein the ether derivative is the hydroxypropyl ether derivative.

10. The tobacco smoking product of claim 8 wherein the wrapper comprises a mixture of tobacco and an ether derivative of tobacco.

11. The tobacco smoking product of claim 10 wherein the ether derivative is the hydroxypropyl ether derivative.

12. The method of preparing reconstituted tobacco comprising etherifying tobacco in the presence of an alkaline catalyst, forming a slurry of the ether derivative of tobacco, casting a sheet from said slurry, and drying.

13. The method of claim 12 wherein the alkaline catalyst is sodium hydroxide.

14. The method of claim 13 wherein the tobacco is etherified with an alkylene oxide containing from 2 to 8 carbon atoms.

15. The method of claim 14 wherein the alkylene oxide is propylene oxide.

16. The method of preparing reconstituted tobacco comprising first hydrolyzing tobacco to at least partially decompose the amorphous portions thereof, mechanically attriting the hydrolyzed tobacco, etherifying the hydrolyzed attrited tobacco in the presence of an alkaline catalyst, forming a sheet with a slurry of the etherified tobacco, and drying the sheet.

17. The method of claim 16 wherein etherified, hydrolyzed, attrited tobacco is mixed with hydrolyzed, attrited tobacco to form the sheet.

References Cited in the file of this patent

UNITED STATES PATENTS

1,188,376 Lilienfeld ---------- June 20, 1916