

1

3,012,914

RECONSTITUTED TOBACCO PRODUCTS AND METHOD OF MANUFACTURE

Orlando A. Battista and Alan M. Belfort, Drexel Hill, and Edwin G. Fleck, Jr., Wallingford, Pa., assignors to American Viscose Corporation, Philadelphia, Pa., a corporation of Delaware

No Drawing. Filed Nov. 14, 1960, Ser. No. 68,676
12 Claims. (Cl. 131-17)

This invention relates to tobacco products. More particularly, this invention relates to improved reconstituted tobacco products and methods of manufacture.

Reconstituted tobacco of which many tobacco products are now being made is formed by disintegrating tobacco leaf or parts of the leaf in a mill or grinder to reduce the tobacco to a fine powder-like form. A binding agent such as sodium carboxymethyl cellulose or other suitable aqueous adhesive is incorporated in the disintegrated tobacco, the product is rolled out to form a flat sheet and is then dried to the desired moisture content. While it is necessary that a suitable adhesive and sufficient of that adhesive be used to form a reconstituted tobacco sheet that has sufficient strength so that it may be handled during further processing, it is desirable that the adhesive not deleteriously affect the flavor or burning properties of the tobacco. It is also desirable that the adhesive not perceptibly change the physical appearance of the reconstituted tobacco. In forms of reconstituted tobacco of the prior art, the product does not have the stiffness or hand of the natural product but often has a rubbery feel or character.

This invention has for its principal object to provide improved reconstituted tobacco products.

Another object of this invention is to provide reconstituted tobacco products having improved binder material therein.

A further object of the invention is to provide methods of making improved reconstituted tobacco products having an improved binding material between the particles thereof.

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

The invention, in general, comprises disintegrating already sweated, fermented or cured tobacco leaf or parts of the tobacco leaf in a grinder or mill until it is in a fine powder-like form. As an example, the ground tobacco is fine enough to pass through a 20 mesh screen. An aqueous suspension of cellulose crystallite material or a water-insoluble alkali-soluble ether thereof, which suspension and material will be more particularly described hereinafter, is thoroughly mixed with the ground-up tobacco. It is also contemplated in accordance with this invention that an aqueous solution of a water-soluble ether of the cellulose crystallite material may be used. The mixing of the cellulosic suspension and the tobacco forms an aqueous plastic or pasty mass. This mass is then spread in a relatively thin layer on a flat sheet or belt and part of the water is removed. The resulting sheet of the reconstituted tobacco is then ready for further processing.

The reconstituted tobacco of this invention in sheet form has much the same stiffness as the natural tobacco and is particularly characterized by an absence of any limp or rubbery feel.

Cellulose crystallite aggregates, together with their properties and the manner of obtaining them, are described at length in the copending application of O. A. Battista, Ser. No. 33,941, filed June 6, 1960. For convenience, some salient features of such description are given here it being understood that the said application may be consulted for a more complete description. The aggregates are products obtained by the controlled acid

2

hydrolysis of cellulose, there being formed an acid-soluble portion and an acid-insoluble portion. The latter comprises a crystalline residue or remainder which is water washed and recovered, being referred to as cellulose crystallite aggregates, or as level-off D.P. (degree of polymerization) cellulose. These aggregates, in the state resulting from the hydrolysis and washing steps, in which state they may be designated as-formed aggregates, are then subjected to mechanical disintegration or attrition, as described below. It being apparent that the as-formed aggregates are the precursors of the disintegrated aggregates, the preparation and characteristics of the former will be described.

In the acid hydrolysis, the acid destroys or removes amorphous portions of the original cellulose chains, the remaining unattacked portions being in a particulate, non-fibrous or crystalline form as a result of the disruption of the continuity of the fine structures between crystalline and amorphous regions of the original cellulose. Although hydrolysis may be effected by various specific methods, including the use of various acids, a direct method which is free of secondary reaction comprises the treatment of the original cellulosic material with 2.5 normal hydrochloric acid solution for 15 minutes at boiling temperature.

The cellulose undergoing the hydrolysis reaches, within the time period noted, a substantially constant molecular weight, or in other words, the number of repeating units or monomers, sometimes designated anhydroglucose units, which make up the cellulosic material, becomes relatively constant, from which it is apparent that the degree of polymerization of the material has leveled off, hence the name level-off D.P. cellulose. In other words, if the hydrolysis reaction were continued beyond the period noted, the D.P. would change very little if at all. In all cases, the level-off D.P. value reflects the fact that destruction of the fibrous structure has occurred as a result of the substantially complete removal of the amorphous regions of the original cellulose.

It may be observed that "crystallite," as used herein, is a cluster of longitudinally disposed, closely packed cellulose chains or molecules, and that "aggregates" are clusters of crystallites. The aggregates may also be said to comprise straight, rigid, relatively non-twistable groups of linear chains. As indicated by X-ray diffraction tests, the crystallites and crystallite aggregates have a sharp diffraction pattern indicative of a substantially crystalline structure. Although the crystallite chains are of very uniform lengths, particularly by comparison with the original cellulose chains, strictly speaking they do exhibit some variation, and for this reason it is preferred to speak of average chain length, or of average level-off D.P. values.

The hydrolysis methods noted are particularly characterized in that in each crystallite aggregate resulting from the hydrolysis, no constituent chain is connected to a chain in a neighboring aggregate; rather, all the chains in an aggregate are separate from and free of those in neighboring aggregates.

The cellulose crystallite aggregates, or level-off D.P. cellulose, suitable for use in the invention is characterized by having a preferred average level-off D.P. of 125 to 375 anhydroglucose units. Ideally, within this range all of the material should have the same D.P., or chain length, but as this is difficult if not impossible to achieve, it is preferred that at least 85% of the material have an actual D.P. not less than 50 and not more than 550. It may thus be apparent that the chain length of the level-off D.P. cellulose is very uniform, a consequence of the hydrolysis, wherein the longer chains of the original cellulose were converted to shorter chains and the very short chains were removed. As an example, reference to crystallite aggregates having an average level-off D.P.

of 125 means that the aggregates have an average chain length corresponding to 125 anhydroglucose units, and at least 85% of this material is made up of chains containing 50 to 350 such units; the remaining 15% may comprise shorter and/or longer chains.

More preferably, the average level-off D.P. is in the range of 200 to 300, of which material at least 90% has an actual D.P. in the range of 75 to 550.

Associated with the foregoing D.P. properties of the crystallite aggregates is the fact that their chemical purity is very high, the material comprising at least 95%, preferably at least 97% or 99%, polyglucose, or anhydroglucose units, based on chromatographic analysis. In terms of ash content, the aggregates preferably contain less than 100 p.p.m. (parts per million), although ash may range from about 10 to about 400 or 500 or 600 p.p.m. By comparison, conventional fibrous cellulose may have 1000 to 4000 p.p.m. of ash.

Other suitable cellulose crystallite aggregates may have lower average level-off D.P. values, say in the range of 60 to 125, or even 15 to 60. Aggregates from both of these ranges have the chemical purity and other characteristics noted above. Crystallite aggregates in the 60 to 125 average level-off D.P. range are obtainable from the acid hydrolysis of alkali swollen natural forms of cellulose, of which a preferred source is cellulose that has been mercerized by treatment with 18% caustic soda solution at 20° C. for two hours. Aggregates in the 15 to 60 average level-off D.P. range are suitably prepared from regenerated forms of cellulose, including tire and textile yarns, other regenerated cellulose fibers, and cellophane.

In every case the cellulosic source material has a D.P. greater than the level-off D.P. thereof.

As obtained from the acid hydrolysis and water washing steps, i.e., in their as-formed state, the aggregates in the over-all average level-off D.P. range of 15 to 375 are in a loosely aggregated state, and particularly in the larger particle sizes, say from 40 to 250 or 300 microns, are characterized by the presence of many cracks in their surfaces, including similar surface irregularities or phenomena like pores, depressions, voids, fissures, and notches. Because of such irregularities, the apparent or bulk density of the aggregates is much less than their absolute density. In terms of lbs. per cu. ft., the bulk density of the aggregates may range from 7 or 8 to about 34 or 35 lbs. per cu. ft.

The as-formed aggregates are further characterized by having a particle size in the over-all range of 1 or 2 to 250 to 300 microns, as determined visibly by microscopic examination. By subjecting the foregoing product to mechanical disintegration, there is produced a material having a size in the over-all range of less than 1 to about 250 or 300 microns, and as will be understood, the proportions of material in the lower size ranges will be increased over those of the non-disintegrated aggregates. It will also be understood that the particle size and size distribution may be selected to suit a particular end use.

Either before or after mechanical disintegration, preferably after, the aggregates may be dried.

Mechanical disintegration of the aggregates may be carried out in several ways, as by subjecting them to attrition in a mill, or to a high speed cutting action, or to the action of high pressures on the order of at least 5,000 or 10,000 p.s.i. The disintegration is preferably carried out in the presence of an aqueous medium. Whatever method is used, the disintegration is extensive enough so that the resulting disintegrated aggregates are characterized by forming a stable suspension in the aqueous medium in which they are being attrited, or in which they may be subsequently dispersed. By a stable suspension is meant one from which the aggregates will not settle out but will remain suspended indefinitely, even for periods measured in terms of weeks or months. As described hereinafter, at lower concentrations of aggregates, the

suspension is a dispersion, while at higher concentrations it is a gel.

If the as-formed aggregates are not mechanically disintegrated, they will, upon being mixed with water, settle much like fine sand, and this is true if they are merely stirred in water. Furthermore, mechanical milling or grinding of the dry aggregates will not disintegrate them to a state where, without further treatment, they will disperse in water to form stable dispersions or gels.

The preferred disintegration method is to attrite the aggregates by means of a high speed cutting action in the presence of an aqueous medium. It is preferred that the water content of the mixture undergoing attrition should be at least 10, 15, or 20% by weight; and the aggregates content should be at least 3% by weight and desirably higher as the efficiency of the cutting action increases with the aggregates content. Suitable consistencies are those of mixtures containing up to about 35%, say about 7 to 35%, by weight of aggregates and the balance water; such mixtures lend themselves well to good attrition, are convenient to handle and have the advantage of directly producing a gel. At consistencies of 3 to 6% the attrited product is usually a dispersion but can be a gel, especially at 4 to 6% consistency. Attrition may be performed of mixtures of consistencies above about 35%, say from 35 to 70%, and although the attrited products are not gels, they have the distinctive property of forming indefinitely stable, smooth gels of varying thickness and striking appearance upon the addition of water and stirring manually, as with a spoon, for a few minutes. At about 70 to 80% consistency, attrition results in a damp but free flowing material comprising discrete grains or granules and clumps of grains; the moisture content is apparent to the touch rather than the eye; and the material forms a gel upon being manually stirred or beaten in water. At 80 to 90% consistency, the product of attrition is a crumbly, free flowing, grainy, dry-appearing material that does not have a damp feel and which requires energetic beating in the presence of water to form a gel.

Surveying briefly the characteristics of the dispersions and gels, they comprise attrited products of an attritable mixture having a solids content of at least 3% by weight during the attrition step. Necessarily, the resulting attrited product will also have at least 3% solids, although some useful materials are obtainable by diluting such attrited product. In the next place, at least 1% by weight of the solids in the product of attrition have a particle size of up to 1 micron. In the third place, the attrited product forms substantially adherent films, preferably substantially continuous and self-supporting films, when applied to suitable surfaces. Finally, the attrited product is, or forms, a stable and homogeneous colloidal dispersion or gel, the term "homogeneous" referring to the uniform visual appearance of the dispersion or gel. With respect to the last mentioned characteristic, it will be understood that stable, homogeneous, colloidal dispersions and gels, as contemplated herein, are free of layers of sediment; there is no bottom layer of sediment; nor is there a top layer of visibly lower solids content than the balance of the mixture. Rather, the stable dispersions and gels are uniform and homogeneous throughout; have a uniformly white color, some mixtures being more, or less, intensely white than others, depending on the aggregates content and particle size distribution; and are further characterized by having a very smooth butterlike mouth feel. The preferred dispersions and gels are those that are stable for at least a month, and another preferred group comprises those stable for at least a week. Dispersions and gels that are stable for at least a day, or even an hour or less, are also useful for some purposes, as where they are to be used almost immediately. But as may be apparent, the more stable dispersions and gels have the advantage of being storable for a considerable period of time.

For purposes of the invention, a dispersion may be defined as having about 1 to about 8% by weight of the aggregate dispersed in the aqueous or other liquid, the latter constituting the continuous phase of the mixture. The dispersion has the physical form or appearance of a liquid, and is flowable like a liquid. A gel may be defined as having about 3 to 35% by weight of aggregates dispersed in the aqueous or other liquid, and in this case the aggregates constitute the continuous phase of the mixture. The gel has the physical form of a jelly, paste, plastic mass or the like. As noted, both dispersions and gels are included by the term suspension.

Following mechanical disintegration of the aggregates, the resulting product, whether a dispersion or gel, may be taken and used as such; or it may be de-watered and dried; or it may be desirable to fractionate it into fractions having a more homogeneous particle size and size distribution.

In respect of the drying of the gels, it should be observed beforehand that the preferred gels are those obtained by attriting the never-dried hydrolysis product; these gels have very desirable qualities in respect of smoothness, mouth feel, texture, etc. They may be dried to any practical moisture content, in which state they are redispersible in water, by the aid of a suitable attrition step, to form a gel, and this latter gel may again be dried if desired and again redispersed to form a subsequent gel. Gels are also obtainable by attriting the dried hydrolysis product, and these gels may be dried, or dried and attrited to again form gels.

For producing the dried products, a number of drying procedures are available, and while redispersible materials result from each procedure, some procedures are more advantageous than others. For example, freeze drying, spray drying, drum drying, and drying by solvent displacement each produce a material which has an appreciably lower bulk density than conventionally oven-dried materials, with freeze drying producing the lowest bulk density by far, viz., 8 or 9 lbs./cu. ft. as against 14 lbs./cu. ft. for oven-dried aggregates; each produces a material which is more easily redispersible in water, by the aid of an attrition step, to form a stable suspension than air- or oven-dried materials; and each yields a more reactive product than air-dried or oven-dried products, as judged by acetylation with a conventional acetylating reagent mixture. Freeze-dried, spray-dried, drum-dried, and solvent displacement-dried materials are noticeably softer to the touch than products of the other drying steps; and freeze drying also produces the most porous products. With regard to the mouth feel of the various materials, those made by freeze drying and spray drying are superior.

Fractionation of the attrited products may be accomplished by means of such separation procedures as mechanical sifting, settling in water, or centrifuging, a number of useful fractions being obtainable, including fractions having a particle size of up to 0.2, 1, 2, 5, or 10 microns. Still another desirable fraction is one whose dimensions are all below 100 microns, or below 40 or 50 microns. Preferably, each dimension of the particles should be within the size range noted for each fraction; however, particles having two dimensions within the size range are quite useful, as are particles having but one dimension within the size range although they are less preferred.

Water is a preferred medium in which to disintegrate the crystallite aggregates. Other suitable media are aqueous mixtures comprising water and one or more water-miscible oxygen-containing, preferably hydroxy- and carbonyl-containing, compounds. Hydroxy compounds are a preferred class, particularly polyols, comprising aliphatic compounds having two or more hydroxy groups, of which glycerol is an example. Glycol ethers are suitable, as are water-miscible, low molecular weight alcohols. Other useful water-miscible compounds are those

containing carbonyl groups, including organic acids, esters, aldehydes and ketones. Other compounds are ethers and oxides like ethylene oxide, propylene oxide, etc. Aqueous sugar solutions are useful. Water may be omitted from any of the foregoing aqueous mixtures and the non-aqueous compound itself, or mixtures of two or more thereof, may be employed as the medium.

The cellulosic crystallite material of this invention may be either the disintegrated cellulose crystallite material or water-insoluble alkali-soluble ethers thereof. Examples of the water-insoluble ethers that may be used are: methyl, ethyl, carboxymethyl, hydroxyethyl, and hydroxypropyl ethers of the cellulose crystallites.

The cellulose crystallite material may be etherified to the alkali-soluble, water-insoluble state either before the cellulose crystallite material is disintegrated or the resulting ether may be disintegrated. In either practice the etherification is carried out with the usual catalysts and reagents. The conditions and quantities are so controlled that the degree of etherification and the resulting ether of the crystallite cellulose is water-insoluble. For most purposes a water-insoluble ether having an average etherification of 0.2 to 0.6 ether groups per anhydroglucose unit is satisfactory.

The water-soluble ether of the cellulose crystallite material may be formed by etherifying either the disintegrated or the non-disintegrated cellulose crystallite material. The etherification is carried out to a higher degree of substitution and it is found that an average degree of substitution or etherification of from 1.0 to 1.5 ether groups per anhydroglucose unit is satisfactory. Examples of the water-soluble ethers that may also be used are: methyl, ethyl, sodium carboxymethyl, hydroxyethyl, and hydroxypropyl ethers of the cellulose crystallites.

The usual flavorings, sauces, aroma materials, humectants and the like may be added to the tobacco either before or after the tobacco has been disintegrated; or they may be added to the cellulose crystallite material or the ethers thereof either before or after the material is disintegrated and the suspension thereof if formed. The usual flavors include invert sugar, licorice, chocolate, cocoa, fruit materials and pastes, aromatic oils and resins, maple syrup, vanilla, rum flavors and the like. The humectant may be the usual glycerine, ethylene glycol, propylene glycol or the like.

The amount of the cellulosic material that is incorporated into the tobacco is, in general, an amount that is sufficient to bind the particles together with sufficient strength but is not so great an amount that the cellulosic crystallite material is readily apparent on the tobacco product as it is smoked. Because of the improved binding properties of the cellulosic crystallite material, less of this material is required than would be required if some conventional adhesive or binding agent were used. For most purposes of this invention, the dry weight of the cellulosic crystallite material that may be added to the tobacco is from approximately 2 to 15% of the weight of the resulting tobacco product when the cellulosic crystallite material is the only binding material used. If desired, for certain purposes, part of the cellulosic crystallite material may be replaced by conventional adhesive or binding agents for reconstituted tobacco.

For uses where the reconstituted tobacco sheet is subjected to more handling or manipulative steps, the reconstituted tobacco sheet may be further reinforced by incorporating into the tobacco mix short lengths of rayon, hemp or other vegetable fibers. The reconstituted tobacco sheet may also be reinforced by spreading the aqueous mixture of the suspension of the cellulosic crystallite material and the disintegrated tobacco on one or both sides of a sheet of high strength cellulose tissue-like paper. The tobacco product is then dried with the reinforcing sheet incorporated in it.

The reconstituted tobacco product of this invention may be used as a filler, binder or wrappings for cigars or may

be cut or shredded for use in cigarettes or in pipe tobacco.

The cellulosic crystallite material of this invention leaves practically no ash as compared to other cellulosic materials.

With the ethers of the cellulose crystallite material, solutions or suspensions of relatively low viscosity can be prepared with a higher solids content. It is easier to mix this binder with the tobacco. A stiffer, thinner, and more natural tobacco produced is obtained in which the binding material is practically imperceptible and does not detract from the feel and appearance of the product.

The following examples are illustrative of the invention:

Example 1

In a suspension comprising 8 parts by weight dry of the disintegrated cellulose crystallite material in 375 parts of water, 12 parts of glycerine is stirred. To this suspension 100 parts by weight of disintegrated tobacco of 20 mesh size or less is added and the mixture is stirred and kneaded to form an aqueous paste-like or plastic mixture of the tobacco and the cellulose crystallite material. The plastic mass is spread in a thin layer on a plate glass surface and a smooth aluminum foil is placed on top of the layer. The plastic mass is pressed between the glass plate and the aluminum foil by means of a roller to a thickness of $\frac{1}{60}$ to $\frac{1}{30}$ of an inch. The aluminum foil is removed and the tobacco product layer on the plate glass is dried at a temperature of 50 to 60° C. until the layer or sheet of reconstituted tobacco is self-supporting but still pliable. The reconstituted tobacco sheet is then ready for further processing such as cutting or shredding for use in cigars, cigarettes or pipe tobacco.

Example 2

The same procedure is followed as in Example 1 except that instead of the 8 parts by weight of the disintegrated cellulose material, a mixture of 6 parts by weight dry of the disintegrated cellulose crystallite material and 2 parts by weight dry of commercial sodium carboxymethyl cellulose is used.

Example 3

An amount of 100 parts by weight of disintegrated tobacco that will pass through a 20 mesh screen is thoroughly mixed into a suspension of 500 parts by weight of water, 1 part by weight sodium alginate, 1 part by weight gum tragacanth, 8 parts by weight of glycerine, and 12 parts by weight dry of the disintegrated cellulose crystallite material. The aqueous plastic mixture is formed into a sheet of reconstituted tobacco by the procedure of Example 1.

Example 4

An aqueous paste-like or plastic mass was formed by thoroughly mixing together 0.81 part by weight dry sodium carboxymethyl cellulose, an aqueous suspension containing 0.81 part by weight dry of the disintegrated cellulosic crystallite material, 2.42 parts by weight glycerine and 75.76 parts by weight of tobacco. The aqueous paste-like or plastic mass is formed into a sheet of reconstituted tobacco containing 7.4% by weight water by the procedure of Example 1.

Example 5

The aqueous plastic mass of tobacco of Example 2 is spread on one side of a sheet of 10 pound rayon fiber tissue-like paper. The sheet is dried as in Example 1 to form a sheet of reconstituted tobacco reinforced with the layer of paper tissue.

The paper tissue is 10% by weight of the composite reconstituted tobacco product.

Example 6

The aqueous plastic mass of tobacco of Example 2 is spread in thin layers on both sides of a sheet of 10 pound

rayon fiber tissue-like paper. The composite sheet is dried as in Example 1 to form a composite reconstituted tobacco sheet in which the reinforcing tissue paper is 5% by weight of the composite sheet.

Example 7

An aqueous suspension comprising 2 parts by weight dry of disintegrated water-insoluble hydroxypropyl ether of cellulose crystallite material is mixed together with 2 parts by weight of glycerine, 12 parts by weight of disintegrated tobacco that will pass through a 20 mesh screen and additional water so that the total water present is 60 parts by weight. The mixture is thoroughly stirred and forms a paste or plastic mass of the tobacco and the cellulose material. The plastic mass is spread in a thin layer on a flat surface and is pressed by a roller to evenly spread the plastic mass on the flat surface and to reduce its thickness. The tobacco product layer is then dried on the flat surface until it is sufficiently strong that it may be handled but continues in the flexible or pliable state.

Example 8

An aqueous suspension comprising 2 parts by weight dry, of disintegrated water-insoluble hydroxypropyl ether of cellulose crystallite material is mixed together with 2 parts by weight of glycerine, 12 parts by weight of disintegrated tobacco that will pass through a 20 mesh screen and additional water so that the total water present is 70 parts by weight. The mixture is thoroughly stirred and forms a paste or plastic mass of the tobacco and the cellulose material. The plastic mass is spread in a thin layer on a flat surface and is pressed by a roller to evenly spread the plastic mass on the flat surface and to reduce its thickness. The tobacco product layer is then dried on the flat surface until it is sufficiently strong that it may be handled but continues in the flexible or pliable state.

The invention may also be carried out using the procedures of Examples 1 to 8 and substituting for the suspension containing the disintegrated cellulose crystallite material or the hydroxypropyl cellulose, an aqueous suspension or solution containing the other ethers of the cellulose crystallite material disclosed above.

While preferred embodiments of the invention have been disclosed and described, it is to be understood that changes and variations may be made without departing from the spirit and scope of the invention as defined in the appended claims.

We claim:

1. A reconstituted tobacco product comprising disintegrated tobacco and a binding agent binding particles of the tobacco together comprising cellulosic crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units of the group consisting of cellulose crystallite aggregates and ethers thereof.

2. A reconstituted tobacco product comprising disintegrated tobacco and a minor amount of binding agent binding particles of the tobacco together comprising cellulosic crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units of the group consisting of cellulose crystallite aggregates and ethers thereof.

3. A reconstituted tobacco product comprising disintegrated tobacco and 2 to 15% by weight of binding agent for binding particles of the tobacco together comprising cellulosic crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units of the group consisting of cellulose crystallite aggregates and ethers thereof.

4. A reconstituted tobacco product comprising disintegrated tobacco and a minor amount of binding agent binding particles of the tobacco together comprising disintegrated cellulose crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units.

5. A reconstituted tobacco product comprising disintegrated tobacco and a minor amount of binding agent binding particles of the tobacco together comprising disintegrated water-insoluble ethers of cellulose crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units.

6. A reconstituted tobacco product comprising disintegrated tobacco and a minor amount of binding agent binding particles of the tobacco together comprising water-soluble ethers of cellulose crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units.

7. A method of making a reconstituted tobacco product comprising mixing together disintegrated tobacco and a binding agent comprising aqueous disintegrated cellulosic crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units of the group consisting of cellulose crystallite aggregates and ethers thereof, forming a layer of the mixture, and removing water from the layer to form a sheet of reconstituted tobacco.

8. A method of making a reconstituted tobacco product comprising mixing together disintegrated tobacco and a minor amount of a binding agent comprising aqueous disintegrated cellulosic crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units of the group consisting of cellulose crystallite aggregates and ethers thereof, forming a layer of the mixture and removing water from the layer to form a sheet of reconstituted tobacco.

9. A method of making a reconstituted tobacco product comprising mixing together disintegrated tobacco and a minor amount of a binding agent comprising aqueous disintegrated cellulosic crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units of the group consisting of cellulose crystallite aggregates and ethers thereof, forming a layer of the mixture and removing water from the layer to form a sheet of reconstituted tobacco, the amount of the binding agent being from approximately 2 to 15% by weight of the sheet.

10. A method of making a reconstituted tobacco product comprising mixing together disintegrated tobacco and a minor amount of a binding agent comprising an aqueous suspension of disintegrated cellulose crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units, forming a layer of the mixture and removing water from the layer to form a sheet of reconstituted tobacco the amount of the binding agent being from approximately 2 to 15% by weight of the sheet.

11. A method of making a reconstituted tobacco product comprising mixing together disintegrated tobacco and a minor amount of a binding agent comprising an aqueous suspension of disintegrated water-insoluble ethers of cellulose crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units, forming a layer of the mixture and removing water from the layer to form a sheet of reconstituted tobacco, the amount of the binding agent being from approximately 2 to 15% by weight of the sheet.

12. A method of making a reconstituted tobacco product comprising mixing together disintegrated tobacco and a minor amount of a binding agent comprising an aqueous solution of a water-soluble ether of cellulose crystallite aggregates having an average level-off D.P. of 15 to 375 anhydroglucose units, forming a layer of the mixture and removing water from the layer to form a sheet of reconstituted tobacco, the amount of the binding agent being from 2 to 15% by weight of the sheet.

References Cited in the file of this patent

UNITED STATES PATENTS

2,978,446 Battista et al. ----- Apr. 4, 1961

OTHER REFERENCES

Journal of Polymer Science, vol. X, No. 6, pages 577-586.

Textile Research Journal, vol. XXV, No. 6, June 1955, pages 534-540.