

US 20090038629A1

# (19) United States(12) Patent Application Publication

# Ergle et al.

# (10) Pub. No.: US 2009/0038629 A1 (43) Pub. Date: Feb. 12, 2009

# (54) FLAVOR SHEET FOR SMOKING ARTICLE

 (76) Inventors: J. Dennis Ergle, Greensboro, NC (US); Michael A. Zawadzki, Greensboro, NC (US); Simon F. Yee, Greensboro, NC (US)

> Correspondence Address: MORGAN & FINNEGAN, L.L.P. 3 WORLD FINANCIAL CENTER NEW YORK, NY 10281-2101 (US)

- (21) Appl. No.: 11/835,092
- (22) Filed: Aug. 7, 2007

# **Publication Classification**

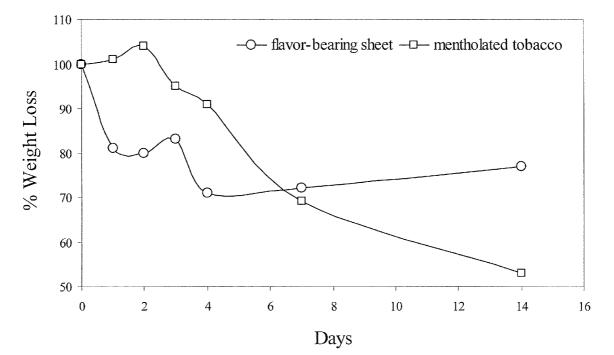
(51) Int. Cl.	
A24B 15/30	(2006.01)
A24B 15/00	(2006.01)
A24D 1/00	(2006.01)
A24D 3/06	(2006.01)

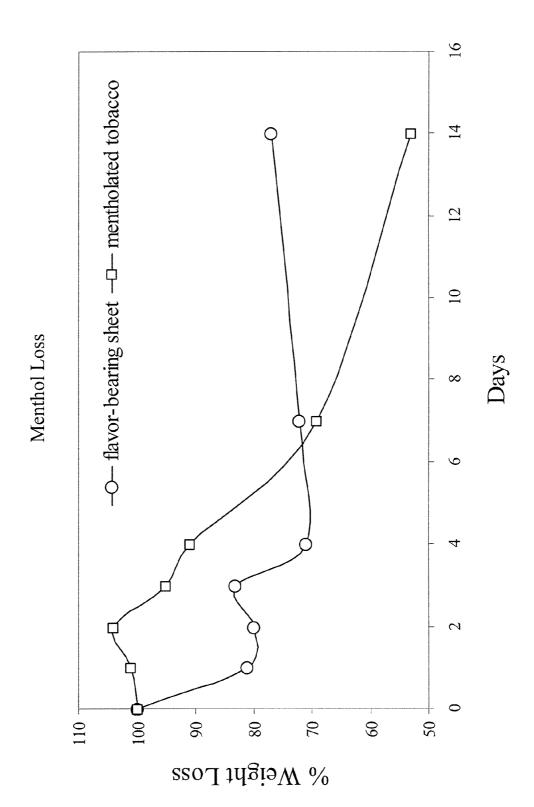
(52) U.S. Cl. ..... 131/276; 131/274; 131/331; 131/364

# (57) **ABSTRACT**

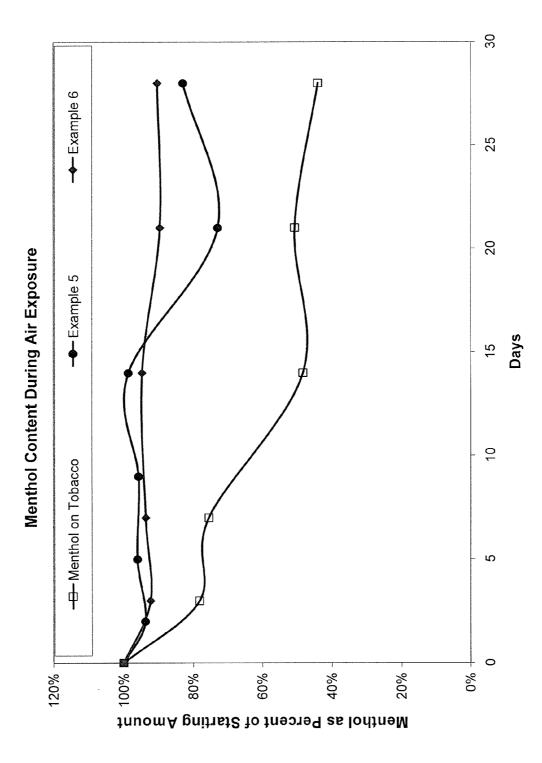
Flavor-bearing sheets for the controlled delivery of volatile flavorants in a smoking article are disclosed. The flavorbearing sheets comprise a non-volatile vitreous matrix of a film-forming coagulating material having one or more volatile flavorants dispersed therein.

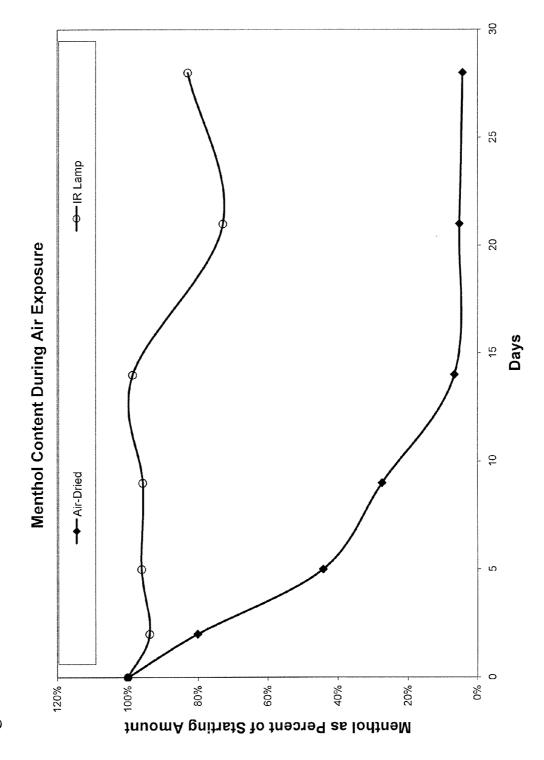


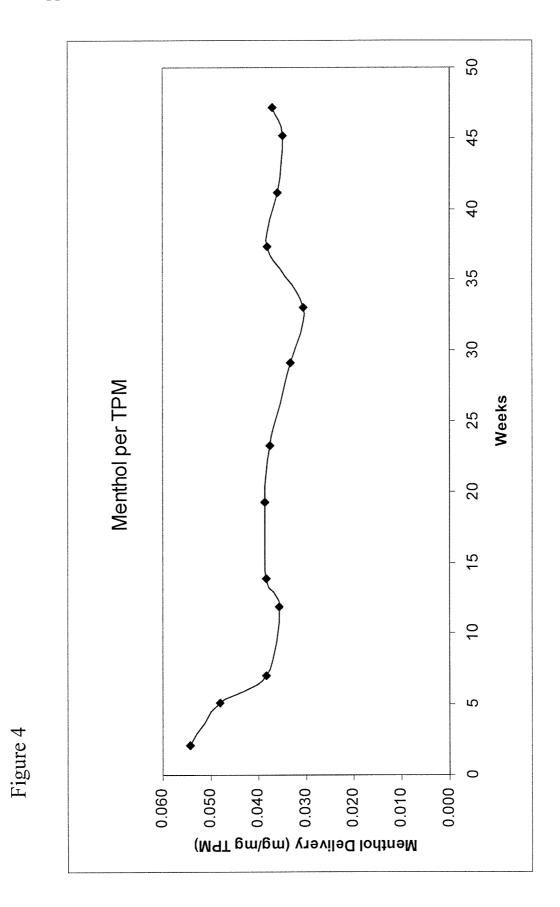


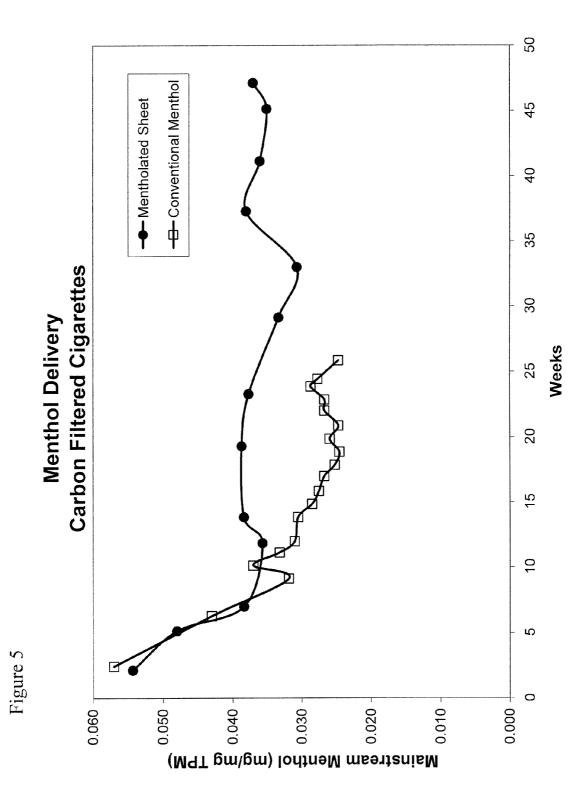




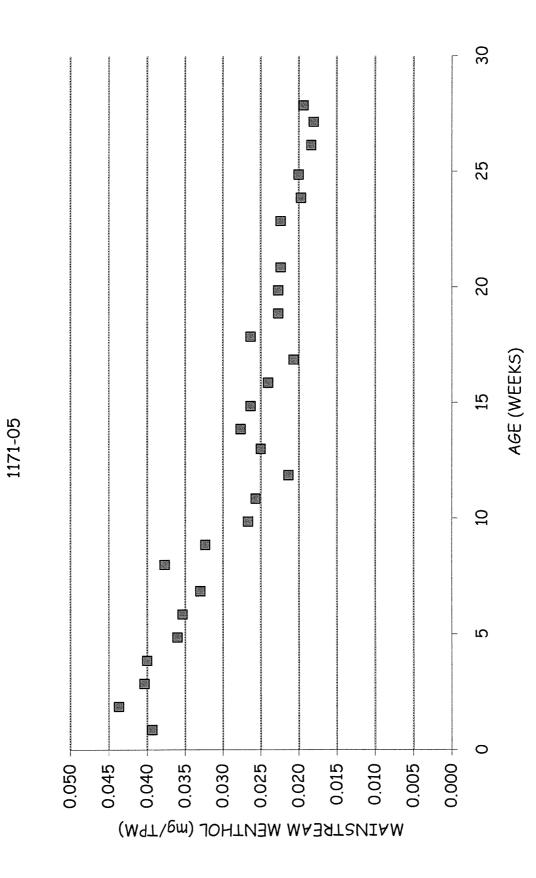


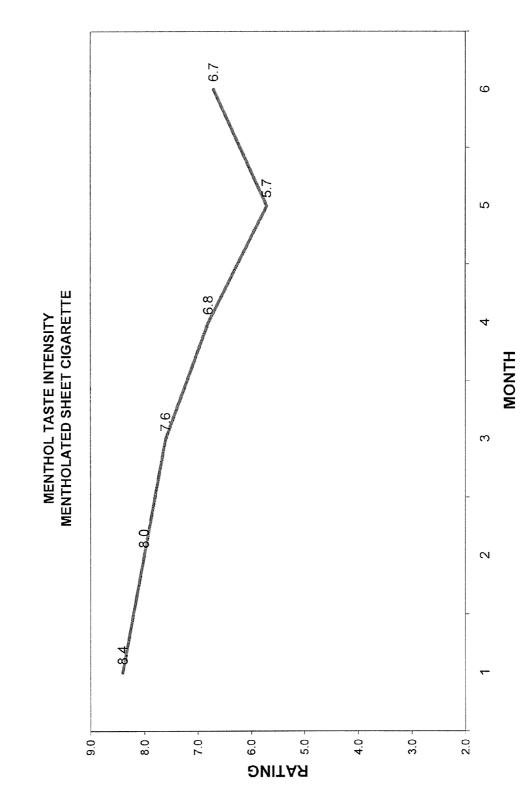












# FLAVOR SHEET FOR SMOKING ARTICLE

**[0001]** This application claims priority under 35 USC 119 (e)(1) of U.S. Provisional Patent Application Ser. No. 60/690, 759, filed Jun. 14, 2005, the entirety of which is incorporated herein by reference as if fully set forth herein.

# FIELD OF INVENTION

**[0002]** The present invention relates generally to compositions for delivering flavor to smoking articles. More specifically, the invention relates to flavor-generating compositions which reduce migration of volatile flavor constituents such as menthol from the tobacco column of a cigarette.

#### BACKGROUND OF THE INVENTION

[0003] The popularity of mentholated cigarettes has grown rapidly since their introduction in the United States in the 1920's. Today, mentholated cigarettes account for approximately one quarter of all cigarette sales in the United States. [0004] Conventionally, menthol has been incorporated into cut tobacco filler in mentholated cigarettes. Because menthol is a crystalline solid at room temperature, it is usually applied by spraying the filler with an ethanolic solution of menthol. Tobacco filler treated in this manner typically contains between 0.3 and 1.3% by weight menthol.

**[0005]** However, the amount of menthol ultimately delivered to the smoker has traditionally proven difficult to control. There are several factors that affect the menthol delivery of a cigarette. For example, much of the menthol in conventional mentholated cigarettes does not enter the mainstream smoke when the cigarette is smoked and is consequently not delivered to the smoker. Approximately, 75% of the menthol is lost to the sidestream smoke.

**[0006]** Loss of menthol on storage of mentholated cigarettes further reduces the amount of menthol ultimately delivered to the smoker. In fact, the menthol delivery of conventional mentholated cigarettes can be reduced by 57% after six months of storage in sealed cigarettes packages. Temperature and humidity conditions under which cigarettes are stored exert a substantial impact on menthol loss and consequently, significant differences in menthol delivery have been observed for the same brand of cigarettes in different regional markets.

**[0007]** The loss of menthol upon storage is due to the volatile nature of menthol which freely sublimes at room temperature. If menthol is initially located in the tobacco filler of a cigarette, substantial quantities will migrate to the packaging and atmosphere, during storage.

**[0008]** In addition, if the cigarette includes a filter, the menthol will also tend to migrate from the tobacco filler to the filter. The degree of migration of menthol to the filter depends on the characteristics of the components comprising the filter. Conventional cigarette filters are formed from fibrous material, such as cellulose acetate, that has been gathered into a plug (i.e., a filter "tow"). The tow is held together by a plasticizer, commonly triacetin, which has been applied to the fibers. Studies have demonstrated that up to 35% of the menthol initially added to the tobacco filler migrates to the filter tow within a few weeks of storage. Menthol may be associated strongly with the plasticizer and therefore be unavailable for delivery to the smoker.

**[0009]** Conventional cellulose acetate tow filters are designed to remove particulate matter from mainstream smoke but are ineffective to remove or reduce gas phase constituents. The gas phase of mainstream cigarette smoke

contains certain components alleged to be harmful to a smoker, including certain aldehydes and olefinic constituents. Filters have been designed for the removal of gas-phase constituents along with particulates. Such filters typically incorporate an adsorbent material such as activated carbon (also known as "carbon," "charcoal," or "activated charcoal") in a section of the filter. High surface area activated carbon is recognized as an effective adsorbent for removing gas phase components from mainstream smoke.

**[0010]** However, the use of activated carbon filters in mentholated cigarettes has met with only limited success to date. This is due largely to the fact that activated carbon is a very effective adsorbent of menthol. Thus, the greater part of menthol added to tobacco filler is irreversibly adsorbed by the activated carbon during storage and is therefore not available to be delivered to the smoker. For that reason, mentholated cigarettes having adsorbent filters traditionally have required larger amounts of menthol to be added to the tobacco filler during manufacture to offset adsorption by the carbon.

**[0011]** In light of the foregoing considerations, it is therefore an object of the present invention to provide cigarettes flavored with volatile flavorants, such as menthol, which limit the dissipation of volatile flavorant from the cigarette during storage.

**[0012]** It is another object of the invention to provide volatile flavorants, such as menthol, in a form which is compatible for use with adsorbent-bearing cigarette filters.

# SUMMARY OF THE INVENTION

**[0013]** In accordance with the foregoing objectives and others, the present invention provides compositions in the form of flavor-bearing sheets for the controlled delivery of volatile flavorants, such as menthol, to a smoker during smoking while reducing the migration of flavorant throughout the cigarette and packaging during storage.

[0014] In one aspect of the invention, a flavor-bearing sheet for the controlled delivery of volatile flavorants in a smoking article is provided. The flavor-bearing sheet comprises a nonvolatile vitreous matrix having a volatile flavorant, such as menthol, dispersed therein. The non-volatile matrix is provided by film-forming coagulating materials such as, for example, polyols, polymeric ethers, polymeric esters, natural polymers and derivatives thereof, and combinations thereof. The film-forming coagulating materials comprise between about 15 to about 80% by weight of the flavor-bearing sheet and the volatile flavorant comprises between about 20 to about 75% by weight of the flavor-bearing sheet. The flavorbearing sheets of the invention function to inhibit migration of volatile flavorants through physical entrapment of the flavorant in the low-vapor pressure matrix. The flavor-bearing sheets are dispersed in the tobacco column of a cigarette and release flavor into mainstream smoke upon combustion.

**[0015]** The flavor-bearing sheets of the present invention are particularly useful for cigarettes having filters which incorporate gas phase adsorbents, such as activated carbon, because the volatile flavorant is isolated from the adsorbent until the cigarette is smoked. In accordance with this aspect of the invention, a cigarette is provided comprising an adsorbent-bearing filter and a tobacco column abutting the filter, wherein the tobacco column includes a smokable material comprising shredded tobacco filler in admixture with a plurality of flavor-bearing sheets. In one implementation, the cigarettes include a so-called "plug-space-plug" filter having an adsorbent, such as granular activated carbon, disposed in a cavity formed between two fibrous filter components. Alternatively, the cigarettes may include so-called "dalmation" filter components wherein adsorbents such as activated carbon are dispersed in a fibrous tow material. The cigarettes of the present invention exhibit greatly diminished menthol loss on storage as compared to conventionally mentholated cigarettes. For example, the mainstream smoke menthol delivery of the cigarettes having flavor-bearing sheets according to the present invention may be greater than about 70% of its initial value after storage for about 25 weeks in a sealed cigarette package.

**[0016]** These and other aspects of the present invention will become apparent to those skilled in the art after a reading of the following detailed description of the invention, including the illustrative embodiments, examples and figures.

#### BRIEF DESCRIPTION OF THE DRAWINGS

**[0017]** The drawings are for the purpose of illustrating the invention and are not intended to be limiting.

**[0018]** FIG. **1** is a graph comparing the weight loss of menthol from shredded flavor-bearing sheets of the present invention prepared according to Example 1 and conventional mentholated tobacco.

**[0019]** FIG. **2** is a graph comparing the weight loss of menthol from shredded flavor-bearing sheets of the present invention prepared according to Examples 5 and 6 and conventional mentholated tobacco.

**[0020]** FIG. **3** is a graph comparing the menthol content of shredded flavor-bearing sheets dried using an infrared lamp and shredded flavor-bearing sheets dried ambiently.

**[0021]** FIG. **4** is a graph illustrating the mainstream smoke menthol levels for cigarettes having shredded flavor-bearing sheets prepared according to Example 1.

**[0022]** FIG. **5** is a graph comparing the mainstream smoke menthol levels for cigarettes having shredded flavor-bearing sheets prepared according to Example 1 and conventional mentholated cigarettes.

**[0023]** FIG. **6** is a graph of the mainstream smoke menthol content for cigarettes having shredded flavor-bearing sheets of the present invention prepared according to Example 11 after the cigarettes have been removed from sealed packages at weekly intervals of time.

**[0024]** FIG. 7 is a graph of the taste ratings of cigarettes having shredded flavor-bearing sheets of the present invention prepared according to Example 11 after the cigarettes have been removed from sealed packages at monthly intervals of time.

#### DETAILED DESCRIPTION

**[0025]** The present invention is founded on the discovery that migration of menthol and other volatile flavorants in a cigarette may be retarded by physically entrapping them in a non-volatile matrix which is capable of releasing the flavorant in response to heat or moisture generated upon combustion of the cigarette. The matrix takes the form of a vitreous (i.e., glassy) sheet which may be shredded and mixed with tobacco in the column of cigarette.

[0026] a. Flavor Sheets

**[0027]** The non-volatile matrix is provided by a film-forming coagulating material. While there is essentially no limitation on the selection of the film-forming coagulating material, it should be a material which is compatible with a smoking article, i.e., one which does not yield harmful products of combustion. Suitable materials include without limitation, polyols, polymeric ethers, polymeric esters, natural polymers and derivatives thereof, and combinations thereof. **[0028]** Useful polyols are exemplified by sugars and sugar alcohols, including, but not limited to, erythritol, glycerol, isomalt, mannitol, sorbitol, xylitol, maltitol, lactitol, hydro-

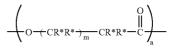
genated starch hydrolysate, dextrose, glucose, fructose, sucrose, maltose, galactose, lactose, inositol, corn syrup and the like. Glucamine, glucose glutamate, glucuronic acid, glycerin, 1,2,6-hexanetriol, hydroxystearyl methylglucamine, malitol, methyl gluceth-10, methyl gluceth-20, methyl glucose dioleate, methyl glucose sesquicarylate/sesquicaprate, methyl glucose sesquicocoate, methyl glucose sesqui sostearate, methyl glucose sesquilaurate, methyl glucose sesquistearate, phytantriol, riboflavin, sorbeth-6, sorbeth-20, sorbeth-30, sorbeth-40, and thioglycerin are also non-limiting examples of polyols which may be useful in the practice of the invention.

**[0029]** Polymeric ethers include, for example, the reaction products of alkylene oxides, represented by the general formula:

$$R - CH_2 - CH_1 - OH$$

wherein R and  $R_1$  are independently selected from hydrido and  $C_1$ - $C_{20}$  branched or straight chain alkyl and n is an integer greater than 2. Preferred polymeric ethers are polyalkylene glycols, such as polyethylene glycol (PEG) and polypropylene glycol (PPG). Copolymers of polymeric ethers, including for example, PEG/PPG copolymers, are also contemplated to be useful. Polymeric ethers also include alkoxylated alcohols such as polyoxyl 20 cetostearyl ether (Atlas G-3713), poloxyl 2 cetyl ether (ceteth-2), poloxyl 10 cetyl ether (ceteth-10), poloxyl 20 cetyl ether (ceteth-20), poloxyl 4 lauryl cetyl ether (laureth-4), poloxyl 23 lauryl cetyl ether (laureth-23), poloxyl 2 oleyl ether (oleth-2), poloxyl 10 oleyl ether (oleth-10), poloxyl 20 oleyl ether (oleth-20), poloxyl 2 stearyl ether (steareth-2), poloxyl 10 stearyl ether (steareth-10), poloxyl 20 stearyl ether (steareth-20) and poloxyl 100 stearyl ether (steareth-100), and the like.

**[0030]** Suitable polymeric esters include without limitation those represented by the general formula:



wherein each occurrence of R\* is independently selected from hydrido and  $C_1$ - $C_{20}$  branched or straight chain alkyl; m is an integer from 0 to 5; and n is an integer greater than 2. Such polymeric esters include the polyhydroxyalkanoates poly-3-hydroxybutyrate (PHB), poly(3-hydroxypropionate), poly(3-hydroxybutyrate), poly(3-hydroxyvalerate), poly(3hydroxyhectanoate), poly(3-hydroxyoctanoate), poly(3hydroxydodecanoate), poly(4-hydroxybutyrate), poly(5-hydroxyvalerate), and the like. Other interesting polymeric esters include polylactic acid (PLA), polyglycolide, and polycaprolactone (PCL).

**[0031]** Suitable natural polymers or natural polymer derivatives include without limitation starch and starch derivatives, including maltodextrin; cellulose and cellulose derivatives, including for example, methyl cellulose, ethyl cellulose, cellulose acetate, cellulose propionate, cellulose butyrate, cellulose acetate-butyrate, hydroxyethyl cellulose, hydroxypropyl cellulose, carboxymethyl cellulose, carboxyethyl cellulose, hydroxypropyl carboxymethyl cellulose, 3

hydroxypropyl methyl carboxyethyl cellulose, hydroxypropyl carboxypropyl cellulose, hydroxybutyl carboxymethyl cellulose, and alkali metal salts of these carboxyalkyl celluloses; alginate and alginate derivatives such as alginic acid, sodium alginate, potassium alginate, ammonium alginate, magnesium alginate, calcium alginate, sodium triethanolamine alginate, and propylene glycol alginate (hydroxypropyl alginate); and other vegetable gums including but not limited to carrageenan, dextran, furcellaran, pectin, gelatin, gum agar, locust bean gum, gum ghatti, guar gum, gum tragacanth, acacia, gum arabic, xanthan gum, karaya gum, and tara gum. [0032] The film-forming coagulating materials of the present invention stand in contrast to the heat-irreversible coagulating (i.e., gelling) glucans disclosed in U.S. Pat. Nos. 4,109,663, 5,778,899, 6,109,272, and 6,499,490, the disclosures of which are hereby incorporated by reference herein. Such heat-irreversible coagulating or gelling glucans are typically 1,3-\beta-glucans, as exemplified by curdlan. Unlike the heat-irreversible coagulating glucans, it is not necessary to apply heat to the cast emulsion in order to gel the matrix materials of the present invention. Second, in the practice of the present invention, it is desirable for the flavor-bearing sheets to release flavorant into the mainstream smoke prior to combustion of the flavor-bearing sheet in order to minimize loss of menthol to side stream smoke. This may be accomplished by any non-combustion release mechanism, whereby hot gases generated by the advancing fire cone induce decomposition of the flavor sheet. Without wishing to be bound by any theory, it is believed that water vapor produced by combustion of tobacco dissolves or decomposes the flavor-bearing sheets, thereby liberating the entrapped flavorant. This mechanism is not possible with the heat-irreversible coagulating glucans described above.

**[0033]** The film-forming coagulating materials typically comprise between about 15 to about 80% by weight, more typically between about 50 to about 80% by weight, and preferably between about 60 to about 80% by weight of the flavor-bearing sheet.

[0034] The flavor-bearing sheets comprise a flavorant. While it is contemplated that any flavorant will be useful, the advantages of the present invention will be most fully realized where the flavorant is a volatile flavorant. As used herein, the term "volatile flavorant" is intended to refer expansively to any flavorant which readily enters the gas phase through evaporation or sublimation at ambient temperatures. Thus, the term "volatile flavorant" is meant to specifically include, in addition to volatile oils, flavorants which are solid at room temperature but readily sublime, including for example menthol, camphor, vanillin and the like. Volatile flavorants suitable for flavoring tobacco smoke are well known in the art and include, without limitation, acetaldehyde, amyl acetate, anethole, anisole, benzaldehyde, benzylformate, 2,3-butanedione, butyraldehyde, camphor, 1-carvone, d-carvone, cinnamaldehyde, citral, citronellol, p-cresyl methyl ether, cymene, dihydrocarvone, dihydrocarveol, 2,4-dimethylacetophenone, dipropyl ketone, ethyl acetate, ethyl amyl ketone, ethylbutyrate, ethyl butyl ketone, ethyl valerate, ethyl vanillin, eucalyptol, eugenol, hexenal, geraniol, isoamyl isovalerate, limonene, linalool, menthol, menthone, 4-methyl benzaldehyde, methyl ethyl ketone, methyl hexyl ketone, methyl salicylate, 3-methyl valeric acid, pinene, d-piperitone, propylisobutyrate, pulegone, santalol, thujone, vanillin, zingerone and the like. Menthol is the preferred volatile flavorant.

**[0035]** The volatile flavorant will typically comprise between about 10 to about 75% by weight, more typically

between about 15 to about 50% by weight, and preferably between about 20 to about 30% by weight of the flavor-bearing sheet.

**[0036]** Illustrative flavor-bearing sheet compositions include without limitation:

- [0037] a. polyethylene glycol (about 70 weight %); menthol (about 30 weight %)
- [0038] b. polylactic acid (about 70 weight %); menthol (about 30 weight %)
- [0039] c. sorbitol (about 79 weight %); menthol (about 20 weight %); sodium dodecylsulfate (about 1 weight %)
- [0040] d. hydrogenated starch hydrolysate (about 69 weight %); menthol (about 30 weight %); propylene glycol alginate (about 1 weight %)
- [0041] e. sodium carboxymethyl cellulose (about 69 weight %); menthol (about 30 weight %); sodium dode-cylsulfate (about 1 weight %).

**[0042]** In addition to the volatile flavorant, the flavor-bearing sheets may optionally contain one or more nonvolatile flavorants. Such flavorants are well-known in the art and include, for example, cocoa, licorice, powdered tobacco, tobacco extract and the like. Volatile flavorants stabilized in microcapsules may also be present in the flavor sheets.

**[0043]** The flavor-bearing sheets may optionally comprise various additives such as fillers, emulsifiers, humectants, fragrances, colorants, burn rate modifiers and the like. In one interesting embodiment, the flavor-bearing sheets comprise an adsorbent material for removing gas phase components of mainstream smoke. Suitable adsorbent materials are described elsewhere herein.

[0044] The flavor-bearing sheets may be prepared from solutions or emulsions comprising the film-forming coagulating material, volatile flavorants, and a solvent. The solvent is typically a protic solvent such as water, ethanol, glycerin, and combinations thereof. The film-forming coagulating material and volatile flavorants may be added to the solvent in any order. It is desirable to apply efficient stirring or agitation to achieve a homogenous solution or emulsion. Stirring may be accomplished with any conventional mixer, including vertical mixers, planetary mixers, high-shear mixers and the like. Heating may optionally be employed to increase the solubility or dispersibility of poorly miscible components. Temperatures of about 40 to about 60° C., and preferably about 50° C. have been found suitable for this purpose. It may be useful to employ an emulsifier to improve the stability of resultant emulsions.

**[0045]** The mixtures are cast onto a substantially flat surface, such as a glass sheet, stainless steel belt or polystyrene block, to form a layer typically having a thickness of about 0.5 mm to about 3 mm, and preferably from about 0.5 mm to about 1.0 mm. It has been found desirable to employ a casting blade for this purpose. Solvent is removed by evaporation under ambient conditions to produce a dried sheet of the non-volatile vitreous matrix material having the volatile flavorant dispersed therein. Drying times will depend on the selection of solvent and drying method. For example ambient drying will typically range from about 8 hours to about 24 hours and force warm air drying (e.g., infrared lamp) will typically range from 5 to 45 minutes.

**[0046]** The flavor-bearing sheets formed from the layer of the menthol mixture have a thickness typically ranging from about 2 mil to about 5 mil. It will be understood that the final moisture content of the flavor-bearing sheet will depend on

the thickness of the flavor-bearing sheet as well as the atmospheric humidity. For example, a flavor-bearing sheet having a thickness of 5 mil may have a moisture content of  $15\%\pm5\%$ and a flavor-bearing sheet having a thickness of 3 mil may have a moisture content of  $10\%\pm2\%$ .

[0047] b. Smokable Materials

**[0048]** In one aspect of the invention, smokable materials are provided by shredding the flavor-bearing sheets described herein and mixing with shredded tobacco filler. The flavor-bearing sheets may be shredded with a paper shredder or the like to produce a plurality of shredded flavor-bearing sheets. The precise dimensions of the shredded flavor-bearing sheets are not critical. A confetti cut of about  $\frac{1}{32}$ " by about  $\frac{7}{16}$ " has been found to be useful. However, any other dimensions, such as, for example, a square cut of about  $\frac{1}{32}$ " by about  $\frac{1}{32}$ ", are also within the scope of the invention.

**[0049]** Alternatively, the flavor-bearing sheets may be pulverized to provide a powdered composition which can be added to shredded tobacco filler in essentially the same manner. In practice, it has been found less desirable to provide the compositions in the form of a powder due to the increase in total surface area from which menthol may migrate.

**[0050]** The shredded flavor-bearing sheets are mixed with tobacco filler using any conventional method, including, by way of example, tumbling in a rotating drum mixer. The weight ratio of shredded flavor-bearing sheets to tobacco filler will typically range from about 1:100 to about 1:5 depending on the desired level of menthol delivery. Preferably, the weight ratio of shredded flavor-bearing sheets to tobacco filler will be from about 1:10 to 1:20.

**[0051]** Any shredded tobacco may be used with the present invention. Examples of suitable tobaccos include, but are not limited to, flue-cured, Burley, Turkish, Oriental, expanded tobacco, and reconstituted tobacco. Other tobacco materials suitable for use in the present invention are described in U.S. Pat. No. 5,404,890 to Gentry et. al., the disclosure of which is hereby incorporated by reference. A preferred tobacco is low tobacco-specific nitrosoamine (low TSNA) tobacco. The tobacco may be treated with additives and the like according to conventional practice.

[0052] c. Flavored Cigarettes

**[0053]** In another aspect of the invention, flavored cigarettes are provided. Flavored cigarettes according to the invention typically comprise a paper-wrapped cylindrical column of tobacco. The tobacco column comprises shredded flavor-bearing sheets in admixture with cut tobacco filler, as described above.

**[0054]** It is contemplated that the present invention may be applicable to cigarettes having filters, as well as cigarettes without filters. With regard to cigarettes having filters, the filter may be attached at the mouth or buccal end of the tobacco column. The filter may be attached to the tobacco column using any method known in the art, such as a conventional tipping overwrap.

**[0055]** The filter may be any of various types of filters suitable for cigarettes. For example, the filter may comprises a plug (i.e., "tow") of filter medium capable of removing particulate material from mainstream smoke. The filter plug may comprise fibrous, webbed, and corrugated materials, formed from polyolefins, polyesters, cellulosics and the like. Cellulosics may include paper and cellulose acetate fiber. While such filters are effective for removing particulate material such as tar from mainstream smoke, they are ineffective for removing or reducing gas phase constituents of main-

stream smoke. Therefore, preferred filters will incorporate an adsorbent material, such as activated carbon or Sepiolite, in a section of the filter.

**[0056]** As used herein, the term "adsorbent" is intended to have its ordinary and accustomed meaning in the art, but should not be construed as limiting the invention to any particular mechanism or mode of action by which gas-phase constituents in mainstream smoke are reduced. For instance, in accordance with the ordinary and accustomed nomenclature in the art, activated carbon and Sepiolite are referred to herein as an "adsorbents," but carbons and minerals which reduce gas phase constituents by adsorption, absorption, chemisorption, or otherwise are contemplated as being within the scope of the invention.

**[0057]** Suitable adsorbent materials for use in the filters of the present invention include, but are not limited to, activated carbon, zeolite, magnesium silicates, aluminum silicates, silica gel, meerschaum, aluminum oxide, and florisil. Synthetic adsorbents such as, for example, carbonaceous resins derived from the pyrolysis of sulfonated styrene-divinylbenzene and sold under the trademarks Ambersorb 572 or Ambersorb 563 (Rohm and Haas, 5000 Richmond Street, Philadelphia, Pa. 19137) may also be employed.

[0058] Activated carbon adsorbents are preferred. Any activated carbon material may be used in the practice of the invention, including but not limited to carbon materials derived from coal, tobacco material, peat, wood pulp, coconut hulls, kapok fibers, cotton fibers, cotton linters, and the like. Activated carbon materials of any degree of activation (surface area) may be used according to the present invention. Preferably, the activated carbon materials will have a degree of activation so as to provide about 25 to about 125 weight percent pickup of carbon tetrachloride. More preferably, the activated carbon material will provide about 60 weight percent pickup of carbon tetrachloride. Any mesh size activated carbon is useful in the practice of the invention. However, larger mesh size activated carbons may provide advantages during the manufacture of the cigarette, particularly in those embodiments of the invention having a filter cavity which is charged with a bed of activated carbon. Preferred activated carbons are granular coconut carbons with a mesh size of about 18×40 U.S such as coconut hull based carbons available from Calgon Corp. as PCB, PCGB and GRC-11 and those available from PICA USA (Columbus, Ohio) as G278.

**[0059]** Certain minerals are also preferred adsorbents in the practice of the invention. Suitable minerals include, for example, the hydrated magnesium silicate Sepiolite (TOLSA, S.A.) and the hydrated aluminum silicate Attapulgite (TOLSA, S.A.). Sepiolite and Attapulgite belong to the palygorskite family of minerals. They are lightweight, porous clays having a large specific surface and low chemical activity.

**[0060]** In one embodiment, the filter comprises a so-called "dalmation" filter component. A dalmation component comprises a fibrous filter material, such as cellulose acetate, which has been impregnated with an adsorbent. Typically, the fibrous filter material is treated with a plasticizer, such as triacetin, followed by dispersion of the adsorbent particles into the fibrous material. Dalmation filter components, and their method of manufacture, are well known in the art as described in U.S. Pat. Nos. 6,257,242 B1 (Stpyridis), 5,622, 190 (Arterbery et al.), 5,568,819 (Gentry et al.), 3,101,723 (Seligman et al.), the disclosures of which are hereby incorporated by reference.

**[0061]** It is also known to incorporate adsorbents, such as activated carbon, into paper filter components. Accordingly, the filter may be provided as, for example, a cellulose acetate tow having a carbonaceous paper gathered within the tow or wrapped concentrically around the tow, as described in U.S. Pat. No. 5,568,819 (Gentry et al.), the contents of which are hereby incorporated by reference.

[0062] In another embodiment, the filter is provided in a so-called "plug-space-plug" configuration. In this embodiment, the filter will comprise a first particulate filter component at the buccal end of the filter and a second particulate filter component abutting the tobacco rod. The particulate filter components may be formed from any material suitable for removing particulates from mainstream smoke, as described above. The first and second particulate filter components are spaced apart to form a filter cavity therebetween. The filter including all three components is circumscribed by a conventional paper wrapper. The filter cavity will contain a bed of filter material comprising a gas phase adsorbent, such as granular activated carbon. The cavity is preferably filled to a 90% full condition, and more preferably to at least a 95% full condition, with the adsorbent filter medium. The amount of adsorbent should be selected to achieve the most effective gas phase reduction within the limits of the smoking article. Preferably, the cavity is filled with about 125 mg to about 150 mg of activated carbon. In one interesting embodiment, the cavity is charged with activated carbon and Sepiolite in weight ratio ranging from 0:100 to 100:0.

**[0063]** The cigarettes of the invention are not limited to any dimension. Typical cigarettes are cylindrically shaped rods having circumferences of about 22 mm to about 25 mm. The cigarette may be any length, including but not limited to, 80 mm, 84 mm, and 99 mm. In a preferred embodiment, the cigarette is 84 mm long and the downstream tow is 10 mm in length. The cavity ranges from about 3 mm to about 8 mm in length, and is preferably between about 5 mm and about 6 mm in length. In an embodiment where the cavity is 5 mm in length, the upstream tow is 10 mm in length. In another embodiment, the cavity is 6 mm in length and the upstream tow is 9 mm in length.

**[0064]** In another preferred embodiment, the cigarette is 99 mm long and the downstream tow is 10 mm in length. The cavity ranges from about 3 mm to about 8 mm in length, and is preferably between about 5 mm and about 6 mm in length. In an embodiment where the cavity is 5 mm in length, the upstream tow is 12 mm in length. In another embodiment, the cavity is 6 mm in length and the upstream tow is 11 mm in length.

**[0065]** Ventilation may be provided by one or more circumferential rows of perforations through the tipping paper. The perforations may be located between the upstream and downstream ends of the filter cavity containing the adsorbent. Ventilation is preferably provided in the filter cavity by perforations located 14.5 mm from the buccal end of the downstream tow. As is well-known, ventilation reduces the amount of mainstream smoke reaching the smoker through dilution by ambient air and also tends to increase filtration efficiency by decreasing the velocity of mainstream smoke upstream of the perforations and thereby increasing its residence time in the filter.

**[0066]** Examples of commercially available adsorbent filters include Caviflex, Dualcoal, Recessed Dualcoal, Sel-X-4, and Triple Filter from Baumartner Fibertec (Switzerland); Active Acetate Dual, Active Charcoal Triple Solid, Active Myria White, Active Patch Mono, Adsorbent Coated Thread, Triple Granular, and V.P.A. Dual from Filtrona International Incorporated (Milton Keynes, U.K.), each of which are contemplated to be useful in the practice of the invention.

**[0067]** The wrapping papers and tipping papers used in the practice of the invention may be any of the papers known in the art, including low-sidestream paper and reduced ignition propensity paper such as those disclosed in U.S. Pat. No. 6,837,248 to Zawadzki et al., which is hereby incorporated by reference.

**[0068]** The present invention is not limited to the filter designs described above. It is contemplated that other filter arrangements are suitable for use with the present invention, including but not limited to those described in European Patent Application No. 579,410 and U.S. Pat. Nos. 5,568,819 (Gentry et al.), 5,365,951 (Arterbery et al.), 5,067,499 (Banerjee et al.), 4,881,556 (Clearman et al.), 4,357,950 (Berger et al.), 3,894,545 (Crellin et al.), which are hereby incorporated by reference. It will be appreciated by one skilled in the art that certain modifications and variations of the above described embodiments are within the scope of the invention. **[0069]** The following non-limiting examples are provided to illustrate various embodiments and attributes of the present invention. Examples 1-4 demonstrate various embodiments of the flavor bearing sheet of the present invention.

#### EXAMPLE 1

**[0070]** A flavor-bearing sheet according to the present invention is provided by mixing 100 g of carboxymethyl cellulose with 900 g of water using an electric overhead stirrer to yield a 10% by weight aqueous carboxymethyl cellulose solution. To the solution was added 100 mg of sodium dode-cylsulfate and the mixture was warmed to 50° C. 25 g of menthol was added and the mixture was stirred until the menthol was melted and homogenously dispersed into the solution. The mixture was allowed to cool to room temperature with continued stirring. The mixture was then cast onto polystyrene blocks using a casting blade set to 1.5 mm thickness and dried overnight under ambient conditions. The resultant sheet was separated from the polystyrene block and shredded to a confetti cut of about  $\frac{1}{32}$ " by about  $\frac{7}{16}$ " with a paper shredder.

#### EXAMPLE 2

**[0071]** Another embodiment of a flavor-bearing sheet of the present invention is provided by slowly adding 11 g of carboxymethyl cellulose to a mixture of 190 g of water, 1 g of insoluble cellulose, and 0.5 g of caramel coloring. The mixture was continuously stirred at 500-1000 rpm for about 30 minutes until the carboxymethyl cellulose dissolved resulting in a brown opaque viscous mixture without lumps.

**[0072]** Separately, a menthol solution consisting of 9.5 g of propylene glycol, 2.4 g of glycerol, 11.9 g of menthol, and 0.09 g of polysorbate 80 were stirred together until dissolved. The opaque viscous carboxymethol cellulose mixture was placed in a warm water bath at about 50° C. and the menthol solution was added. The mixture was stirred at 500-1000 rpm until creamy (about 15 minutes). The mixture was cast onto glass plates coated with cooking spray at 1.0 mm thickness and dried in oven at 90° C. for 45 minutes.

# EXAMPLE 3

**[0073]** A further embodiment of a flavor bearing sheet of the present invention is provided by stirring 1 g of an

insoluble cellulose with 112 g of water at 300 rpm for 10 minutes. 11 g of pectin powder was then stirred into the mixture before it was heated to 185° F. The pectin mixture was removed from the heat and the viscous liquid was allowed to cool to room temperature with stirring.

**[0074]** Separately, a menthol mixture consisting of 7.2 g of propylene glycol, 1.8 g of glycerol, 9 g of menthol, and 0.13 g of polysorbate 80 was stirred together until dissolved. The menthol solution was stirred into the pectin mixture at 500 rpm in a warm water bath at 50° C. for 20 minutes. The mixture was cast onto glass plates coated with cooking spray at 1.0 mm thickness and dried in oven at 90° C. for 45 minutes.

#### **EXAMPLE 4**

**[0075]** In another embodiment of the a flavor bearing sheet of the present invention 5 g of sodium alginate and 2 g of Gum Arabic were sprinkled into a stirred solution of 95 g of water and 4 g of propylene glycol and stirred until the solids dissolved. 4 g of solid menthol and 0.35 g of sodium dodecyl-sulfate were added and stirred into the mixture in a warm water bath at 50° C. for 20 minutes. The mixture was cast onto glass plates coated with cooking spray at 1.0 mm thickness and dried in oven at 90° C. for 30 minutes.

**[0076]** The following example demonstrates the improvement in film quality of the flavor bearing sheet of the present invention by adding propylene glycol.

# EXAMPLE 5

**[0077]** Five flavor bearing sheets were prepared having the ratio of ingredients as set forth in the following table. In each of these sheets 0.10 g of sodium dodecylsulfate was used as the emulsifier and the sheets were dried under an infrared lamp.

Sample	Propylene Glycol	Menthol	Carboxymethyl Cellulose
Sheet 1:	5%	30%	65%
Sheet 2:	15%	30%	55%
Sheet 3:	25%	30%	45%
Sheet 4:	35%	30%	35%
Sheet 5:	45%	30%	25%

**[0078]** Sheet 1 exhibited visible streaks of crystallized menthol in the finished sheet and on the surface while Sheet 2 had less. Sheets 3, 4, and 5 exhibited no evidence of crystallized menthol in the sheets.

**[0079]** The following example demonstrates the improvement in film quality of the flavor bearing sheet of the present invention by adding glycerol, and the improvement in the taste quality of cigarettes made with such flavor bearing sheet.

#### EXAMPLE 6

**[0080]** Flavor bearing sheet 1 was prepared by dissolving 12 g of carboxymethyl cellulose into a solution of 108 g of water and 9 g of propylene glycol. When the carboxymethyl cellulose was dissolved (after about 30 minutes), the mixture was stirred in a warm water bath where 9 g of solid menthol and 0.1 g of sodium dodecysulfate were added to the mixture. The mixture was stirred about 15 minutes until the menthol melted to yield a creamy mixture. The mixture was cast onto

a polystyrene plate at 1.0 mm thickness and dried under an infrared lamp for 30 minutes, then air-dried until dry to the touch.

**[0081]** Flavor bearing sheet 2 was prepared by dissolving 12 g of carboxymethyl cellulose into a solution of 108 g of water, 6 g of propylene glycol, and 3 g of glycerol. When the carboxymethyl cellulose was dissolved (after about 30 minutes), the mixture was stirred in a warm water bath where 9 g of solid menthol and 0.1 g of sodium dodecysulfate were added to the mixture. The mixture was stirred about 15 minutes until the menthol melted to yield a creamy mixture. The mixture was cast onto a polystyrene plate at 1.0 mm thickness and dried under an infrared lamp for 30 minutes, then airdired until dry to the touch. It was observed that flavor bearing sheet 2 was more flexible than flavor bearing sheet 1.

**[0082]** The prepared flavor bearing sheets were cut and blended with tobacco to make cigarettes. Three out of four panelists preferred the taste of cigarettes having flavor bearing sheet 2 containing glycerol compared to the taste of cigarettes having flavor bearing sheet 1.

**[0083]** Examples 7-9 provide stability studies of various embodiments of flavor bearing sheets of the present invention described in the Examples above to evaluate their ability to retain menthol when exposed to atmospheric conditions.

# EXAMPLE 7

**[0084]** Samples of shredded flavor bearing sheets of Example 1 were weighed into vials and left open to ambient conditions for 14 days. Control samples of mentholated tobacco prepared by spraying tobacco with an ethanolic solution of menthol were similarly weighed into open vials. The control samples were obtained from freshly prepared Newport® brand cigarettes. Menthol content of the shredded flavor bearing sheet samples and control samples was determined by measuring weight loss after 1, 2, 3, 4, 7, and 14 days exposure to ambient conditions. The results are plotted in FIG. 1 as the percentage loss of menthol based on the initial weight.

[0085] As illustrated in FIG. 1, about 20% of the menthol was lost from the shredded flavor-bearing sheets over the first day followed by a rapid stabilization in menthol loss. Without wishing to be bound by any theory, it is believed that the initial menthol loss might be explained by the migration of loosely encapsulated menthol at or near the surface or the sheet. After loss of loosely encapsulated menthol over the first day, only about 10% additional menthol is lost over the next 13 days, with a total menthol loss after 14 days of about 25%. In the control samples, a very slight increase in weight was observed over the first three days. It is believed that this effect may arise due to the hydroscopic nature of dried tobacco which allows it to absorb water from the air, thereby preventing the menthol loss to be accurately measured until the tobacco moisture levels establish equilibrium with ambient water over the first three days. Thereafter, the weight loss in the control samples rapidly accelerates to yield a total weight loss of about 50% after 14 days.

#### EXAMPLE 8

**[0086]** Flavor bearing sheets as prepared in Examples 5 and 6 were shredded. Twenty samples of each flavor bearing sheet were weighed into vials (40 mg each) and left open to allow menthol to evaporate. Mentholated tobacco from a commercial product was likewise weighed into 20 vials. Three vials

from each sample were extracted and evaluated for menthol content weekly for 4 weeks. As shown in FIG. **2**, the mentholated sheets showed superior retention of menthol when compared to conventional mentholated tobacco.

# EXAMPLE 9

**[0087]** This example demonstrates the improvement in the retention of menthol in the flavor bearing sheet by using an infrared lamp to quickly dry the flavor bearing sheet before separation of a hydrophobic flavor from the aqueous medium.

**[0088]** In this example, a flavor bearing sheet according to the present invention was prepared by slowly admixing 22.5 g of carboxymethyl cellulose to a solution of 0.9 g of propylene glycol, 0.1 g of sodium dodecylsulfate (0.1 g), and 150 g of water stirred at 500 rpm. The mixture was stirred an additional 30 minutes until the carboxymethyl cellulose dissolved to yield a clear viscous liquid. The mixture was placed in a warm water bath at 50° C. while 7.5 g of solid menthol was added. The mixture was stirred warm for 15 minutes at which time the menthol had completely dispersed into a creamy emulsion.

**[0089]** About one-half of the mixture was cast onto a polystyrene plate at 0.75 mm thickness and allowed to dry ambiently overnight. The resulting sheet had a rough texture on the top surface with a noticable amount of crystalline menthol on the surface of the film.

**[0090]** The other half of the mixture was cast onto a polystyrene plate at 0.75 mm thickness and dried under an infrared lamp to speed the drying of the film. The resulting sheet was smooth on both surfaces and had very little evidence of crystalline menthol at the surface.

**[0091]** As shown in FIG. **3**, the flavor sheet sample that was dried quickly by the infrared lamp proved to be superior compared to the air dried flavor sheet sample with regard to the retention of menthol over a 4 week period.

**[0092]** Examples 10 and 11 provide stability studies of menthol in a shredded flavor bearing sheet included in the tobacco rod of a cigarette.

#### EXAMPLE 10

**[0093]** A smokable composition was prepared by mixing 2.20 pounds of the shredded flavor-bearing sheets of Example 1 with 25.0 pounds of cut tobacco for four revolutions in a rotating drum. Cigarettes having plug-space-plug filters were prepared with the resultant mixture. The cigarette filters had a 10 mm plug of fiber tow at the buccal end, a 9 mm plug of fiber tow at the upstream end and a 6 mm cavity charged with 150 mg of sepiolite granules. The mean menthol content of each cigarette was 7.52 mg with a Relative Standard Deviation of 10.0%. As shown in FIG. **4**, after 47 weeks in a sealed package, the menthol levels in the mainstream smoke of the cigarettes was greater than 70% of the initial value.

**[0094]** FIG. **5** shows a comparison of the menthol levels in the mainstream smoke of the cigarettes of Example 10 having shredded menthol sheets of the present invention prepared according to Example 1 and conventional carbon filter cigarettes having mentholated tobacco prepared by spraying tobacco with an ethanolic solution of menthol. As illustrated in FIG. **5**, after about 10 weeks in sealed packs, the menthol levels in the mainstream smoke of the cigarettes having the shredded mentholated sheets is superior to that of the conventional cigarettes having the ethanolic menthol solution sprayed on the tobacco.

#### EXAMPLE 11

[0095] A flavor bearing sheet according to the present invention is provided by dissolving 48 g of carboxymethyl cellulose in a solution of 3.6 g propylene glycol and 352 g water with mechanical stirring. 20.5 g of solid menthol was added to the viscous liquid and the mixture was stirred in a warm water bath until all of the menthol had melted. 0.4 g of sodium dodecylsulfate was added and the mixture was stirred vigorously at 800 rpm for 1 minute. The warm water bath was exchanged for a cool water bath and the mixture was stirred for 10 minutes to bring the mixture to room temperature. The mixture was cast onto PTFE-coated glass plates at a thickness of 1.0 mm and dried under an infrared lamp for 30 minutes. The sheets were then placed under a fan until dry to the touch, and shredded to  $\frac{1}{32}$ "× $\frac{7}{16}$ " strips using a paper shredder. In this manner, 3500 g of the mentholated sheet was prepared. [0096] The shredded sheet was loaded into a metered solids addition funnel and the discharge was directed into the tobacco flow of a cigarette maker. The mentholated sheet was added at 60.0 g/min while the maker speed was set to 1100 cig/min to give an average content of 54 mg sheet per cigarette. The cigarettes were packed and evaluated weekly for menthol delivery. The menthol delivery in the mainstream smoke is plotted in FIG. 6. In addition, the cigarettes were evaluated monthly for taste. As shown in FIG. 7, the menthol taste intensity of the cigarettes was deemed to be acceptable for at least six months after packaging.

**[0097]** Certain modifications and improvements will occur to those skilled in the art upon a reading of the foregoing description. It should be understood that all such modifications and improvements have been deleted herein for the sake of conciseness and readability but are properly within the scope of the following claims.

We claim:

1. A flavor-bearing sheet for the controlled delivery of volatile flavorants in a smoking article, said flavor-bearing sheet comprising a non-volatile vitreous matrix having one or more volatile flavorants dispersed therein, said non-volatile matrix comprising a film-forming coagulating material; wherein said film-forming coagulating material comprises between about 15 to about 80% by weight of said flavor-bearing sheet and said one or more volatile flavorants comprise between about 20 to about 75% by weight of said flavor-bearing sheet.

2. The flavor-bearing sheet of claim 1 wherein said filmforming coagulating material comprises a material selected from the group consisting of polyols, polymeric ethers, polymeric esters, natural polymers and derivatives thereof, and combinations thereof.

**3**. The flavor-bearing sheet of claim **2** wherein said polyol is selected from the group consisting of erythritol, glycerol, isomalt, mannitol, sorbitol, xylitol, maltitol, lactitol, inositol, hydrogenated starch hydrolysate, dextrose, glucose, fructose, sucrose, maltose, galactose, lactose, corn syrup and combinations thereof.

**4**. The flavor-bearing sheet of claim **2** wherein said filmforming coagulating material comprises a polymeric ether selected from the group consisting of polyethylene glycol (PEG), polypropylene glycol (PPG), PEG/PPG copolymers, alkoxylated alcohols and combinations thereof. **5**. The flavor-bearing sheet of claim **2** wherein said filmforming coagulating material comprises a polymeric ester selected from the group consisting of polylactic acid (PLA), poly-3-hydroxybutyrate (PHB), polyglycolide, polycaprolactone (PCL), and combinations thereof.

6. The flavor-bearing sheet of claim 2 wherein said filmforming coagulating material comprises a natural polymer or natural polymer derivative selected from the group consisting of cellulose, ethyl cellulose, methyl cellulose, hydroxypropyl cellulose, carboxymethyl cellulose, hydroxypropyl alginate, and combinations thereof.

7. The flavor-bearing sheet of claim 1 further comprising an emulsifier.

**8**. The flavor-bearing sheet of claim **1** wherein said one or more volatile flavorants comprises menthol.

**9**. The flavor-bearing sheet of claim **8** wherein the loss of menthol upon storage in ambient conditions for 100 days is less than about 25% by weight based on the initial weight of menthol.

**10**. A smokable composition comprising a plurality of flavor-bearing sheets according to claim 1 dispersed in shred-ded tobacco filler.

11. A cigarette comprising a tobacco column, said tobacco column comprising the smokable composition of claim 10.

**12**. The cigarette of claim **11** further comprising a filter abutting said tobacco column, wherein said filter comprises an adsorbent material which reduces at least one gas phase constituent of mainstream smoke.

**13**. The cigarette of claim **12** wherein said adsorbent material comprises activated carbon.

14. The cigarette of claim 12 wherein said filter comprises:

 (i) an upstream fibrous filter component and a downstream fibrous filter component, said upstream and downstream fibrous filter components defining a filter cavity therebetween; and (ii) a filter material disposed in said filter cavity, said filter material comprising an adsorbent material which reduces at least one gas phase constituent of mainstream smoke.

**15**. The cigarette of claim **12** wherein said filter comprises a fibrous filter component having an adsorbent material which reduces at least one gas phase constituent of main-stream smoke dispersed therein.

16. The cigarette of either claim 14 or 15 wherein said adsorbent material comprises activated carbon.

17. A cigarette comprising:

(a) a tobacco column including a smokable material comprising:

(i) shredded tobacco filler; and

(ii) a plurality of flavor-bearing sheets in admixture with said tobacco filler, said flavor-bearing sheets comprising a non-volatile vitreous matrix having menthol dispersed therein, said non-volatile matrix comprising a film-forming coagulating material selected from the group consisting of polyols, polymeric ethers, polymeric esters, natural polymers and derivatives thereof, and combinations thereof;

wherein said menthol is released into the mainstream smoke upon combustion of said smokable material.

18. The cigarette of claim 17 further comprising a filter abutting said tobacco column, wherein the filter comprises an adsorbent material.

**19**. The cigarette of claim **18** wherein said adsorbent material comprises activated carbon.

**20**. The cigarette of claim **17** wherein the mainstream smoke menthol delivery of said cigarette is greater than about 70% of its initial value after storage for about 25 weeks in a sealed cigarette package.

\* \* \* \* \*