ABSTRACT: The formation of reconstituted tobacco sheet material from tobacco stems, said sheet having a subsident layer of microencapsulated flavoring material embedded in the reconstituted tobacco sheet and the incorporation thereof into smoking articles such as cigarettes, cigars, etc.

The microcapsules are of an average diameter smaller than the sheet thickness and in such quantity as to be confined within the sheet, being protectively nested in the spaces between the tobacco fibers. They are incorporated within an aqueous tobacco slurry before sheeting or applied to a wet web of fibrous tobacco on a support in a suspension which contains a film forming polymeric material.
1 PROCESSES FOR INCORPORATING ENCAPSULATED FLAVORS AND THE LIKE IN RECONSTITUTED TOBACCO SHEET

This is a division of U.S. Pat. application Ser. No. 660,775, filed Aug. 15, 1967.

This invention relates to an improvement in reconstituted tobacco processing whereby a plurality of minute, heat rupturable capsules each containing an aromatic substance such as menthol as an inner phase are mixed in integral contiguous relationship with tobacco. As tobacco surrounding each discrete capsule burns, the elevation in temperature is such that capsule walls rupture, thereby releasing a discrete quantity of aromatic substance which becomes entrained in smoke issuing from the burning tobacco. The capsules being spaced homogeneously throughout the length of the smoking article, the capsule walls rupture successively, or consecutively, with the advance of the burning front of ignited tobacco. The ex- eunt smoke inhaled by the smoker is homogeneously aromatic.

This invention relates to methods for producing reconstituted sheet or film material having capsules contained therein of encapsulated flavoring material.

This invention relates to a system for providing a substantial layer of microcapsules containing flavoring material in reconstituted fibrous tobacco sheets; and to the novel product produced thereby.

The present invention can be practiced by spraying a capsule slurry onto reconstituted tobacco sheet material or film made from a selected portion of the stems of tobacco. It has been found that although tobacco stems tend to burn with acidity, the acidity is masked or nullified by the flavoring of smoke from the burning of said tobacco stems with encapsulated odoriferous flavors such as menthol, lime, lemon, peach, pineapple, cherry, spearmint, peppermint and kola.

It has been recognized that the addition of aromatic substances to tobacco imparts a pleasant flavor to the tobacco. In methods hitherto used the tobacco has been treated by soaking it in a suitable solution of the aromatic substance, or the solution has been sprayed on the tobacco. One disadvantage of such a process is that most aromatic substances which are suitable for this use have an appreciable vapor pressure at ordinary temperatures and for that reason the substance more or less rapidly vaporizes and escapes. In the process of the invention a wide variety of aromatic substances may be used and when encapsulated in microcapsules the substances do not vaporize and escape.

A means of overcoming the acidity of reconstituted tobacco sheets made from stems of tobacco by including flavor containing capsules on or under the surface of the sheet material, is to mix the capsules with the pulp prior to the formation of the sheet, or to otherwise generally distribute the capsules through the body of the sheet. With such a technique in reconstituted tobacco sheet manufacture, the capsules would hopefully and at best be distributed throughout the sheet or web. However, efforts to form the sheet material by mixing the capsules and the tobacco fibers, without more, have been unsatisfactory due to the enormous percentage of escape and loss of capsules through drainage from the embryonic reconstituted tobacco sheet or web.

The subject invention provides a system for the penetration of capsule material applied to a wet fibrous tobacco sheet which affords the advantages inherent in having said capsules disposed within the body of the sheet material and yet accomplishes this end without concomitant intolerable loss of the capsules to achieve it.

In the tobacco industry, and particularly that part of the industry relating to the manufacture of cigarettes, cigars, and like smoking articles, large quantities of tobacco are rejected annually and sold as waste because it is either impossible or impracticable to utilize them in the manufacture of smoking articles. Obviously the loss of these materials is a serious matter from the point of view of the manufacturers and, therefore, it would be highly desirable if such material could be salvaged and used in the manufacture of smoking articles. Materials now disposed of as waste are tobacco stems, dust, scrap, clippings and other portions which are unsatisfactory in connection with the manufacture of high-grade smoking articles.

From time to time attempts have been made to form tobacco sheets or films which would solve the problem of satisfactorily disposing of or utilizing tobacco waste materials. Processes have been developed for forming tobacco paper utilizing well-known paper processes and techniques in which tobacco waste, such as for example stems and scraps, has been reduced to pulp by the use of chemicals, and then according to ordinary paper making techniques converted into paper. These techniques are unsuitable for use either in connection with the present invention in forming improved tobacco sheet material, carrying out the processes therefor, and producing novel smoking articles, such as cigarettes or cigars, because of the radical changes to which these tobacco materials are subjected, and the loss of valuable constituents and properties of natural tobacco.

According to this invention, tobacco sheet or film material may be made along lines similar to those described and disclosed in U.S. Pat. No. 2,433,877, in which the final product contains substantially all of the desirable properties and natural characteristics of tobacco, especially insofar as aroma, taste and color are concerned, and has the same general appearance of tobacco.

Another object of this invention is to coat reconstituted tobacco sheet material with capsules containing aromatic material such as menthol, lime, lemon, peach, pineapple, cherry, spearmint, peppermint, and kola.

Another object of this invention is to prevent premature rupture of capsules coated on the surface of reconstituted tobacco sheets by providing a substantial layer of capsules within a reconstituted tobacco sheet such that said adjacent capsules are protected from abrasive rupturing forces, in smoking article manufacture, transport, and storage.

Another object of this invention is to provide flavor containing reconstituted tobacco sheet material in which the flavor is invisible and undetectable prior to smoking said reconstituted tobacco sheet material.

Another object of this invention is to provide a novel method of making reconstituted tobacco sheet which has the advantage of physical protection of capsules containing flavoring from touch and sight. To the end that the particles (capsules) may be confined with the sheet, they are of necessity much smaller in their average diameter size than the sheet thickness and preferably are of such size and present in such quantity, that they are protectively nested in the interstitial spaces between the tobacco fibers of the reconstituted tobacco support sheet, in addition to being adherent to the tobacco fiber, even if calendered, without disturbing, from a user's viewpoint, the normal physical structure of such a sheet made without them.

The instant invention encompasses the method of forming continuous, self-supporting reconstituted tobacco films or sheets which comprises forming an aqueous slurry of comminuted tobacco stems, forming said slurry into a continuous thin web, and applying to said web microscopic capsules each containing an inner phase comprising flavors such as menthol, lime, lemon, peach, pineapple, cherry, spearmint, peppermint, and kola, and removing moisture from said web.

The tobacco stems, veins, and tobacco leaf material can be comminuted by means of a suitable type of mill, such as a ball mill or colloid mill. Any desired ratio of tobacco material to water can be used. It has been found that ratios of 1.8 to 1.14 give satisfactory results. When the milling or slurry forming operation is completed, the slurry is deposited in a film forming device and applied in the form of a continuous film or web upon a suitable film forming surface, such that when the slurry is spread out in the form of a film or web thereon, substantially all soluble and insoluble constituents of the tobacco stems and veins will be contained in a reconstituted tobacco sheet or
3,540,456

film. The tobacco film can be coated with encapsulated flavoring material while in either wet or dry condition.

It is not necessary to rupture the capsules contained in a tobacco smoking article since heat from a burning front of tobacco will rupture the capsules, and the resulting aerosol thereby released may be any aromatic material such that the smoke emanating from the tobacco smoking article is homogeneously flavored.

One of the features of the instant invention is the saving in volatile material effected by encapsulation. The evanescence of the volatile materials is greatly inhibited by the intact capsule wall.

A desirable result obtained by this invention is the formation of a reconstituted tobacco sheet in which the cross-sectional area is permanently increased as a result of the included capsules so that if cigarettes are formed from this material, either wholly or from admixtures with natural shredded tobacco, there is a definite increase in filling power of reconstituted capsule-containing material. Due also to the increased area and decreased thickness of those portions of the sheet or web which contain capsules, the ash formed when this material is burned approximates more closely the ash of natural tobacco and the odor is more nearly that of natural tobacco although a large portion of the tobacco material in the sheet or film consists of stems.

This invention includes the application of one or more other sets of capsules throughout the thickness of the reconstituted tobacco sheet or on the outside of the sheet, and it does not preclude the presence of other material, particulate or other, present in, or on the outside of, or otherwise associated with the sheet, to cooperate, or not, with the set of capsules positioned in the sheet by the practice of this invention cumulatively or countervailing, which produce a beneficial effect. There may be two or more sets of capsules in the sheet being confined or not confined to the surface, or spread throughout the thickness of the sheet.

It is contemplated that the embedded particles (capsules) need not be of uniform substance, content, and size. When the particles are capsules, they may be a mixture as to size and wall material or of different content or origin, to serve one or more utilitarian purposes in behavior, or, if capsules, to have walls of varying resistance to rupture or fracture, or to be different in any other aspect of structure or performance (such as resistance to heat) or different in any olfactory sense characteristics—or to contain chemical reactants, medicines, perfumes—absorbs or absorbs—between the fracture and perforated state or in either structure of energy in various manners which will be evident to those skilled in the art. Each of the capsules may be self-contained, in that each, upon rupture, releases its volatile content. The particulate liquid may contain either liquid or solid solute material dissolved therein, or have such materials dispersed therein in any physical state, and may contain materials which may be subject to growth, metamorphosis, or degeneration.

To obtain uniformity among capsules if such are used they may be manufactured in separate batches according to the characteristics of the capsules as to origin, size, contents, or wall material and structure, and thereafter blended in the desired proportions for introduction into the paper as a single applied slurry. This is important where more than one kind of capsule contents (solid or fluid) is to be made use of either by rupture of the capsules or by their characteristics in the unruptured state. It is within the province of this invention to provide capsules having different thicknesses of walls or different strengths of walls, to the extent that they may be differentially fractured by variations in temperature, so that some capsules may be ruptured by one temperature and the rupture of other capsules will follow in response to application of a greater temperature.

In a preferred embodiment, though not limited thereto, a novel system is capable of limiting the penetration of capsules containing flavoring material into a reconstituted tobacco sheet as the sheet is being formed, so that the particles are substantially concentrated subsident to either surface thereof in a firm, well defined stratum of binder material.

The capsules are, preferably, capable of containing an aqueous slurry containing a solvent-responsive or solvent-activated adhesive such as a vegetable gum or starch. Efficacious natural adhesives or binders include hydrolyzed starches (especially tapioca starch), gum arabic, gum tragacanth. These adhesives or binders are used exclusively in water solution. (While karaya gum is not actually soluble in water, karaya gum particles absorb large amounts of water and swell to great size). Certain water soluble synthetic adhesives or binders such as polyvinyl alcohol, sodium cellulose glycolate, and methyl cellulose can be used to bind capsules in a reconstituted tobacco web.

The capsules preferably should contain an oily, aromatic liquid comprising about 90 percent, by weight, of the encapsulated structure. The oily liquid is released by the rupturing of the capsule wall material. The oily liquid may be a mixture of oils or dispersions of solids in oils. The aggregation of capsules can contain disparate substances within each discrete capsule.

The process of this invention may be employed to form smoking articles containing two disparate encapsulated substances which act synergistically when the capsules containing them are ruptured. The relative proportions of solid particles to capsules may vary according to the effect desired.

The capsule walls can be made of a gelatin-gum arabic complex, gelatin-carrageen complexes, phattened succinylated gelatins, ethyl cellulose, or other material. Any standard method of encapsulation such as the one set forth in example 1 may be used to form the capsules.

The size of the capsules can range from approximately 5 microns in diameter to approximately 40 microns in diameter, and may be mixed as to size, or even mixed as to content. Capsules spaced in the cigarette should not exceed 40 microns in diameter, since larger capsules tend to rupture with a loud report.

Capsules of the 5 to 40 micron size can be made according to the process disclosed in U.S. Pat. No. 2,800,437, which issued on July 23, 1957 to Barrett K. Green and Lowell Schleicher. Briefly, that patent discloses film-forming polymeric materials of opposite electric charge in aqueous solution which are caused to form a separate liquid dispersed phase (a coacervate) by modifying the conditions of the solution.

The intended oil contents of the capsules are emulsified in the solution, and the coacervate forms a liquid film about the emulsion. The film is made firm and solid, and is then hardened. The capsules are recovered ready for their use as part of the heat rupturable composition. The oily material of the preferred embodiment (e.g., a solution of menthol) may be replaced in whole or in part by the previously mentioned or other materials of aromatic or therapeutic properties.

In the accompanying drawings, which illustrate a preferred embodiment of the invention, and form a part of this specification, and wherein the several reference characters designate the same or like elements:

FIG. 1 is a diagrammatic view showing mechanism suitable for carrying out and performing the method;

FIG. 2 is a leaf of tobacco having capsules applied to the surface thereof;

FIG. 3 is a cross-sectional view of a sheet of reconstituted tobacco having capsules in a subsident layer therein;

FIG. 4 is a view of a cigarette made from filler of reconstituted tobacco containing a subsident layer of capsules each containing an inner phase of aromatic flavoring material;

FIG. 5 is a sectional view of a cigar having a binder formed of reconstituted tobacco sheet material having a subsident layer of microcapsules each containing an inner phase of aromatic flavoring material, and a composition tobaccoumor fill;

FIG. 6 is an end view of a cigarette made from filler of reconstituted tobacco containing a subsident layer of capsules each containing an inner phase of aromatic flavoring material.
As mentioned hereinabove, the reconstituted sheet which is made in accordance with this invention, can be used either in the manufacture of cigars, cigarettes or other like smoking articles. In the following description, no distinction is made between the types of tobacco employed. In making sheet or film material to be expanded and made available for use in the manufacture of smoking articles, a suitable slurry can be formed from 75-percent stems, which are run through a conventional comminuting machine in order to reduce the stems to extremely fine size after which they are mixed with water, and 25-percent tobacco scrap leaf portions, dust or fines which have also been passed through the comminuting machine and preferably screened to remove any foreign material such as sand mixed therewith. The mixture of comminuted stems and leaves is then placed in a ball mill of known design, and ball milled for a period of time, preferably sufficient to completely hydrate or gelatinize the particles of tobacco being ball milled. The size of the ball mill and balls, and speed of rotation, control the amount of time necessary to reach the desired state of hydration or gelatinization of the tobacco particles. For instance, it has been found that in 1-nm nominal size flint pebbles, the ball milling time may approximate from 6 to 16 hours at 50 r.p.m. whereas in a 5-quart ball mill using the same size pebbles at 60 r.p.m., the minimum time may run as high as 13 hours.

Upon completion of the slurry, it is removed from the ball mill and placed in a film forming device and formed into a continuous sheet or web. The sheet or web material is then ready for the addition of capsules in a slurry. Mechanism suitable for forming films is shown diagrammatically in Fig. 1. In this mechanism there is provided a conventional type of film forming unit, designed generally 2, which delivers the sheet material in the form of an endless web 4 onto a traveling conveyor 6 running on pulleys 8, 10 mounted on a driving and driven shaft 12, 14, respectively. Conveyor 6 preferably is imperforate and is made in the form of an endless band from material, such as stainless steel or other nonrusting and nonstaining material. In this way there is no danger of staining or discoloring the sheet or web being formed thereon.

Furthermore, the use of an imperforate surface substantially prevents the loss of valuable insoluble constituents of tobacco. It will be seen, therefore, that at all stages in the process of making the reconstituted tobacco sheet care is taken not to lose from the aqueous colloidal dispersion or from the cast sheet films any of the natural soluble ingredients or constituents of tobacco and that these soluble constituents are re incorporated in the finished sheet and in substantially the proportions in which they occur in the component tobacco after evaporation of the excess moisture present in the aqueous hydrated tobacco dispersion. A primary objective of the invention is to provide processes for making reconstituted tobacco sheet with limited penetration of liquid-containing capsules as the particles that, because of their position in the sheet, when ruptured by heat, release the volatilized flavoring liquid. This objective is accomplished by forming a sheet of reconstituted tobacco with the capsule contained in it. The amount of capsules applied should be related to the final sheet thickness and the amount of encapsulated liquid necessary or desirable to be provided.

Considering now the application of the subject invention to the production of sheet material, the preferred system comprises laying down a wet web of tobacco fibrous material on a supporting member to form an embryonic sheet, and applying to said wet web minute capsules, the contents of which comprise flavoring material. Following the formation of the sheet or web, it is moved through a moisture removing zone and excess moisture is removed therefrom. In Fig. 1 there is shown a hood designated generally 16 through which hot air can be delivered against the surface of the web or sheet material to remove the desired amount of moisture therefrom. This may be all or only a part depending upon the condition of the sheet or web.

It should be understood that the foregoing description of Fig. 1 is merely illustrative. It is within the scope of the invention to add the capsules by any means at any point in the tobacco web formation provided that the web at the time of addition of the capsules 21 is sufficiently wet and penetrable, either by reason of undrained liquid or because the capsular particulate material was added as a liquid slurry, to permit fixation on the tobacco fibers. Thus, while the preferred procedure, as shown in Fig. 1, calls for the addition of the capsules 21 at 22, the capular slurry might be added at another point, again provided that the web of tobacco sheet material allowed sufficient penetration of the capsular material into the tobacco web.

Following are examples of systems that produce a controlled penetration of capsules within a reconstituted tobacco sheet or web.

**EXAMPLE I**

The following is a description of the preferred embodiment of the invention as carried out using the process steps and apparatus shown in Fig. 1, wherein minute capsules were added to a wet web of reconstituted tobacco. The capsules and binder materials, when placed among the tobacco fibers, wet them and entangle with them and coat them, thus in effect securing and binding the capsules against migration through the sheet, thereby forming a subsident stratum. The majority of binder and associated capsules are caught in the sheet. Substantially no capsules migrate through the sheet. When the wet tobacco web is dried, the binder shrinks by loss of solvent, leaving the dried polymeric binder material, and the capsules remain in place relatively with respect to sheet thickness. The apparatus and the procedure for adding the components were as shown in Fig. 1.

Tragacanth gum solution and starch solution were prepared in the following manner:

**Concentrated Tragacanth Gum Solution**

Four and five-tenths pounds of dry tragacanth gum powder was stirred into 50 gallons of water, using a suitable mixer. Five minutes after all the powder had been added, the mixer was turned off. The tragacanth gum solution was allowed to sit for 2 hours, and then the mixer was turned on for 5 minutes. After sitting for two additional hours, which period enabled the tragacanth gum to hydrate. After 5 minutes, the mixer was turned off, and the 55-gallon drum was covered. Just prior to combining the tragacanth gum solution and the capular slurry, 50 gallons of tragacanth gum solution was diluted with water to 3 percent tragacanth gum on a solids basis.

**Starch Solution**

The hydrolyzed starch solution was prepared by heating a slurry of the starch at 195°F for a minimum of 15 minutes to provide a 1 percent by weight, starch-in-water solution.

**Preparation of Capsular Slurry**

**Encapsulation of Lemon Oil**

Ten grams of gum arabic were dissolved at room temperature in 200.0 grams of deionized water. The mixture was agitated until the gum arabic was fully dissolved. In a separate 250 milliliter Erlenmeyer flask, 10.0 grams of modified gelatin was mixed with 200.0 grams of deionized water. The gelatin was allowed to thicken at room temperature and also then warmed in a water bath to about 40°C with stirring so that the gelatin was dissolved.

The gelatin solution and the gum arabic solution were poured into a beaker equipped with a stirrer. A flocculence indicating the precipitation of the gelatin was noted. The tem-
perature of the mixture was decreased to 35°C. The speed of the stirrer was adjusted so that it was turning only enough to keep the phases mixed. The pH of the mixture was 4.2.

Into the beaker containing the mixture of gum arabic and gelatin was poured 118.0 grams of lemon oil. The speed of the stirrer was then adjusted to mix the colloids and the oil. The oil separated into droplets. Two drops of octyl alcohol were added to prevent foaming. The progress of the coacervation was monitored by means of microscopic examination.

The temperature of the mixture was lowered to room temperature, i.e., 24°C. At the higher temperature of 31°C, colloid deposition was observed on the oil droplets. At 24°C, little colloid could be observed in aqueous portions of the mixture. Deposition had ceased. Stirring was continued for 30 minutes, whereupon the reaction mixture was cooled on an ice bath to 4°C. The reaction mixture was maintained at this temperature for 200 minutes. (When hardening was desired, 1.0 milliliters of a 25 percent glutaraldehyde in water per gram of gelatin is added.)

After stirring, a mass of capsules thus formed were refrigerated to 4°C. and maintained at that temperature for a time interval of 12 to 16 hours. After refrigeration, the cold capsule mass was placed in a separatory funnel and undeposited colloid material allowed to separate to the bottom of the vessel whereupon it was withdrawn and discarded. The drying of the capsules formed supra was effected by first rinsing the capsules with cyclohexane. The cyclohexane was decanted. Thirteen and three-tenths grams of finely divided, hydrated calcium silicate was mixed with cold denitized water to form a thick, mobile slurry. The slurry thus formed was stirred into the mass of capsules. Cold denitized water was added as needed to form a viscous, yet fluid base. The paste thus formed was poured into a Buchner funnel and vacuum filtered; the top of the funnel being sealed with a sheet of rubber. When no further water was observed being compressed from a filter cake thus formed, the filter cake was placed on a clean, absorbent paper and allowed to dry under ambient laboratory conditions.

An alternative drying method comprises adding 200.0 grams of cold hexylene glycol for each 100.0 grams of moist, rinsed, capsules. The capsules are mixed with the hexylene glycol. The hexylene glycol is drained from the capsules through the pores of a 200 mesh sieve. The foregoing was repeated twice. After the third alcohol rinse, however, the hexylene glycol was removed by Buchner funnel filtration. After the filtration was complete, the capsules were placed on a clean, absorbent paper and dried at ambient laboratory temperature.

The internal phase of the capsules thus formed was approximately 80-90 percent of the total weight of the capsules.

Variation of the Encapsulation of Lemon Oil

A variation of the encapsulation procedure set forth immediately supra is shown below:

The solution of gum arabic was warmed to 38°C, placed in a Waring blender and stirred. The lemon oil was added gradually while the speed of the blender was being increased until the size of the lemon oil droplets was approximately 2 to 5 microns. The mixture thus formed was poured into a 1.000 milliliter beaker containing gelatin, also at 38°C., and was stirred thoroughly. The temperature was then allowed to drop to room temperature and then further decreased to a temperature of 4°C to 10°C, by means of an ice bath. The capsules thus formed were dried in the procedure set forth above.

Encapsulation of Kola Flavor

The procedure for the encapsulation of lemon oil, set forth supra, was followed except that the inner phase was kola flavor, the droplet size of said kola flavor being 10 microns. The hardening agent for the capsule walls was a 25-percent solution of glutaraldehyde in water.

A further aspect of the subject invention concerns the nature of the fibrous tobacco sheet material resulting from carrying out the process of this invention. The sheet of the invention differs from earlier fibrous sheets in that the penetration of the particulate material is substantially uniform. In the usual fibrous sheet, though attempts are made to achieve a uniform laydown of fiber, this is generally difficult to obtain, and, for this reason, penetration of capsules in a uniform stratum is impossible of attainment without the agency of this invention. In the subject invention, it has been found that the capsules penetrate over the area applied, to a substantially uniform degree through a thickness of the tobacco web to form a subsident layer therein.

Referring again to FIG. 1, shaft 12 can be driven continuously at a rate of speed required to remove either all or only a part of the moisture contained in the web.

Obviously conveyor 6 can be driven stepwise if desired. The partially dried web or sheet material is delivered by conveyor 6 onto conveyor 15 which in the embodiment selected for purposes of illustration is also an endless band consisting preferably of stainless steel or other heat resistant material.

Conveyor 15 runs upon pulleys 17, 19 mounted on shafts 26, 28. Shaft 28 is driven in timed relation with driving shaft 12 so that the movements of the conveyors 6 and 15 are at the same rate of speed. It is preferred that the movement of conveyors 6 and 15 be continuous because it is believed that it results in a more efficient handling of the web or sheet material being treated. In this manner the web material 4 advancing past the capsule addition 22 is gradually heated.

The speed of conveyor 15 is governed in part by the thickness and moisture content of web 4. If a web is, say, one-thousandth of an inch in thickness and contains say 5-percent moisture, it is necessary to run conveyor 15 faster than when the web being treated is, say four-fourths-of an inch thick and contains the same amount of moisture. Speed of conveyors 6 and 15 is thereby adjusted in accordance with web thickness and moisture content. If conveyor 15 is driven at a faster rate of speed than conveyor 6, provisions can be made in any known manner for controlling movement of web 4 from one conveyor to the other. It is also necessary to take into consideration the temperature and proximity of heat source to the web.

As the capsule containing sheet material 5 is moved by conveyor 15 from the heat treating zone, lengths are cut therofrom by a cutting mechanism consisting of a rotary knife 23, and coating roller 27 operated in timed relationship with the movement of conveyor 15. The lengths can then be packed for storage or they may be used in the formation of cigar filler or binders. If the capsule-containing tobacco sheet material is to be used as a cigarette tobacco filler, it can be shredded by any known means and added in desirable quantities to form a desired cigarette tobacco blend.

FIG. 2 shows a tobacco leaf with capsules 21 adherent to the surface thereof.

FIG. 3 disclosed a cross-sectional view of a sheet of reconstituted tobacco 5 containing capsules 21 and tobacco material.

FIG. 4 discloses a cigarette designated generally 40, in which the conventional wrapper 42 encloses a filler consisting of shredded cigarette tobacco 44 and shredded capsule containing reconstituted tobacco filler material 46, or capsule containing reconstituted tobacco filaments 46 in any desired proportion. It is evident that the tobacco film or filaments can be made from various types and combinations of tobacco. For instance, the tobacco sheet material can be made from relatively expensive tobacco such as Latakia in which it is highly desirable to use all waste because of the high price thereof. So, also, it may be formed of Burley or one or more scrap or waste cigarette-type tobaccos and incorporated in accordance with a particular cigarette manufacturer's formula as if it were a natural cigarette tobacco leaf. Any desired formula can thus be maintained in accordance with the demands of a manufacturer's particular brand using one or more types of natural
shredded tobacco leaves and admixed desired quantities of shredded capsule containing tobacco film material or filmments, either as a blending or flavoring medium or both or for purposes of bulkling.

FIG. 5 shows a representation of a cigar 50, which may be a long or short filler cigar depending upon the kind of filler used. Cigar 50 has a wrapper 52, a binder 54 formed from capsule containing tobacco sheet or film material and a composite filler consisting of long or short filler tobacco 56, and a desired quantity of pieces of capsule containing tobacco sheet material or film 58 of desired size employed for blending and/or bulkling or natural tobacco filler 56 alone. The amount of capsule containing tobacco used depends upon the types of tobacco employed in making the reconstituted tobacco sheet, and the requirements of a particular blend. If desired, instead of a binder formed of tobacco film material, such as 58, a conventional type of tobacco leaf binder may be used. FIG. 6 shows a cigar in which the filler consists entirely of long or short filler reconstituted tobacco sheet material containing capsules 60, and a natural leaf binder 62.

The invention described may be varied in construction within the scope of the claims, for the particular device, selected to illustrate the invention, is but one of many possible concrete embodiments of the same. It is not therefore to be restricted to the precise details of the structure shown and described.

EXAMPLE II
Encapsulation of Menthol

Three-thousand seven-hundred and fifty cubic centimeters of a 60-percent solution of oil of menthol in water and 297 grams of gelatin in 5,403 cubic centimeters of water was stirred in a dispenser until the particle size was in the range of 5 microns to 40 microns (75 minutes stirring). A solution of 297 grams of gum arabic in 5,403 cubic centimeters of deionized water was then added. The pH of the resulting mixture was adjusted to 4.8 by the addition of a 10-percent solution of sodium hydroxide in water. Six-thousand cubic centimeters of water was added at a temperature of 38°C. The mixture was slowly cooled on a water bath at a temperature of 38°C. One-hundred and forty-eight and five-tenths cubic centimeters of a 25-percent solution of glutaraldehyde in water was added. Approximately 1 gram of gelatin (additional) was added and the resulting mixture was stirred for 12 hours. Seven and seven-tenths grams of methyl parahydroxybenzoate was added. The resulting capsule slurry was stirred for 30 minutes and then filtered using a No. 30 sieve.

The capsule mass was allowed to stand until the capsules can be decanted from the excess colloid. The capsules were washed twice with cold deionized water and that colloidal material separating from the capsule being decanted. A slurry was prepared by mixing 400 parts by weight of finely divided hydrated silica in 250.0 parts by weight of deionized water. The capsules were suspended in the slurry, then the slurry mass was filtered over a Buchner filter. The resultant filter cake was broken up and allowed to dry for 24 hours.

The capsules were suspended in a mixture of 3 parts gum tragacanth and 97 parts water and the resulting suspension was sprayed on tobacco leaves. (See FIG. 2.)

EXAMPLE III
Encapsulation of Menthol-Peppermint Oil Slurry

Eighteen parts by weight of spray dried gum arabic was dissolved in 384.5 parts by weight of deionized water in a beaker equipped with a stirrer. In a separate 250 milliliter Erlenmeyer flask, 18.0 parts by weight of gelatin was dissolved in 364.5 parts by weight of deionized water.

Fifty-eight and five-tenths parts by weight of menthol crystals and 136.5 parts by weight of peppermint oil were added to the gum arabic solution. The temperature of the mixture was adjusted to 33°C. Thereafter, the solution of gelatin was added with rapid stirring. The reaction mixture was cooled to a temperature of 10°C. On an ice bath while continuously stirred. The mixture was stirred for one hour at 10°C.

Thereafter, 18.0 parts by weight of a 25-percent solution of glutaraldehyde in water was added to the reaction mass. The reactant mass was stirred at 10°C for 8 hours. The capsules thus formed were dried in the manner set forth in example II. The capsules which were sprayed onto cut tobacco leaves exhibited an inner phase of menthol-peppermint oil slurry.

EXAMPLE IV

The procedure of example II was followed except that the menthol was substituted with a menthol-methane slurry.

The mixture in the inner phase of the capsules was comprised of 136.5 parts by weight of menthol crystals and 58.5 parts by weight menthone.

EXAMPLE V

The procedure of example II was followed except that the menthol of example II was substituted with a menthol-peppermint slurry.

EXAMPLE VI

The procedure of example II was followed except that spearmint oil was substituted for the menthol oil of example II.

EXAMPLE VII

The procedure of example II was followed except that a lime oil was substituted for the menthol oil of example II.

The solution for the inner phase was comprised of 195.0 parts by weight lime oil and 0.975 parts by weight butylylated hydroxy anisole.

In the case of the manufacture of cigarettes, according to the present invention, tobacco films are shredded into strands or the film is formed directly into filmments substantially the width of the strands of natural shredded tobacco and of any desired length, in the case of cigars, the capsule-containing films are used in large pieces much as long filler tobacco in forming long filler cigars or in smaller pieces for use in the formation of short filler cigars. In all cases the shredded films or filmment or film used in cigarettes and cigars can be handled either manually or by machine in the same manner as natural shredded tobacco leaves or whole leaves or portions thereof.

The amount of shredded capsule-containing reconstituted tobacco or pieces of this material employed in a particular blend in cigarettes or cigars, respectively, will vary according to types of tobacco used in the sheet material and the requirements of a particular manufacturer.

We claim:
1. A method of forming continuous self-supporting reconstituted tobacco sheets which comprises comminuting tobacco-co stems and tobacco leaves, forming an aqueous slurry from said comminuted tobacco stems and tobacco leaves, forming said slurry into a continuous web, adding an aqueous slurry of tragacanth gum and a plurality of minute rigid-walled, heat-rupturable capsules, each containing an inner phase of a flavoring material, said capsules having an average diameter smaller than the thickness of the sheet and preferably of such size and in such quantity that they are protectively nested in the interstitial spaces between the tobacco fibers, and removing moisture from said web.

2. A process for limiting the penetration of microcapsules through an embryonic reconstituted tobacco sheet, comprising forming a wet web of fibrous tobacco on a suitable support member, said wet tobacco web being sufficiently free to allow drainage of liquid and passage of microcapsules thereinto, and applying a plurality of microcapsules in the form of a liquid suspension to said wet web, said microcapsules having an average diameter smaller than the thickness of the sheet and
preferably of such size and in such quantity that they are protectively nested in the interstitial spaces between the tobacco fibers, said suspension containing in solution a film-forming polymeric material whereupon the film-forming polymeric material envelopes the said capsules and simultaneously adheres to the proximate tobacco fibers, thereby to affix said capsules to tobacco fibers.