[72] Inventor Howard Martin Halter Norwalk, Conn. [21] Appl. No. 844,652	3,528,434 9/1970 Halter et al 131/17 AC X FOREIGN PATENTS
[22] Filed July 24, 1969 [45] Patented Dec. 7, 1971 [73] Assignee AMF Incorporated	1,055,473 1/1967 Great Britain
[54] RECONSTITUTED TOBACCO 5 Claims, No Drawings	No. 64- 22188. Pages 490- 492 cited. Primary Examiner—Samuel Koren
[52] U.S. Cl	Assistant Examiner—George M. Yahwak Attorneys—George W. Price and Murray Schaffer
[51] Int. Cl	
[56] References Cited UNITED STATES PATENTS 3,288,148 11/1966 Detert et al 131/17 AC X	ABSTRACT: A reconstituted tobacco composition and method of manufacturing same wherein a slurry of tobacco and solvent is created and has added thereto ethylhydroxyethyl cellulose as a burn improver.

RECONSTITUTED TOBACCO

This invention relates to reconstituted tobacco products with improved burn and taste characteristics. More particularly, this invention relates to the use of a particular grade of 5 ethylhydroxethyl cellulose in reconstituted tobacco compositions and to the novel and beneficial properties which result therefrom.

The usage of reconstituted tobacco products as filler in cigarettes and as wrapper and binder in cigars has increased 10 significantly in recent years. In general, the incentive to use such products in smoking articles is largely economic, since the reconstitution process allows the utilization of tobacco plant portions which were formerly waste because their physical form was unsuitable for manufacturing operations; i.e., 15 tobacco leaf processing fines, tobacco stems, etc. Equally as important for cigar applications are the labor savings which result from the automation of cigar manufacture with reconstituted tobacco sheet material which, in roll form, leads itself to automatic machine dispensation.

However, although the economics of usage of reconstituted tobacco products are unquestionably favorable, the acceptance and usage of such products by manufacturers has not developed as rapidly as would be anticipated. A major reason for this retarded rate of usage is that the favorable economics is the deciding factor only if consumer acceptance of smoking articles containing reconstituted tobacco is equal to or better than the corresponding products containing natural tobacco which are being replaced. However, consumer acceptance of these reconstituted tobacco products, especially in cigars has not been equal to that of the natural tobacco counterparts. The major problem areas involve burn and taste, which have been noticeably deficient for reconstituted tobacco products. These problem areas are related, since poor burn characteristics result in different combustion products and are generally accompanied by poorer taste characteristics. The differences in burn and taste of reconstituted tobacco products as compared to natural tobacco are probably the resultant of the use of nontobacco ingredients in such 40 products (gums, fibers, insolubilizing agents, humectants, etc.), structural differences related to the reconstitution process employed (paper process, slurry process, etc.) and changes caused by the particular processing conditions employed (drying, losses of volatiles, etc.).

This problem which reconstituted tobacco compositions has been recognized for a number of years. Frankenburg, in U.S. Pat. No. 2,706,695, issued Apr. 19, 1955, attributed the problem mainly to the water-soluble cellulosic derivatives used as adhesive binders in such formulations, and proposed the use of siliceous catalysts to alter the combustion characteristics of such formulations. Although some improvements due to such burn catalysts were noted, the experience of the last 14 years has shown that this approach was far from a complete solution to the problem.

It is interesting to note that Frankenburg (and his successors in the art) considered the various adhesives and particularly adhesives falling in the class of cellulose derivatives as being largely responsible for the burn-taste problems encountered in the manufacture of reconstituted tobacco. By and large, this 60

was and is the case; however, we have now discovered a unique cellulose derivative, providing novel function and results opposite to those obtained from the prior art teachings.

The burn deficiencies of reconstituted tobacco types in the prior art which are visibly overcome by the present invention 65 include "burn ring" and "burn duration" problems. The term "burn ring" refers to the black char ring on a cigar wrapper directly behind the burning zone of the cigar. With natural-tobacco wrapped cigars, the burn ring is very thin, about one sixty-fourth of an inch in width, while the smoker is puffing on 70 the cigar, and may widen to perhaps one thirty-second to one-sixteenth inch when the smoker lays his cigar down for several minutes in an ashtray. With reconstituted cigar wrappers, these width values may be approximately one-sixteenth to one-eighth inch while smoking and one-eighth to three-eighth 75

inches after several minutes without puffing. The appearance of an enlarged burn ring has been found to be particularly objectionable to cigar smokers, and is indicative of poor combustion with resultant adverse taste implications. "Burn duration" refers to the amount of time that a lit cigar will remain lit when placed in an ashtray without puffing. With natural-tobacco wrapped cigars, burn duration varies somewhat with cigar shape, and may range from 6 minutes on a cigarillo to about 10 minutes on large cigars. Reconstituted tobacco cigars which go out significantly faster than the conventional natural tobacco types are annoying to cigar smokers. In fact, burn durations which are somewhat longer than those for the natural tobacco cigars are considered desirable.

Accordingly, it is an object of this invention to provide a novel reconstituted tobacco product and a process for making same.

It is a further object of this invention to provide a reconstituted tobacco product which, because of its composition and mode of formation, will have substantially improved and commercially acceptable burn properties.

It is still a further object of this invention to provide a reconstituted tobacco composition which, because of its substantially improved burn properties, will have significantly improved organoleptic properties as well.

Additional objectives will become apparent from the description of the invention which follows.

While the invention will be described in connection with tobacco sheet, the burn and taste improvement additive of the 30 present invention may likewise be advantageously used with tobacco in reconstituted compositions with other shapes such as rods, cylinders, plugs, shreds and the like. Accordingly, it will be understood that the invention contemplates also tobacco manufacture of this type.

According to the present invention, when ethylhydroxyethyl cellulose (EHEC) with an ethoxyl degree of substitution (D.S.) of 1.2 to 1.6 and a hydroxyethyl molar substitution of 0.5 to 1.2 is incorporated in a reconstituted tobacco product at a level of at least 3 percent, the burn and taste advantages described previously result.

As used herein, the terms "degree of substitution" (D.S.) and "molar substitution" (M.S.) can be explained as follows: There are three hydroxyl groups in each anhydroglucose unit in the cellulose molecule. D.S. is the average number of hydroxyl groups substituted in the cellulose per anhydroglucose unit. M.S. is the average number of moles of reactant combined with the cellulose per anhydroglucose unit. For the alkyl, carboyxalkyl, or acyl derivatives of cellulose, the M.S. is generally greater than the D.S. The reason for this is that each time a hydroxyalkyl group is introduced into the cellulose molecule, an additional hydroxyl group is formed which itself is capable of hydroxyalklation. As a result of this, side chains of considerable length may form on the cellulose molecule. The M.S./D.S. ratio represents the average length of these side chains. Thus, from the foregoing, it will be seen that the total D.S. of a cellulose derivative can be no higher than 3, whereas the M.S. may be considerably higher than 3, depending on the extent to which side chains are formed.

With a mixed ether such as ethylhydroxethyl cellulose, the ethoxyl content is reported as D.S. since, as noted above, the D.S. and M.S. must be the same for this constituent. The hydroxyethyl substituent, on the other hand, is reported as M.S. since addition to side chains is possible in this case.

The two most widely used methods for determining M.S. are the Zeisel-Morgan method and the terminal methyl method. The Zeisel-Morgan method is reported beginning at page 500, vol. 18, 1946, of *Industrial and Engineering Chemistry*, Analytical Edition. The terminal methyl method is reported by Lemieux and Purves beginning at page 485, vol. 25B, 1947, of *Canadian Journal of Research*. As used herein, the ethoxyl D.S. determined by the terminal methyl method and the hydroxethyl M.S. was determined by the Ziesel-Morgan method.

these width values may be approximately one-sixteenth to one-eighth inch while smoking and one-eighth to three-eighth 75 Referring to the present invention, other 0.8 of ethylhydroxyethyl cellulose are referred to in the prior art. More specific

than the use described in the aforementioned Frankenburg patent is the use of the normal commercial grade of ethylhydroxyethyl cellulose as the adhesive in reconstituted tobacco compositions reported by Rosenberg and Schmidt in U.S. Pat. No. 3,042,552 (July 3, 1962). The ethoxyl content of this material is listed by Rosenberg and Schmidt as 17-18 percent (D.S. = about 0.8), and the hydroxyethyl content at about 17.3 percent (M.S. = about 0.9). The ethylhydroxyethyl cellulose used by Rosenberg and Schmidt is different in composition than that of the present invention, which involves an 10 ethoxyl degree of substitution of 1.2 to 1.6 and hydroxyethyl molar substitution of 0.5 to 1.2. The differences are even greater than one might anticipate from the substitution differentials, and are in fact differences in kind, not just in degree. The normal ethylhydroxyethyl cellulose (EHEC) used by Rosenberg and Schmidt will dissolve in water at temperatures up to about 60° C. The EHEC of this invention will not dissolve in water if the temperature is over 25° C. and, for complete solubilization, the temperature should be below 15° C. The Rosenberg and Schmidt EHEC will gel or precipitate from aqueous solution when the temperature is raised to 55°-70° C.; the EHEC of the present invention will gel or precipitate from aqueous solution when the temperature is raised to only about 32° -38° C. The Rosenberg and Schmidt 25 EHEC is insoluble in methanol or ethanol or 90 percent isopropanol; the EHEC of the present invention is completely soluble in these solvents. In spite of the fact that both materials are ethylhydroxyethyl celluloses, the differences in substitution between the two grades have resulted in physical and 30 chemical properties for the two grades of gum that are as different from each other as one would expect from adhesives of entirely different classes.

The properties contributed to burn and taste of reconstituted tobacco compositions by the incorporation of the 35 EHEC of the present invention are also radically different from those which are obtained using other carbohydrate, protein or synthetic gums. Furthermore, the burn and taste properties are radically different from those which are obtained with any other cellulose ether and even from other 40 grades of ethylhydroxyethyl cellulose with different levels of substitution. Among the carbohydrate adhesives investigated were animal gums, such as glycogen, plant gums and derivatives, such as cellulose ethers, cellulose esters, starches, starch ethers, starch esters, amylose, amylopectin and their ester and ether derivatives, locust bean gum, guar gum, gum arabic and related seed gums and plant exudate gums, marine plant gums, such as algins, carrageenans, laminarins and agar, and microbial gums such as the dextrans, phosphomannans such a USDA B-1459 and B-1428, and the gluconic acid microbial containing gums such as the USDA Y-1409 gums, and waterdispersable protein classes such as animal proteins such as hydrolyzed keratins, egg albumin, and vegetable proteins such as gluten and synthetic gums such as polyvinyl alcohol, polyoxyethylene and polyacrylamide No significant improvements in burn ring, burn duration or taste properties were noted with each of these additive types at usage levels of 5-10 percent of the reconstituted tobacco composition.

It is interesting to note that, in a formulation which was constant other than for the type of experimental burn additive, the use of hydroxyethyl cellulose (hydroxyethyl substitution only), ethylcellulose (ethoxyl substitution only) and the normal grade of ethylhydroxyethyl cellulose referred to by Rosenberg and Schmidt (combination of ethoxyl and hydroxyethyl 65 ployed in the use of reconstituted tobacco sheet: substitution on the same molecule) as burn additives resulted in no significant improvements or trends in the burn and taste results. Yet, the use of the ethylhydroxyethyl cellulose of this invention resulted in significantly different and beneficial burn and taste properties.

According to the present invention substantial burn improvement is obtained when EHEC with an ethoxyl D.S. of 1.2 to 1.6 and a hydroxyethyl M.S. of 0.5 to 1.2 is present at levels of 3 percent or more of the reconstituted tobacco composition higher usage levels, but it seems to be never necessary to use more than 15 percent of this EHEC to achieve optimum burn and taste characteristics.

Investigation of additives other than gums and adhesives were made. Inorganic and organic materials with known burnacceleration properties in other applications were tested in reconstituted sheet, including inorganic and organic nitrates and peroxides, and calcium or magnesium carbonates which have achieved widespread usage as burn additives in cigarette paper. Although some of these additives (e.g., potassium nitrate) resulted in acceleration of the free burn rate of the reconstituted sheet, when this sheet was converted into a smoking article (i.e., by using the reconstituted tobacco sheet as binder and/or wrapper on a cigar), no reduction in the width of the burn ring, no increases in the burn duration, and no taste improvements were noted. Accordingly, it was demonstrated that the problem of burn and taste improvement in shaped smoking articles containing reconstituted tobacco is complex and involves substantially more than just speeding up the free burn of a reconstituted tobacco component.

It was found, however, that although the addition of calcium carbonate or magnesium carbonate to a reconstituted tobacco composition had little effect on the significant burn and taste problems of the ultimate smoking article, when the additives were incorporated into compositions which also contained the particular EHEC of the present invention, a synergistic improvement in the burn taste properties of the ultimate smoking article resulted. For example, reconstituted tobacco compositions containing 4 percent EHEC plus 4 percent calcium carbonate resulted in somewhat better burn/taste properties in the ultimate smoking article than did a similar composition with 8 percent EHEC as the sole additive, and decidedly better burn/taste properties than the similar composition containing 8 percent calcium carbonate. Accordingly, it is seen that although calcium carbonate and/or magnesium carbonate as additives in reconstituted tobacco compositions are ineffective in enhancing the significant burn/taste properties of smoking products, their use in combination with the particular EHEC of this invention synergistically enhances the magnitude of the burn/taste improvements achieved wit the latter. It has been found that combinations of 2 to 10 percent of EHEC (ethoxyl D.S. 1.2-1.6; hydroxyethyl M.S. 0.5-1.2) with 45 2 to 10 percent calcium carbonate and/or magnesium carbonate, based on the dry weight of the total reconstituted tobacco composition, results in optimum burn and taste properties for the various smoking articles of interest. The optimum combination for each type of reconstituted sheet and 50 for each type of smoking article may be determined empirically by those skilled in the art.

It should be noted that the burn and taste advantages associated with the use of the particular EHEC of this invention are due to the degree of substitution of the ethoxyl and hydroxyethyl substituents, with no significant effect of the viscosity of gum on these properties. Of course, the higher the viscosity of the EHEC, the more it will function as an adhesive and the more it will contribute to the strength of the reconstituted tobacco product. However, even the lowest viscosity and lowest molecular weight grades of EHEC, which are poor film-formers and do not function to any extent as adhesives, will contribute the burn and taste advantages described above.

The following are examples of the present invention as em-

EXAMPLE I

A tobacco slurry is prepared in the conventional manner by adding, with agitation, one part (dry basis) of a 3.2 percent 70 dispersion of refined sulfite pulp to one part (dry basis) of a 2 percent aqueous dispersion of ethylhydroxyethyl cellulose with an ethoxyl D.S. of 1.2, a hydroxyethyl M.S. of 0.5 and a viscosity of 15,000 cps, and adding to the resultant dispersion 0.2 parts (dry basis) of a 50 percent aqueous solution of glyox-(dry basis). The burn improvement is even more substantial at 75 al and five parts (dry basis) of a 20 percent dispersion of

minus 120 mesh Wisconsin leaf tobacco dust. The tobacco slurry is then prepared and cast, also in a conventional manner, into a shaped form, namely a sheet, at a thickness such that the tobacco slurry film, after drying, will weight 3 grams per square foot. The cast tobacco slurry film is dried at 5 95°-100bLC.

The resultant reconstituted tobacco sheet containing 13.9 percent ethylhydroxyethyl cellulose (dry basis) was used as wrapper on a perfecto-shaped large cigar which also contained a natural Wisconsin tobacco leaf binder and chopped 10 Puerto Rican filler. The cigar, on combustion exhibited a burn ring only about one sixty-fourth inch in width while smoking, and only one thirty-second inch in width when allowed to rest in an ashtray for 4 minutes without puffing. The burn duration when the cigar was not being puffed was 14 minutes before the 15 cigar self-extinguished. The taste characteristic of the cigar was considered by a panel of smoking connoisseurs to be comparable to that of a natural wrapped control cigar. For comparison, the same cigar with a natural Connecticut shade wrapper had the same width of burn rings while smoking and 20 upon resting in an ashtray for 4 minutes. The burn duration was 12 minutes before the cigar self-extinguished.

EXAMPLE Ia

For comparison, the same shape and size cigar made with a reconstituted tobacco wrapper comparable to the one described, except for the use of 13.9 percent guar gum in place of the 13.9 ethylhydroxyethyl cellulose in the formulation, exhibited a burn ring of one-eight inch in width while smoking, and one-fourth inch in width when allowed to rest in an ashtray for 4 minutes without puffing. The burn duration when the cigar was not being puffed was 7 minutes before the cigar self-extinguished.

EXAMPLE II

A tobacco slurry is prepared by first adding one part (dry basis) of a 3.2 percent dispersion of refined sulfite pulp to 0.6 parts (dry basis) of a 2 percent aqueous dispersion of guar gum. To the resultant dispersion is added 0.4 parts (dry basis) of a 2 aqueous dispersion of ethylhydroxyethyl cellulose with an ethoxyl D.S. of 1.6, a hydroxyethyl M.S. of 1.2 and a viscosity of 300 cp.s., 0.2 parts (dry basis) of a 40 percent aqueous solution of glyoxal and 10 parts (dry basis) of a 20 percent dispersion of minus 120 mesh Wisconsin leaf tobacco dust. The tobacco slurry is cast and dried as in example I, and was evaluated as wrapper on a cigar comparable to that employed in example 1. The cigar exhibited a burn ring one thir-

ty-second inch while smoking and one-sixteenth inch in width when allowed to rest in an ashtray for 4 minutes. The burn duration when the cigar was not being puffed was 12 minutes before the cigar self-extinguished. The taste characteristic was considered comparable in quality to that of a natural tobacco wrapped cigar counterpart by a panel of cigar smokers.

EXAMPLE III

A tobacco slurry is prepared as described in example II. To this slurry is added with agitation 0.6 parts of a 10 percent aqueous suspension of calcium carbonate with an average particle size of about 0.5 microns. The resultant slurry is deaerated, cast and dried as in example II, and was evaluated as wrapper on a cigar comparable to that employed in examples I. and II. The cigar exhibited a burn ring one sixty-fourth inch in width while smoking, one thirty-second inch in width when allowed to rest in an ashtray for 4 minutes, and a burn duration of 16 minutes when the cigar was not being puffed.

The three examples given above are illustrative only of the application of the present invention to the production of reconstituted tobacco product. The prior art is complete with literature describing various methods of tobacco preparation, slurry formation, casting and drying. None of these prior art techniques is critical to the application of the present invention, and the present description omits lengthy explanation of apparata and methods relating thereto for the sake of brevity. It will thus be observed that novel, reconstituted tobacco

It will thus be observed that novel, reconstituted tobacco product is obtained within the scope of the objectives set forth previously.

What is claimed is:

- A tobacco composition having improved burn properties comprising finely divided tobacco and ethylhydroxyethyl cellulose with an ethoxyl D.S. of 1.2 to 1.6 and a hydroxyethyl M.S. of 0.5 to 1.2 and having a gel temperature of about 32° to 38° C.
 - 2. The composition according to claim 1 wherein said ethylhydroxyethyl cellulose comprises 3 to 15 percent by weight of the total composition.
 - 3. The composition according to claim 1 wherein said ethylhydroxyethyl cellulose comprises 2 to 10 percent by weight of the total composition and including 2 to 10 percent by weight of a material chosen from calcium or magnesium carbonate.
- 4. A smoking product made with the composition of claim
- 5. A smoking product made with the composition of claim 3.

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