

Sept. 15, 1970

E. STOSSEL

3,528,432

CIGARETTE OR THE LIKE HAVING COMBUSTION STOP

Filed Oct. 4, 1967

FIG. 1

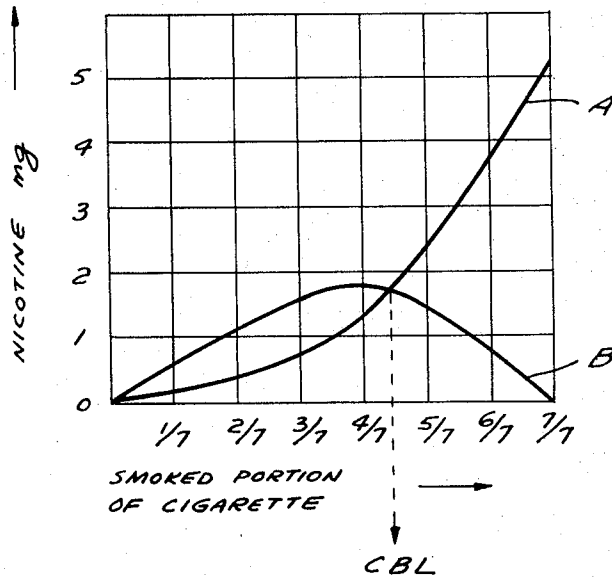


FIG. 2

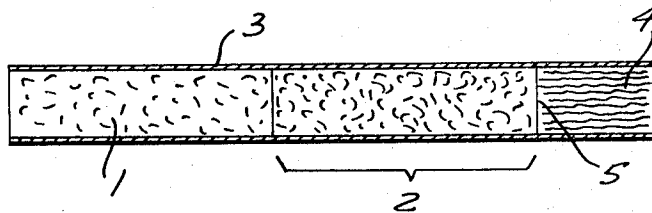
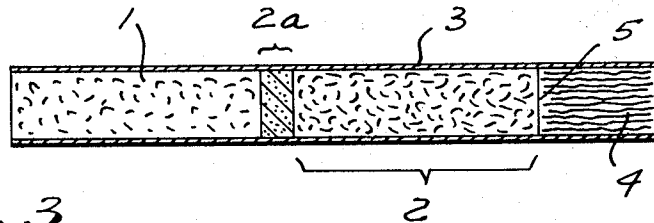


FIG. 3



INVENTOR
Eustace Stossel
BY *Michael S. Striker*
ATTORNEY

1

3,528,432
CIGARETTE OR THE LIKE HAVING
COMBUSTION STOP

Ernest Stossel, 203 W. 81st St.,
 New York, N.Y. 10024

Filed Oct. 4, 1967, Ser. No. 672,762
 Int. Cl. A24d 1/06, 1/10

U.S. Cl. 131-4

10 Claims

ABSTRACT OF THE DISCLOSURE

A smoking product including a tubular wrapper in which a first tobacco filler extends inwardly from one end of the wrapper towards the other end thereof, and in which a gas permeable second filler having the structure of a packed column is located in the tubular wrapper between the other end thereof and the first filler. The second filler consists at least in the portion thereof which is located adjacent to the first filler of a material which inhibits propagation of combustion. Preferably the second filler or at least the portion thereof adjacent the first filler consists of tobacco material which has been flame-proofed by a coating or impregnation consisting essentially of a polyamide of an oxyacid of phosphorus.

BACKGROUND OF THE INVENTION

The present invention is concerned with smoking products, particularly cigarettes, which, upon being smoked will yield a smoke of relatively low concentration of potentially hazardous constituents. It is particularly concerned with the insertion of a flame-proof insert, preferably of flame-proof tobacco in the path of the smoke downstream of the portion of the smoking product which is subjected to combustion. This insert, having the structure of a packed column, will serve to contain and retain toxic or potentially hazardous substances of the smoke and to slow down certain chain reactions occurring in conventional smoking.

The insert will also serve to make the cigarette self-extinguishing when smoked to a predetermined fraction of its entire length, before condensed and adsorbed substances in the insert (which may be a tobacco insert) will be subjected to re-ignition and thermal decomposition which would introduce volatile irritants into the smoke.

Preferably, the material of the flame-proof insert is flame-proofed by being impregnated or coated with impregnating agents disclosed in my copending application, Ser. No. 451,959 filed Apr. 29, 1965 now Pat. 3,414,374, the contents of which are incorporated herein by reference.

The problem of developing "safer" cigarettes apparently cannot be solved by adding more effective filters to a full length cigarette. The quantities of irritants and toxicants developed by burning a full length cigarette seem to be considerably greater than the rate of metabolic removal of these substances by the detoxication mechanism of the body. The statistical evidence shows clearly that the death rate of smokers increases with the amount of cigarettes smoked. Most of the investigators agree that the total amount of smoke introduced into the organism will have to be drastically reduced. And since it proved prac-

2

tically impossible to adsorb the excessive quantities of potentially hazardous substances from the smoke by filters, the solution might be found in reducing the formation of such substances during smoking, or avoiding their being carried away by the smoke stream.

No one knows which of the 500 different compounds identified in the particulate phase and gas phase of the cigarette smoke so far might be responsible but the fact remains that the death rate increases with the amount of cigarettes smoked. Conceivably, the mere process of smoking anything may be unhealthy.

Although most investigators are in agreement that pyrolysis plays a most important role in the formation of potentially dangerous substances in the smoke, the processes, or the sequence of processes of combustion in burning cigarettes have not been fully investigated.

The combustion of a cigarette takes place on one end of a packed column of relatively small diameter and large surface area. In a packed column, however, the interfacial contact area offered to the smoke moving at the rate of up to 3.5 cm. per 0.01 second is considerably increased since the gas or fluid flowing through a series of irregular channels formed by the interstices between the particles of the packing is forced into a turbulent movement which promotes rapid mixing. It is known that the unburned portion of the tobacco column in a cigarette through which the smoke is forced to flow, is a more efficient "adsorber" of nicotine and tar than some filters of equal length. Through filters, where porous media are interposed the smoke flows with practically no cross flow or mixing effect; their effectiveness is due to the pore-size, more or less, and pressure drop (draw) and retention of aroma in the filter increase with increase in the filter efficiency. In contrast thereto, the pressure drop in a packed column depends on the size, shape and arrangement of the particles of the packing. High adsorbing efficiency and low pressure drop can be easily combined.

Upon smoking the cigarette, the length of the packing, and simultaneously the adsorbing surface of the tobacco packing is continuously reduced while more and more products initially adsorbed on the surface are then re-ignited and subjected to repeated cracking. Although the combustion mechanism changes thus continuously, there are two distinct phases of the process which might be considered separately.

This fact has been known since a long time when measurements of nicotine content of the smoke from cigarettes smoked to different lengths of butt ("butt" denotes the unburn portions of the cigarette) were made. When about 60% of the cigarette is smoked, the amount of nicotine condensed on the "butt" is still greater than the amount found in the smoke; at a ratio of about 4.5 unsmoked to 2.5 unburnt of a total length of 7 cm., these conditions become rapidly reversed. The point where the two curves cross might be considered the "critical butt length," since it separates a more or less harmless smoking from an increasingly dangerous one. Critical butt length, or CBL as it will be referred to herein, is not a constant but depends on the composition of the tobacco, length of cigarette, moisture content and other factors which will become apparent in the examples.

The following Table I will illustrate this point:

TABLE I

Length of butt, cm.	Fraction of cigarette smoked (F)	Amount of nicotine in smoke per cigarette whose dry weight equals 1 g. (A), mg.	Nicotine found in the smoke as compared to nicotine in the original tobacco, percent	Nicotine condensed in butt, percent
3-----	4/7	1.3	6.0	60.1
2-----	5/7	2.5	11.5	38.6
1-----	6/7	3.8	17.5	22.3
0-----	7/7	5.7	26.3	0.0

The percentage of nicotine condensed in the butt was calculated from the difference between the amount of nicotine actually found and the nicotine which theoretically should have been found in the smoke

$$\left(\frac{5.7 F - A}{5.7 F} \times 100 \right)$$

The amount of nicotine in the smoke after smoking a predetermined fraction of the total length of the cigarette and the amount of nicotine condensed from the smoke in the as yet unsmoked butt may be graphically illustrated as shown in FIG. 1 of the drawing.

The nicotine content is taken herein as an approximate measure for all of the irritants or at least potentially hazardous substances produced upon smoking of the cigarette (or other smoking product).

SUMMARY OF THE INVENTION

The present invention thus contemplates a smoking product, for instance a cigarette or a Cigarillo which comprises, in a tubular wrapper, a first filler consisting essentially of tobacco and adapted to be smoked. This first filler extends inwardly from one end of the tubular wrapper towards the other end thereof. The tubular wrapper consists, at least in the region of the first filler, of combustible material. A second filler is located in the tubular wrapper between its other end and the first filler. The second filler preferably will be in the nature of a packed column and the entire second filler or at least the portion thereof adjacent to the first filler consists of a material which inhibits propagation of combustion, so that the cigarette or the like will become self-extinguishing as soon as it has been smoked down to the second filler. However, the smoke formed while smoking the first filler will have to pass through the packed column formed by the second filler prior to coming in contact with the smoker.

Preferably, the second filler will be formed of tobacco, but it may be formed of other materials which also may be combustible, and the combustible second filler material is impregnated or coated with a flame-proofing composition, preferably of the type disclosed in the said co-pending application Ser. No. 451,959, such as a polyamide of an oxyacid of phosphorus with the polyamide containing nitrogen and phosphorus present in the group —N—P, and being at least 5% by weight of amido nitrogen and at least 10% by weight of phosphorus.

The novel features which are considered as characteristic for the invention are set forth in particular in the appended claims. The invention itself, however, both as to its construction and its method of operation, together with additional objects and advantages hereof, will be best understood from the following description of specific embodiments when read in connection with the accompanying drawing.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a graphic representation of the nicotine content of the smoke and the nicotine condensed in the butt, upon smoking the cigarette for varying fractions of its entire length;

FIG. 2 is a cross-sectional longitudinal view of a cigarette in accordance with the present invention; and

FIG. 3 is a cross-sectional longitudinal view of another embodiment of a cigarette in accordance with the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring first to the drawing and particularly to FIG. 1, it is noted that the graph has been prepared in accordance with analyses reported by J. Jensen, Agr. Research Vol. 51, No. 3, concerning the nicotine content in the smoke as affected by the length of the butt.

The cigarette had a length of 70 mm., a dry weight of 1 gram, a total nicotine content of 21.7 mg. and a moisture content of 11%.

Curve A shows the nicotine content in milligrams in the smoke upon smoking of the indicated fraction of the total length of the cigarette, and curve B shows the amount of nicotine condensed in the as yet unsmoked portion or butt of the cigarette.

The critical butt lengths (referred to hereinbelow as ("CBL")) is the length of the butt at approximately the point at which Curves A and B cross-section each other.

It is to be noted that the nicotine content is referred to as an approximation of the content or proportion of toxic and/or potentially harmful constituents of, or irritants in, the smoke. To use the nicotine content and changes thereof as an indication of the change in the proportions of other undesirable smoke constituents appears to be of sufficient accuracy for the purpose of describing the present invention.

Continuing to smoke the cigarette beyond the CBL so as to further reduce the butt lengths will increase the nicotine content in the smoke up to five times as much as that found at the "critical butt length" (CBL).

The general introduction of filter tips allows smoking to a much shorter butt length than was possible before the filter separated the lips from the burning tobacco end (coal). The pyrolytically formed bases (nicotine among them) and acids, both volatile and diluted by large volumes of air, are forced to recombine on the large surface of the packing (as long as such a surface remains large enough) to non-volatile salts, which are again dissociated by pyrolysis when the packing is ignited.

Of the more than 500 compounds so far identified in tobacco smoke, about 50 are of a basic nature, among them nicotine.

If the process of burning tobacco in a cigarette with an excess of oxygen (air) would simply consist of combining essentially carbohydrates (cellulose) with oxygen, the end result of this combustion would be carbon dioxide and water, and the detoxication of the smoke from small quantities of undesired by-products (including nicotine) would be easy.

Unfortunately, the combustion of tobacco under the conditions of smoking cigarettes takes place in a much more complicated way. Reactions between oxygen and organic compounds in the gas phase at temperatures of about 700° to 900° C. are almost invariably chain reactions involving atoms and free radicals. Free radicals are molecules or parts thereof in which the normal chemical bond has been modified so that an unpaired electron is left in the system. All oxidation of organic molecules proceeds in two successive univalent steps, the intermediates being free radicals. Free radicals owe their origin and excitation to a highly exothermic reaction step. (Temperature of the cigarette "coal" during puffing jumps rapidly from that of the free burning "coal" temperature.) When organic compounds are rapidly heated to high temperature, a profusion of very reactive free radicals results, the total of which have been found to be increased 50% when the temperature is raised from 850° to 950° C.

Reaction chains are formed because in the reaction of a mono-valent free radical with a neutral molecule one valence remains unsatisfied so that one of the reaction products becomes a free radical capable of further reaction. Free radicals react with each other, or with other substances which might be present, and a chain reaction sets in. Intermediate products seem to be peracetic acid and formaldehyde which are believed to dissociate into free radicals, or alternatively to decompose into neutral products such as $\text{CHO}^* + \text{CO} + \text{H}_2\text{O}$, giving rise to the development of carbon monoxide.

In a packed reaction vessel, chain carriers are efficiently removed from the gas phase by adsorption and their destruction on the surface. As long as the adsorbing surface remains sufficiently large, polymerization processes

are preferentially catalyzed on the surface, and less side-reactions will take place. The actual process of polymerization further affords a very efficient trapping mechanism for the reactive radicals. Relatively large concentrations of stabilized radicals are built up as the process continues, and the active radical ends become trapped in a "dead polymer cage."

In recent years, since the introduction of the electron spin resonance spectroscopy, free radicals have been intensively studied. There is a relatively high concentration of both active and stabilized free radicals in cigarette smoke condensate. Tar constituents from smoke contain about 6×10^{15} free electrons per gram. The concentration of radicals obtained in the frozen (liquid oxygen) smoke condensate before warming was found to be of the order of 10^{15} per gram. On warming, the condensate separated into an aqueous and an organic (tarry) phase. No radical concentration could be detected in the aqueous phase, which might be worth mentioning because of a possible relation of this fact to a report indicating "no tumors were found" when a water soluble fraction was applied to the skin of mice.

Unfortunately, all the tests referred to in the literature were made from smoke condensate derived from burning the full length of cigarettes. Further examination where the necessary facilities are available would be desirable to determine exactly how far the trapping mechanism of the packed column can detoxify the smoke, or eliminate formation of volatile substances which might be carried eventually into the smoke.

The large surface of the tobacco packing acts as an "adsorber" for free radicals and catalyst for polymerization processes. The chain carriers are thus destroyed and trapped by the polymerization process, and removed from further reactions.

The packed column performs similarly to control rods in nuclear reactors whose basic function is to remove reactivity, and thus control the chain reaction.

When this "adsorber" is then partially removed (in the chain reaction of burning cigarettes by cremating the "critical butt length" in nuclear reactions by extracting the rods) the chain reaction sets in again. Simultaneously, condensation products initially fixed on the packing surface are re-ignited and additional chain reactions take place, however, without the benefit of a trapping mechanism for free radicals.

It is to be assumed that during this re-ignition period most carcinogenic polycyclic compounds are formed, and then dispersed in the smoke and carried into the organism in such amounts that the, by then, mostly exhausted filters are ineffective. Formaldehyde might then be carried by the smoke and is capable of reacting rapidly with the amino groups of the tissue protein; carbon-monoxide causing slow, chronic asphyxia; polycyclic compounds and possibly stable free radicals trapped within, to name a few of the undesirable constituents of the smoke, may also be released into the smoke system.

Various filter devices and combination filters have been proposed, however none has been found effective enough to eliminate from the smoke the substances formed after this chain reaction has set in. Obviously, the onset of the chain reaction and the formation of the most dangerous substances caused by these reactions upon removal of the CBL has to be avoided. These chain reactions, which would take place when burning would be continued, can only be stopped when the cigarette is extinguished.

Very few smokers can be educated to discard voluntarily half smoked cigarettes while smoking is still relatively harmless and non-irritating.

It is an object of this invention to achieve this purpose by treating the portion of the cigarette tobacco corresponding to the CBL so that the cigarette can be smoked in a conventional way but to make the cigarette self-extinguishing when the burning zone of the cigarette reaches the CBL.

It is another object to treat the portion of the tobacco-mixture in a cigarette corresponding to the CBL with a flame-proofing agent to make the CBL fire-proof.

It is another object to impregnate the CBL tobacco packing with a flame-proofing agent which will not interfere with the passage of the smoke through the impregnated packing, or cause negligible pressure drop, only, and will extinguish the cigarette without development of obnoxious gases.

It is a further object of the present invention to impregnate the tobacco packing with a flame-proofing agent which will increase the adsorbing efficiency of the packing by enlarging the effective surface of the packing, or by chemically reacting with some of the unwanted compounds in the smoke, and fixing them on the packing. The term "flame-proof tobacco packing" as used herein is meant to denote any incombustible or flame-proof material suitable for substituting for part or all of the tobacco in the packing (filler 2), arranged for the purpose of offering a high contact area and to force a current of gas to change from a stream-lined to a turbulent flow to promote mixing and to increase the interfacial contact area, combined with a high percentage of free space between the particles to minimize pressure drop of the smoke flowing through.

The cigarette according to FIG. 2 comprises a wrapper 3, a first filler 1 consisting essentially of tobacco and extending inwardly from one end of the tubular wrapper 3 towards the other end thereof, and a gas permeable second filler 2 located in the tubular wrapper between the other end thereof and the first filler 1. The second filler 2, consists of a material which inhibits propagation of combustion so that upon combustion of the first filler the cigarette will be self-extinguishing. The second filler will be in the nature of a packed column through which the smoke formed upon combustion of the first filler will flow along a tortuous path. The second filler may consist of tobacco which has been impregnated or coated with flame-proofing material, as will be described in detail further below.

According to the embodiment illustrated in FIG. 3, the second filler 2 may comprise a non-combustible smoke-permeable portion 2a located adjacent first filler 1, so that due to interposition of portion 2a, combustion cannot be propagated from filler 1 to the residual portion of filler 2 which, in this case, may consist of untreated tobacco, i.e., of tobacco which has not been made flame-proof or non-combustible. Reference numeral 4 denotes a conventional mouthpiece or filter tip, and reference numeral 5 the mouth end of second filler 2.

The second filler of the smoking product according to the present invention may be formed of shredded tobacco alone or with absorbent cellulosic material of similar physical properties as tobacco admixed thereto. These materials are well suited as carrier for the flame-proofing agent.

Powdered tobacco dust and wastes might be mixed with fillers, pulp and additives, and the necessary amount of fire-proofing agents incorporated, then rolled into sheets and shredded. Or shredded tobacco might be soaked in the flame retardant solution, and dried to the desired moisture content.

This impregnated flame-proof tobacco is then stuffed into the part of the cigarette between the part of the tobacco meant to be smoked, the first filler, and the mouth-end (preferably containing a filter tip) to a length of from $\frac{1}{8}$ to $\frac{1}{2}$ of the cigarette, preferably to a length corresponding to the CBL determined by analysis.

The assembling of the cigarette or the like may be carried out in conventional manner.

I have found that the flame-proofing agents of the class of amido-polyphosphate complexes, and specially the ammonium salts of the metal-amido-polyphosphate complexes described in my copending U.S. patent application No. 451,959, now Pat. No. 3,414,374 and Belgian Pat. No. 680,287 are specially well suited for the purpose of

the present invention. The ammonium salts of these metals (preferably aluminum or aluminum-iron) amido-polyphosphate complexes may be obtained by a foaming process by reacting concentrated solutions of aluminum-acid-phosphate and urea. They correspond to a composition $\text{NH}_4\text{-Al}(\text{PO}_3\text{NH}_2\text{H}_2\text{O})_n$ and preferably contain from $\frac{1}{3}$ to $\frac{1}{2}$ of the nitrogen directly bound to phosphorus (—N—P—) as amido nitrogen, have an Al_2O_3 content of from 5 to 15%, available as a colloiddally soluble aluminum-hydroxide-phosphate, an acid number of generally from 100 to 300 and a pH of 5-6. They are highly soluble in water and from their concentrated solutions, films are deposited which render cellulosic substrates and the like flame-proof.

Some of the complexes obtained by changing the ratio of phosphoric acid, aluminum hydroxide and urea are characterized in Table II:

TABLE II

H_3PO_4 , in moles	Moles		Complex $\text{P}_2\text{O}_5:\text{Al}_2\text{O}_3:\text{N}_2$	Acid No.
	$\text{Al}(\text{OH})$	Urea		
3	1	1	4.2:8.6	300
3	1	2	4.3:4.4	225
3	1	3	4.2:3.8	145
3	1	2	5.5:4.7	215
4	1	3	6.0:3.6	155
4	1	4	5.7:3.7	123
4	1	6	5.6:2.4	103

The fluffy powder dissolves slowly in 30% of its weight in water whereby two amido groups are hydrolyzed into ammonium groups to form transparent, viscous solutions which can be diluted with water, or ammonia or alkanolamine solutions in water. Of a 60% solution of the complex an amount equal to about 50% of the weight of the tobacco to be treated will be needed to render the tobacco fire-resistant.

I have found that I can prepare an impregnated tobacco of better presentation and adsorptive effectiveness when the mixture of tobacco and complex solution is treated before drying with small amounts of ethanol which may be partly recovered in the drying operation. When a monohydric alcohol is added to a concentrated aqueous solution of the aluminum amido polyphosphate complex under stirring, the solution becomes first more viscous until a voluminous precipitate is formed when the ratio of alcohol to water exceeds about 1:5.

This precipitate separates in the form of curds which are soluble in larger amounts of water or can be made to solvate by adding more of the concentrated complex solution without addition of water. Addition of polyols solvates the jelly precipitate, too.

Tobacco treated with about half of its weight of a concentrated solution of the aluminum complex forms a rather sticky compound which dries and disintegrates with difficulty only into a flame-proof tobacco which is hard to the touch. Surprisingly, when the treated tobacco is mixed with from 5-6% of alcohol, disintegration takes place immediately, the treated tobacco becomes soft and its appearance indistinguishable from that of the untreated tobacco. These metal amidophosphate complexes might also be prepared from a commercially available product, ammonium salt of an amido polyphosphate, sold under the trade name of "Victamide," prepared according to U.S. Pat. 2,122,122, from a dispersion of phosphorus pentoxide in an inert solvent reacted at elevated temperature with ammonia. The complex also contains about one-third of its nitrogen in a "nuclear" form, probably bound directly on phosphorus (—N—P—). These amido-polyphosphate complexes, are excellent water-soluble flame-proofing agents, e.g., for textiles, and form complexes with many metal ions, e.g., aluminum and iron. Aluminum or iron hydroxide can be incorporated and similar compounds or compounds of a similar nature as those described in my copending patent application may be obtained. For instance, according to U.S. Pat. 2,717,198, phosphorus is reacted with oxygen and com-

plexed with ammonia. However, the metal-polyphosphate complexes obtained directly from concentrated acid aluminum phosphate and urea, by foam polymerization according to my copending patent application, has great advantages since aqueous phosphoric acid can be used instead of phosphorus or phosphorus pentoxide, the manufacture therefore greatly simplified, the metal complexes obtained directly in a one-step operation, and low cost bauxite might be used as metal source since it has been found that substitution of iron-hydroxide for part of aluminum hydroxide yields complexes of better adsorptive properties in the performance of this invention.

Additional products to modify or improve flame-proofing characteristics, like urea, ammonium phosphates, ammonium carbamate, borates, glycols, phosphorylated glycols, sugars as well as inorganic humectants like potassium phosphates and polyphosphates, well known to those skilled in the art may be used without changing the gist of my invention.

Additives to the tobacco may be incorporated into the incombustible packing like aromatic substances, glycerol, sorbitol including such which, if they were added to the portion of the tobacco which is to be subjected to combustion, would yield combustion products of unacceptable properties.

Certain additives might be added to the incombustible packing which will not interfere with the flow of the smoke but will become activated when the "hot spot" (coal) approaches the packing. They might be used to eliminate the small amount of ammonia which sometime becomes noticeable when the impregnation is heated. Products like alkali-aluminum-acid phosphate, commercially produced according to U.S. Pat. 2,550,491, and used for leavening purposes might be useful alone or mixed with equal parts of monocalcium-phosphate or sodium acid pyrophosphate, as recommended for that purpose.

I have found that the slight after-taste on extinguishing the cigarette can be eliminated by the proper choice of the flame-retarding agent, according to Table II, see column 7, by selecting complexes of higher acid number. Depending on the composition and pH of the tobacco substrate, particularly the complexes of acid number from 120 to 230 suppress the development of ammonia when being heated, without the necessity of any addition of a neutralizing agent. The complex prepared from 4 moles phosphoric acid, 1 mole aluminum hydroxide and 3 moles urea, acid number 155, has been found to be satisfactory when used in treating the tobacco of one of the most popular brands of cigarettes.

The following examples are given as illustrative only without, however, limiting the invention to the specific details of the examples.

EXAMPLE I

Preparation of a flame-proofing, absorbing agent for the impregnation of filler 2 such as a tobacco insert.

Into 100 ccm. hot water were introduced under stirring 44 grams of Victamide and, after the solution became clear, 12 grams of 85% phosphoric acid added and brought to boiling while stirring was continued. Then 10 grams of aluminum hydroxide of fine particle size (Alcoa C 705, particle size less than one micron) were introduced and boiling and stirring continued until all of the aluminum hydroxide had been dissolved, and a clear, viscous solution was obtained, after 66 ccm. of the water had been evaporated.

Films deposited from this solution were cohesive and glossy, and adhered very well to the substrate (e.g., paper) on which this liquid was coated. This solution was prepared according to Example 24 of my copending patent application Ser. No. 451,959.

50 grams of this viscous solution were diluted with 10 ccm. of water, and 60 grams of shredded tobacco worked into it. The sticky mixture was kneaded into a well-mixed

mass, disintegrated by adding 3 ccm. of ethyl alcohol, and dried at a temperature of about 60° C. until the moisture content was reduced to about 12%. The coated tobacco had the appearance of the original tobacco.

Commercial cigarettes of 10 cm. length of which 7½ cm. consisted of tobacco stuffing were used for the following experiment:

The cellulose filter was removed by a pincher as well as 2½ cm. of the tobacco stuffing. The empty space up to 2½ cm. length was then filled with the flame-resistant tobacco prepared as described in this example, and the cellulose filter re-inserted. A length of 2½ cm. of treated tobacco has been chosen as substantially corresponding to the CBL according to the analyses of Jensen. The appearance of this treated cigarette was identical to that of the original. When this treated cigarette was smoked, no difference in aroma and "draw" could be noticed. The taste was milder and the smoke less irritating. When about ¾ of the cigarette had been consumed by smoking, the cigarette extinguished itself. Analysis of nicotine in the smoke compared to the analysis of nicotine in the smoke of an untreated cigarette smoked to about 0.8 cm. from the filter tip was 5% against 20% of the nicotine contained in the tobacco of the cigarette.

In some of the experiments, a slight off-taste had been noticed before the cigarette stopped burning. Since this appeared to be due to small amounts of ammonia from the pyrolysis of the flame-proofing agent, in subsequent experiments the treated, dried tobacco was mixed with 2% of its weight of powdered sodium-aluminum-phosphate. The aftertaste then became hardly noticeable. The same results were obtained when potassium-iron-acid phosphate was substituted for sodium-aluminum-acid phosphate.

EXAMPLE II

A flame-proofing agent was prepared according to my U.S. Pat. application No. 451,959, Example 13, by mixing 484 grams of 85% orthophosphoric acid, diluted with 40 cc. of water and 78 grams of aluminum hydroxide, and raising the temperature to 125° C. Then, the clear solution was cooled down to below 110° C. and 180 grams of urea was added together with 90 p.p.m. of FC-95 fluorocarbon surfactant. The reaction mixture was then placed into shallow pans to a depth of 1 mm., and the pan placed into an oven heated at 190°. A foam rapidly formed, and a sticky spongy mass was formed, approximately 30 mm. thick which hardened on continued heating at 190° C. within about 10 minutes to a brittle friable sponge. 505 grams of reaction product were obtained which analyzed 55.7% P₂O₅, 9.5% Al₂O₃, 15.8% N₂, about 40% of which were found in the form of amido-nitrogen, after hydration, acid number 155.

30 grams of this ammonium-aluminum-amido-polyphosphate complex was dissolved in 18 grams of water. After about 15 minutes of stirring the pasty mixture became water clear. It was then added to 50 grams of tobacco, kneaded and dried as in Example I, and stuffed (about 0.6 gram) into the lower end of the cigarette as in Example I.

Upon smoking, the cigarette stopped burning when the coal formed of the combustible portion of the cigarette reached the flame-proof insert.

Without the addition of acidic compounds as described in Example I, the taste of the smoke remained excellent to the end. These tests were repeated with alumino-amido polyphosphate complexes prepared with lesser proportions of urea (note Table II). In connection with tobaccos of more alkaline reaction, complexes of higher acid numbers should be used for impregnation of the tobacco packing in order to control the pH of the smoke so as to avoid any undesirable ammonia-caused after-taste.

EXAMPLE III

The flame-proofing agent was prepared as described in Example VI of my above-mentioned co-pending applica-

tion by dissolving 100 grams of bauxite (Al₂O₃ 58%, Fe₂O₃ 1.2%, SiO₂ 6%, TiO₂ 2.2%) in 800 ccm. of 50% orthophosphoric acid under stirring at the boiling point for two hours. Then, the precipitate (mostly SiO₂ and TiO₂) was separated and the filtrate reduced by boiling to a solids content of 80%. The thus-obtained concentrate was mixed with 240 grams of urea and 80 p.p.m. fluorocarbon surfactant, and foamed at 190° C. as in Example II, the reaction product was dissolved in water, worked up into tobacco, and stuffed into the cigarette as in Examples I and II.

EXAMPLE IV

The flame-proofing agent is prepared as in Example II, with the ratio of Al(OH)₃ increased from 48 grams to 55 grams, and processed as in Examples I, II and III. By increasing the ratio of Al₂O₃:P₂O₅, the solubility of the complex in water is decreased, according to the table in Example 36 of my copending application, but the solutions become more adhesive. This solution was worked up with tobacco dust into tobacco sheets made by passing the mixture through steam-heated rollers. From these sheets a shredded tobacco packing was prepared, and stuffed into the cigarette as in Example I.

EXAMPLE V

The flame-proofing agent was prepared in accordance with Example 30 of my copending application, by increasing the ratio of Al(OH)₃ to H₃PO₄ to 1.5:3 and to urea 6, which yielded a better adhesive for sheet forming, and higher content of adsorbent.

EXAMPLE VI

A polyurethane sponge was treated with the flame-proofing agent according to Example II, and then slightly squeezed out so that about 60% of the solid complex remained in the sponge after drying. The thus flame-proofed sponge which had irregular pores about 70% of which were open pores, was cut into pieces of 2½ cm. length and about 0.75 cm. diameter to fit into the cigarette, and was then inserted into the space between the tobacco to be smoked, and the filter tip which was subsequently replaced; the sponge thus forming the "tobacco packing." The "draw" (passage of the smoke) was excellent, the taste greatly improved and burning of the cigarette was stopped when the flame-proofed sponge was reached. Although no strange taste could be noticed when burning was interrupted, vinylite and cellulose sponges were subsequently substituted for the polyurethane sponge since the latter might emit dangerous gases when heated.

EXAMPLE VII

The flame-proofing agent and the mixture thereof with tobacco was prepared as described in Examples I, II and III, and glycerol was added in an amount equal to 4% of the weight of the treated tobacco. The cigarette was stuffed with the thus-treated tobacco as described in Examples I, II and III, and the nicotine content of the smoke was determined. It was found that the nicotine content of the smoke was further reduced by nearly 40% as compared to the nicotine content of smoke which had passed through a filler 2 of treated tobacco which did not include glycerol, i.e., to 3.5% of the nicotine contained in the tobacco.

Even a relatively high proportion of glycerol can be incorporated in the incombustible part of the cigarette (filler 2) since it will not be converted by burning into acrolein. However, when incorporating relatively large proportions of glycerol, the water content of filler 2 should be somewhat decreased.

EXAMPLE VIII

An aluminum-amido-polyphosphate complex prepared as in Examples II and III was added in powdered form to shredded tobacco and intimately mixed while water is sprayed over it. 60 grams of powdered complex are

admixed to each 100 grams tobacco, and just enough water to keep the mixture from becoming sticky. This was achieved with 22 grams water per 100 grams tobacco or by spraying moist steam over the mixture while kneading, the amount of water (steam) needed could be reduced to 18 grams. No further drying was necessary and the treated tobacco obtained in a disintegrated form. It was then conditioned with humectant and mixed with 50 grams of granulated active carbon, and this mixture inserted into the cigarette after 60% of the length of the cigarette (without figuring the part reserved for the filter tip) had been stuffed with untreated tobacco. Then the filter tip again was inserted. The taste of the smoke was very pleasant, and the cigarette was self-extinguishing when the hot spot reached the insert, without change in taste of the smoke.

EXAMPLE IX

The same experiment was repeated but a reaction product which was obtained when the aluminum-amido-polyphosphate complex was heated to 400° C. for 5 minutes was substituted for the activated carbon. A spongy mass was thus obtained which absorbed vapors and gases, as well as liquids very avidly. In other experiments, mixtures of activated carbon and of this reaction product were added to the flame-proof tobacco as well as tobacco aroma and menthol to modify the taste of the smoke.

EXAMPLE X

Comparative analyses were made according to the method of Jensen, by measuring the nicotine content of the smoke at different butt lengths, with cigarettes consisting only of combustible tobacco filling, and cigarettes in which a filler 2 of flame-proof tobacco filling was substituted for varying lengths of combustible tobacco.

The curves compiled from the data obtained were very similar to about a ratio of 9:5 of tobacco smoked-to-unburned tobacco; were slightly flatter when flame-proof tobacco was inserted to a shorter length and showed a steep increase when the CBL (i.e. filler 2) was made shorter and shorter.

EXAMPLE XI

A flame-proofing agent was prepared, mixed with tobacco, disintegrated with alcohol and the mixture dried and consequently worked up and conditioned as described in Examples II, III, IV, V, VIII and IX, and comparative analyses of the nicotine content of the smoke were made in accordance with the method of Heiduschka and Muth, and Jensen. The critical butt length of untreated cigarettes was determined as the point at which the curves of nicotine content in the smoke and nicotine condensed on the butt crossed as exemplified in FIG. 1. Self-extinguishing cigarettes were then prepared by inserting the flame-proof tobacco mixture (filler 2) to a length corresponding to the thus-determined CBL values.

EXAMPLE XII

The flame-proofed tobacco was inserted into a cigarette packed to $\frac{2}{3}$ length with untreated tobacco to an additional length of about $\frac{1}{2}$ centimeter and the remaining part of the cigarette (at the mouth end thereof) was packed with untreated tobacco (and filter tip) so that the flame-proof tobacco insert served only as a barrier to stop the burning of the cigarette. When the insert was reached by the burning zone of the cigarette tobacco, further burning was stopped exactly as if the whole packing or filler 2 would have been treated as in Examples I, II and III.

The cigarette or smoking product of the present invention, as illustrated in FIG. 3, may comprise a wrapper and two fillers of combustible tobacco accommodated in the wrapper axially spaced from each other, and a relatively short layer disk or the like of non-combustible, smoke-permeable material located in the space between the two fillers. This disk or layer is indicated in FIG. 3

by reference numeral 2a and may be a perforated plate of asbestos, or a layer of fibrous asbestos, or a layer of flame-proofed tobacco produced as described further above. Many suitable materials for forming layer or disk 2a will be apparent to those skilled in the art. The material of layer 2a must be non-combustible at the elevated temperature to which it may be exposed upon smoking either one of the two combustible tobacco fillers and it must have a relatively low heat conductivity, so that a relatively thin layer, for instance a layer having a thickness of between 2 and 8 mm., or up to 1 cm., will suffice for preventing propagation of combustion from the smoked tobacco filler to the tobacco filler at the other side of layer 2a.

It is a particular advantage of the last described embodiment of the present invention that by inserting layer 2a between two fillers of combustible tobacco of substantially equal length, the cigarette or the like may be smoked from either end and will be self-extinguishing after combustion of the first ignited tobacco filler has been completed, i.e., when one tobacco filler has been subjected to combustion up to its end portion contacting barrier layer 2a.

It will be understood that each of the elements described above, or two or more together, may also find a useful application in other types of smoking products differing from the types described above.

While the invention has been illustrated and described as embodied in a cigarette, it is not intended to be limited to the details shown, since various modifications and structural changes may be made without departing in any way from the spirit of the present invention.

Without further analysis, the foregoing will so fully reveal the gist of the present invention that others can—by applying current knowledge—readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of the generic or specific aspects of this invention and, therefore, such adaptations should and are intended to be comprehended within the meaning and range of equivalence of the following claims.

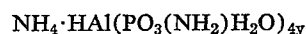
What is claimed as new and desired to be protected by Letters Patent is set forth in the appended claims.

I claim:

1. A smoking product, comprising, in combination, a tubular wrapper; a first filler consisting essentially of tobacco and extending inwardly from one end of said tubular wrapper, said tubular wrapper consisting at least in the region of said first filler of combustible material; and a gas-permeable second filler located in said tubular wrapper between the other end thereof and said first filler, said second filler including a portion located adjacent to said first filler, and at least said portion of said second filler consisting of shredded tobacco coated or impregnated with an ammonium salt of a metal-amido-polyphosphate complex so as to inhibit propagation of combustion, and the said material being arranged to offer a high contact area to smoke pervading said second filler and to cause the smoke to assume a turbulent flow.

2. The smoking product of claim 1, wherein the said metal-amido-polyphosphate complex is an aluminum or aluminum-iron complex.

3. The smoking product of claim 1, wherein the said ammonium salt is of the formula:



where y is a number between 1 and about 20,000.

4. The smoking product of claim 1, wherein the acid number of the said complex is between 120 and 230.

5. The smoking product of claim 1, wherein said second filler additionally includes an alkali-aluminum acid phosphate in an amount sufficient to react with the ammonia developing during smoking from said complex.

6. The smoking product of claim 5, wherein said alkali-aluminum acid phosphate is mixed with about equal parts

13

of mono-calcium phosphate or sodium acid pyro-phosphate.

7. The process of making the smoking product defined in claim 1, comprising applying an aqueous solution of an ammonium salt of a metal-amido-polyphosphate complex to shredded tobacco, forming a filler portion of said tobacco, and aligning the said filler portion with another tobacco filler in a tubular wrapper.

8. The process of claim 7, wherein the said solution of the complex has a 60% concentration and is employed in an amount about 50% by weight of the tobacco treated.

9. The process of claim 7, wherein the tobacco after being treated with the said solution is then treated with a monohydric alcohol.

10. The process of claim 8, wherein the tobacco after being treated with the said solution is then treated with between 5 and 6% by weight of monohydric alcohol.

14

References Cited

UNITED STATES PATENTS

2,185,293 1/1940 Copeman.

FOREIGN PATENTS

121,415 3/1944 Australia.
1,270,093 7/1960 France.
1,402,088 5/1965 France.

OTHER REFERENCES

"MAD" Magazine, "Some Health Saving Filters Now In Use," June 1964, p. 32 cited.

MELVIN D. REIN, Primary Examiner

U.S. Cl. X.R.

131—10.7, 267