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TOBACCO SMOKE FILTER

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ABSTRACT OF THE DISCLOSURE

A novel unitary tobacco smoke filter is provided comprising a non-tobacco filter plug including granules of hydroxy apatite. This filtration component is used in amounts of from about 0.1 to about 80% by weight based on the weight of the tobacco and is desirably used in particle sizes of from about 5 to about 2,000μ. Also contemplated in this invention are filters utilizing auxiliary filters such as charcoal and fibrous cellulose materials.

This appliction is a continuation-in-part of application Ser. No. 665,215, filed Sept. 5, 1967, now abandoned.

This invention relates to tobacco smoke filters exhibiting enhanced physical and chemical filtration properties. More particularly, this invention relates to tobacco smoke filters comprising an inorganic, non-combustible, non-toxic particulate capable of enhancing the normal filtering properties of the tobacco mechanically and capable of removing acidic components chemically and other organic components by adsorption.

The use of tobacco, its reported ill effects, and the many attempts to overcome these effects by filtration are too well known to require elaborate discussion here. Filters which have been proposed for cigarettes have been installed at the end of the cigarette and have usually been mechanical filters designed to remove particulate material from the smoke. Such filters have usually been fibrous in nature although charcoal has been employed, as has water. The fibrous filters have functioned in the same manner as the tobacco itself, i.e., as mechanical filters—although admittedly more efficient. Pipe filters generally comprise a paper or plastic cartridge inserted in the stem of a specially designed pipe to serve about the same function.

It should be noted that a highly efficient filtration device in a cigarette or other tobacco product is undesirable. Such a filter would leave the smoker inhaling warm air. What is desired is an efficient selective filtration means which removes as much nicotine, and tars, as possible but which permits the relatively non-toxic components of the smoke through to provide a satisfactory flavor to the smoker.

In accordance with this invention, it has been found that a tobacco smoke filter comprising a non-tobacco filter plug including granules of hydroxy apatite, provides enhanced filtering porperties in respect to tobacco smoke. The hydroxy apatite filters of this invention can be effectively used for tobacco smoking in pipes, cigars and cigarettes, alone or in combination with other filtering devices, as are known to the art.

By the term comminuted tobacco leaves as used herein, is meant tobacco leaves as they are normally cured for smoking, chopped ground or shredded for use in the particular form desired. For example cigarette tobacco is normally shredded. Pipe tobacco is normally chopped or sliced from plug and rubbed, or is used as flakes cut from the leaves.

The hydroxy apatite utilized in the conduct of the present invention can be represented by the formula:

$$3[Ca_3(PO_4)_2]\cdot M(OH)_2$$

wherein M is a calcium, strontium or tin cation. While hydroxy apatites wherein M is strontium or tin can be effectively utilized in this invention, it is preferred to utilize hydroxy apatites wherein M is calcium. This preference is dictated by the fact that this material is low in cost, non-toxic, commercially available and highly effective.

Because of this preference and for convenience, calcium will be used hereinafter as representative of the hydroxy apatites used in this invention.

The hydroxy apatite used herein is particulate, preferably having an average particle size from about 2000μ to about 5μ in diameter, although particles having diameters from about 800μ to about 100μ inclusive are most preferred, as such particle size ranges provide excellent filtration. While larger and smaller particulates can be used they are less effective in the compositions of this invention. In more general terms the particle size can be stated in terms of mesh size. For example, it is preferred that the particle size of the hydroxy apatite be sufficiently small that it passes a 12 mesh screen and yet sufficiently large that it rests on a 100 mesh screen. In the examples set forth hereinafter, mesh sizes are employed to denote particle sizes. In the examples and throughout this specification mesh sizes, where employed, are U.S. Standard mesh sizes.

While the surface area of the calcium hydroxy apatite can also vary over a wide range, it has been found that materials having high surface areas provide enhanced smoke filtration. Accordingly, it is preferred that the hydroxy apatites exhibit a B.E.T. surface area more than 50 $(meters)_2$ per gram. B.E.T. surface area is the Brunauer, Emmet, and Teller method for determining surface area as described in their article published in J. Am. Chem. Soc. 60, 309–319 (1938).

The material is conveniently prepared by precipitation from aqueous solution at a pH of 7.0 to 7.5. This preparation can be exemplified by the reaction mechanism which follows:

$$10Ca(OH_2+6H_3PO_4\rightarrow 3[Ca_3(PO_4)_2]\cdot Ca(OH_2+18H_2O$$

Hydroxy apatite prepared in this manner is amorphous, highly porous and granular. This material has been found to function in three ways in the filtration of gas. First, the material filters mechanically. Second, this material effects a high degree of adsorption and, third, this material reacts with acidic components in the gas effectively binding them and removing them from the gas stream.

As has been indicated above, the calcium hydroxy apatite is non-toxic to the extent that it is edible. These materials are also non-combustible being inert, inorganic compounds. These properties make the hydroxy apatites ideally suitable for use in the filtration of tobacco smoke as set forth in this invention.

The hydroxy apatite filter component can be used in any amount but is recommended in an amount up to about 80% by weight based on the tobacco to be smoked. Amounts less than 1.0% by weight can be used if desired but provide less effective filtration. If, however, auxiliary filtration is employed, such lesser amounts can be more effectively used. Accordingly, amounts of from about 0.1 to about 80% by weight can be used. Amounts of from about 10 to about 40% are preferred. For most cigarettes, this generally is from about 100 to about 400 milligrams but can vary with both cigarette size and filter size.

The term "filter" as used in this invention is an integral unit disposed between the smoker and the burning tobacco in such a maner that the smoke normally passes through the filter. The filter of this invention can be in the form of a cartridge type, a tip type or any other unit type filter form conventionally used for the filtration of tobacco smoke. Such filters can be composite type filters utilizing more than one type of filter material so long as the hydroxy apatite component of this invention comprises a functional portion of the filter unit. The hydroxy apatite when utilized in a particulate form will generally require the use of additional mechanical type filters as retaining members for the particulate filter material. When the hydroxy apatite is prepared in the form of a pellet, the retained filter members need not be utilized but can be if aesthetic requirements demand. Additionally, whil as indicated above, the filters of this invention are of the unitary type, they can be effectively utilized with auxiliary filtration means.

The hydroxy apatite filter component of this invention can be utilized alone or in combination with other filtration devices as are known in the art. They can be and preferably are used as particulate solid materials which require the use of a retainer at the end of the device employed. Suitable retainers include mechanical fibrous type filters as are commonly used in the art today as well as paper and other retainer means known to the art. The filter component of this invention can also be used in combination with other particulate type filter materials such as active carbon and can be combined by admixture or by strata. When active carbon is employed with this material, it has been found desirable to utilize the particulate material in admixture.

The filter of this invention can also be effectively employed as a pressed cake of particles of the desired size. Such pressed filters are conveniently prepared by pressing wet hydroxy apatite into the desired shape and thereafter drying. Such pressed filter elements are advantageous in that they require no retainer at the end of the filter and are easily handled during manufacture. Such pellet type filter components can also be prepared on standard pelleting devices as are used in the preparation of pills and tablets. These pellets, however, must be prepared in such maner that they remain porous.

The tables below set forth the approximate composition of cigarette smoke.

Cigarete smoke is a heterogeneous mixture of gases, uncondensed vapor and liquid particulate matter. As it enters the mouth, the smoke is a concentrated aerosol with billions of particles per cubic centimeter. The median size of the particle is approximately 0.5 micron. Tabulated below are the major classes of compounds in the particulate and gaseous phases as reported in the "Report of the Advisory Committee to the Surgeon General of the Public Health Service."

A. Particulate phase

Class	Percent	No of comp.	Toxication on lung
Acids	7.7–12.8	25	Some irritant.
Glycerol, glycol, alcohols	5.3–8.3	18	Possible irritation.
Aldehydes and ketones	8.5	21	Some irritant.
Aliphatic hydrocarbons	4.9	64	Do.
Aromatic hydrocarbons	0.44	81	Some carcinogenic.
Phenols	1.0–3.8	45	Irritant and cocarcinogenic.
	66.0	254	

B. Gaseous phase

The gaseous phase of cigarette smoke accounts for 60% of the total cigarette smoke. Seven compounds make up 98.9 mol percent of the gas phase.

	Mol percent
Nitrogen	73.0
Oxygen	10.0
Carbon dioxide	9.5
Carbon monoxide	4.2
Hydrogen	1.0
Argon	.6
Methane	.6
	98.9

Approximately one percent is constituted of:

	P.p.m.
Eethane, propane butane	87,000
Acetylene, ethylene, propylene	31,000
Formaldehyde	30
Acetaldehyde	3,200
Acrolein	150
Methanol	700
Acetone	1,100
Ammonia	300
Nitrogen dioxide	250
Methyl nitrite	200
Hydrogen sulfide	40
Hydrogen cyanide	1,600
Methyl chloride	1,200

Carcinogenic materials have been isolated from cigarette smoke in addition to the above.

	μg./1000 cigarettes
Benzo (a) pyrene	16
Dibenzo (a,i) pyrene	10
Dibenzo (a,h) pyrene	4
Benzo (c) phenanthrene	--

The filtration media of this invention effectively removes acidic components from tobacco smoke by chemical reaction. It removes many of the organic liquid particulates from the smoke by adsorption and mechanical filtration.

Hydroxy apatite is prepared by the addition of dilute phosphoric acid (9.0–20.8%) to a slurry of milk of lime [$Ca(OH)_2$] having 8–12% hydroxides until the pH of the neutralized solution is 7.0 to 7.5. This results in a suspension of hydroxy apatite which is filtered, dried and milled to a specified particle size. Drying can be effected by any conventional means such as spray drying, tray drying, rotary drying and the like.

Variations can be made from the pH specified above which would result in unneutralized calcium hydroxide, which will not materially affect the properties of the hydroxy apatite for use in the subject invention.

In this respect it should be noted that the calcium hydroxy apatites having mole ratios of CaO/P_2O_5 from about 3.20 to about 4.0 are preferred and those having mole ratio of from about 3.25 to about 4.0 are most preferred.

Additionally when the calcium hydroxy apatite has been formed in solution at a pH of about 7, it can be modified by adding a small amount of sodium or potassium hydroxide solution to raise the pH to about 8 to 11, if desired.

The surface area of the hydroxy apatites can also be increased through modification of the conditions of preparation. For example, the neutralization can be conducted in the presence of a protective colloid such as gelatin or carboxy methyl cellulose and the resultant material can be dried at relatively low temperatures under vacuum at, for example, room temperature. Alternatively, this material can be freeze dried to retain the maximum cell structure.

The neutralization can be conducted at temperatures from 25° C. to 110° C.

In the examples which follow and throughout this specification, all parts and percentages are by weight unless otherwise specified.

EXAMPLE 1

A 10% aqueous solution of phosphoric acid was slowly added to a 10% aqueous slurry of 57 grams of calcium

hydroxide until the pH of the reaction solution was essentially neutral, i.e. about 7.0. Both solutions were used at a temperature of 70° C. The reaction mixture contains a suspension of hydroxy apatite which was subsequently filtered, washed and dried. The dried hydroxy apatite was subsequently ground to an average particle size in the range of from about 5 to 2,000μ.

A cigarette filter was prepared from the hydroxy apatite prepared above by mounting a ¼" fibrous cellulose acetate filter in a conventional paper filter tube. Approximately 100 to 200 milligrams of hydroxy apatite was charged to the paper cylinder and was held in place by a 3/16" cellulose acetate conventional filter. The composite filter tube was mounted on a standard brand cigarette.

The tin hydroxy apatites and the strontium hydroxy apatites were prepared in a manner identical to that set out above except that a stoichiometric amount of a soluble tin or strontium salt was added to the acid solution or calcium hydroxide slurry before combining the solutions. The nitrates of these metals can be effectively used.

While as indicated above it is preferred to utilize prepared hydroxy apatite precipitated from solution, it is possible to utilize naturally occurring hydroxy apatite ground to the proper size.

EXAMPLE 2

I. Preparation of filter materials

The materials prepared for evaluation in the examples below were made in accordance with the following procedures.

Preparation 1.—Phosphoric acid (24% H_3PO_4) was added rapidly to an aqueous suspension of $Ca(OH)_2$ containing approximately 1 pound calcium oxide per gallon until the mixture attained a pH of 9. The addition of the acid was then continued at a slow rate until the pH of the reaction mixture was 7. The reaction temperature was between 50° and 70° C. The product, hydroxy apatite, was filtered and dried at 150° C.

The dried hydroxy apatite was then ground and screened to a particular size. Samples of hydroxy apatite made in accordance with this procedure are designated: HA–1.

In preparations 2–5 below the method of preparation 1 was followed except as noted.

2. The final reaction mixture was boiled for 43 hours. The hydroxy apatite prepared by this procedure is designated: HA–2.

3. The reaction temperature was kept at 25–30° C. by cooling. The hydroxy apatite prepared by this procedure is designated: HA–3.

4. The final pH of the reaction mixture was adjusted to 10 by the addition of dilute aqueous sodium hydroxide solution. The hydroxy apatite prepared by this procedure is designated: HA–4.

5. This procedure was identical with preparation 4 except that calcium hydroxide suspension was used in place of sodium hydroxide solution. The hydroxy apatite of this procedure is designated: HA–5.

6. Procedure was conducted in accordance with Synthesis No. 17, "Inorganic Syntheses," volume VII, page 63, McGraw-Hill Co. (1963). This procedure involved reacting calcium nitrate with $(NH_4)_2HPO_4$ in solution at pH 12. The calcium apatite reaction mixture was calcined a 800° C. after drying off ammonium nitrate at 240° C. The calcium hydroxy apatite of this procedure is designated: HA–6.

7. A suspension of ten grams calcium hydroxy apatite in 200 milligrams of water was slurried with 100 grams of activated coconut charcoal (−12+40 mesh), filtered, and the filter cake dried at 105° C. This filter material was designated: CA–7.

8. Ten grams of calcium hydroxy apatite and 100 grams of activated coconut charcoal (−12+40 mesh) were combined in the dry state and mixed mechanically. This filter material is designated: CA–8.

9. Equal parts of calcium hydroxy apatite HA–1–1 (−12+40 mesh) and calcium hydroxy apatite HA–1–3 (−40+80 mesh) were combined and mixed mechanically. This calcium hydroxy apatite, both mesh sizes prepared by preparation 1 above, was designated: HA–9.

10. Commercially available hydroxy apatite was also employed as a filter material. This material was prepared by the addition of dilute phosphoric acid (9.0–20.8%) to a slurry of milk of lime [$Ca(OH)_2$] containing 8 to 12% hydroxide until a pH of the neutralized solution was 7.0 to 7.5. This results in a suspension of hydroxy apatite which was filtered, dried, and milled to a specified particle size. Drying of this material was done on a conventional rotary dryer. This material was designated: HA–10.

Mesh sizes were determined by passage through one screen (−) and hold on a finer screen designated (+). Mesh sizes are U.S. standard.

Designation:	Mesh size
HA–1–1	−12+40
HA–1–2	−40+100
HA–1–3	−40+80
HA–2	−40+80
HA–3	−40+80
HA–4	−40+80
HA–5	−40+80
HA–6	−40+80
CA–7	−12+40
CA–8	−12+40
HA–9	−12+40/−40+80
HA–10–1	−40+80
HA–10–2	−12+40
HA–10–3	−40+100

Surface area and chemical analysis of calcium hydroxy apatites of −40+80 mesh particle size was determined by a number of samples. The results of this determination are indicated below.

Sample design	B.E.T. surface area M_2/gm.	Chemical analysis: Percent Ca.	Percent P	Ml. Ca/P	Mol CaO/P_2O_5
HA–1–3	69.6	37.2	17.5	1.64	3.28
HA–2	60.3	37.9	18.0	1.63	3.26
HA–3	72.5	37.1	18.0	1.59	3.18
HA–4	75.5	37.1	17.5	1.64	3.28
HA–5	72.7	37.7	17.5	1.66	3.32
HA–6	18.2	39.3	18.4	1.65	3.30
HA–10	66.6	38.7	17.8	1.68	3.37

Particle size distribution of hydroxy apatites of −40+80 mesh particle size was determined for the samples designated. The results of this determination are shown below in units of weight/percent.

Mesh size U.S. std.	HA–1–3	HA–2	HS–3	HA–4	HA–5	HA–6
+40	2.7	2.6	2.3	1.9	2.1	1.9
−40+50	33.8	34.2	43.0	41.0	37.2	43.6
−50+60	22.2	21.5	22.0	22.4	17.4	19.7
−60+70	15.4	14.3	13.2	13.0	9.9	12.1
−70+80	12.2	10.6	9.1	9.5	7.3	9.2
−80	13.7	16.8	10.6	12.2	26.1	13.5

II. Evaluation of filter materials

Filter preparation.—Filters were prepared in all instances by removing the cellulose acetate filter plug from a single brand of commercial cigarettes. Two 2 millimeter cross-sections were cut from the 104 millimeter filter plug. One 2 millimeter section was replaced in the paper tube of the filter section and pushed down against the tobacco. Approximately 300 milligrams of the test sample to be evaluated was then placed in the paper tube on top of this 2 millimeter section. The second 2 millimeter section of the plug was then placed on top of the test sample in the paper tube of the filter to retain the test sample and close the filter.

Twenty filter cigarettes were prepared for each sample to be tested and 20 of the commercial cigarettes from

which the standard cellulose acetate filters were removed were used as a control. These cigarettes were smoked according to the procedures approved by the Federal Trade Commission.

Determination of particulate matter.—Cambridge filter assemblies were weighed to the nearest 0.1 milligram and connected to the smoking machine by a short piece of rubber tubing so that the cigarettes and filter assemblies were held in a horizontal position. The smoking machine and filter assemblies were tested for leaks.

A cigarette was inserted through the hole in the rubber membrane on each filter assembly until the end of the butt was approximately flush with the end of the holder tube and then withdrawn slightly so that the rubber membrane formed a snug seal around the cigarette.

The cigarettes were lighted at the beginning of the first puff with an electric coil lighter. Each cigarette was smoked until the burning coal reached the predetermined marked butt length where the port was deactivated. The cigarette was removed from the holder and the next cigarette inserted. In this manner, five cigarettes were smoked through each filter. After five cigarettes were smoked through each of the filter assemblies, the assemblies were disconnected from the machine, wiped and weighed to the nearest 0.1 milligram.

The weight gain of each filter assembly was recorded and the smoked samples were retained for the determination of moisture and nicotine. The weight of particulate matter for each Cambridge filter was calculated in milligrams per cigarette. The average for all filters is the value reported for the particulate matter in the smoke of the sample.

Determination of moisture in particulate matter.—This determination was made according to the procedure described in a paper entitled "Determination of Moisture in Total Particulate Matter' by Schultz and Spears in Tobacco Science, 10, 75–76 (Tobacco, 32–33, June 17, 1966).

As soon as the filter assembly for particulate matter determination had been weighed, it was opened and the filter placed in a dry 30 milliliter serum bottle. The inner face of the filter holder was then wiped with one-fourth of a Cambridge filter pad and this added to the serum bottle. Ten milliliters of dry dioxane-isopropanol (100:1) was measured from an automatic buret into the serum bottle and the stopper inserted. The bottle was shaken for 20 minutes on a Wrist-Action shaker.

A sample of 10 microliters was withdrawn with a Hamilton syringe through the rubber serum cap and subjected to gas chromatography in an Aerograph 90–P3 chromatograph under the following conditions:

Column	Porapak Q 3' x ¼".
Column temperature	240° C.
Injection port temperature	180° C.
Detector	260° C.
Carrier gas	Helium at 40 ml./min.

The moisture content of the particulate matter was read from a calibration curve made by adding known amounts of water to the solvent mixture. Filters conditioned at 75° F. and 60% relative humidity were run and the average value of the moisture content subtracted from the amounts found with the used filters to obtain the net moisture content.

Determination of nicotine in particulate matter.—The dioxane-isopropanol solution containing the dispersed Cambridge filter, from which only 10 microliters has been withdrawn for moisture determination, was transferred to a Griffith Still quantatively and 25 milliliters of 1:500 sulfuric acid added. The acid solution was steam distilled for 5 minutes. The distillation was stopped and the condensate discarded. A 250 milliliter volumeric flask, containing 10 milliliters of 1:4 hydrochloric acid, was placed under the receiver tip. Five milliliters of the sodium hydroxide-sodium chloride solution was added to the distillation flask and the alkaline solution was rapidly distilled until about 225 milliliters of distillate was obtained. The volume of the solution was made up to 250 milliliters with distilled water.

The absorbance of the distillate was determined at 236,259 and 282 mmμ against a blank of 0.05 N hydrochloric acid, using 1 centimeter cells.

The net absorbance of the nicotine solution corrected for background was calculated as follows:

$$A'_{259}=1.059[A_{259}-\tfrac{1}{2}(A_{236}+A_{281})]$$

and the weight of nicotine in the distillate was calculated from the absorbance of a solution of pure nicotine in 0.05 N hydrochloric acid at 259 millimeters.

A separate analysis was made on each filter and the weight of nicotine calculated and expressed as milligram per cigarette.

In the evaluations which follow, the commercial cigarette used as control was tested after the cellulose acetate filter plug had been removed so there was essentially no filter in the control. The butt length in all cases was 33 millimeters.

TEST 1

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	33.2	6.5	1.30	25.4
Run 2	32.8	6.3	1.36	25.1
Run 3	32.8	6.2	1.43	25.2
Run 4	33.2	6.6	1.47	25.1
Average	33.0	6.4	1.39	25.2

Average number of puffs 8.2.

TEST 2

[Absorbent HA-10-2 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	23.7	3.3	0.97	19.4
Run 2	24.7	3.8	0.94	20.0
Run 3	23.3	3.3	1.07	18.9
Run 4	22.5	3.3	0.95	18.2
Average	23.6	3.4	0.98	19.1

Average number of puffs 8.7.

Pressure drops in inches of water	5
Percentage reduction:	
Tar	24.2
Nicotine	29.5

TEST 3

Control no filter	Total particulate matter	Mg. in smoke of 1 cigarette		
		Moisture	Nicotine	Tar
Run 1	35.4	4.2	1.56	29.6
Run 2	36.4	4.8	1.67	30.0
Run 3	33.4	3.3	1.52	28.6
Run 4	34.2	3.9	1.54	28.8
Average	34.9	4.1	1.57	29.2

Average number of puffs 8.6.

TEST 4

[Absorbent HA-10-3 (300 milligrams per filter)]

Control no filter	Total particulate matter	Mg. in smoke of 1 cigarette		
		Moisture	Nicotine	Tar
Run 1	13.0	1.6	0.61	10.8
Run 2	13.6	1.8	0.58	11.2
Run 3	13.1	1.5	0.60	11.0
Run 4	14.1	2.2	0.59	11.3
Average	13.5	1.8	0.60	11.1

Average number of puffs 9.0.

Pressure drops in inches of water	9
Percentage reduction:	
Tar	62.0
Nicotine	61.8

TEST 5

[Absorbent HA-10-3 (200 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	10.9	1.2	0.27	9.4
Run 2	9.7	0.9	0.28	8.5
Run 3	11.2	1.0	0.29	9.9
Run 4	11.8	0.6	0.30	10.9
Average	10.9	0.9	0.29	9.7

Average number of puffs 8.0.

Pressure drops in inches of water ---------- 10.4

Percentage reduction:

Tar ---------- 66.7

Nicotine ---------- 81.5

TEST 6

[Absorbent HA-10-3 (300 milligrams per filter)]

Control no filter	Total particulate matter	Mg. in smoke of 1 cigarette		
		Moisture	Nicotine	Tar
Run 1	10.6	1.1	0.26	9.2
Run 2	9.5	0.6	0.21	8.7
Run 3	9.8	0.8	0.22	8.8
Run 4	10.2	1.1	0.25	8.8
Average	10.0	0.9	0.24	8.9

Average number of puffs 10.0.

Pressure drops in inches of water ---------- 13.0

Percentage reduction:

Tar ---------- 69.5

Nicotine ---------- 84.0

TEST 7

[Absorbent HA-10-3 (350 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	7.4	0.5	0.17	6.4
Run 2	7.0	0.8	0.16	6.0
Run 3	8.1	0.5	0.16	7.4
Run 4	8.6	0.8	0.13	7.7
Average	7.8	0.7	0.16	6.9

Pressure drops in inches of water ---------- 13.0

Percentage reduction:

Tar ---------- 76.4

Nicotine ---------- 89.8

TEST 8

Control no filter	Total particulate matter	Mg. in smoke of 1 cigarette		
		Moisture	Nicotine	Tar
Run 1	31.3	9.5	1.39	20.4
Run 3	33.8	10.5	1.28	22.0
Run 3	32.6	10.2	1.32	21.1
Run 4	30.6	9.0	1.33	20.3
Average	32.1	9.8	1.33	21.0

Average number of puffs 8.1.

Pressure drops in inches of water 2.7.

TEST 9

[Absorbent CA-7 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	13.4	2.7	0.53	10.2
Run 2	11.9	1.5	0.53	9.9
Run 3	12.1	4.0	0.52	7.6
Run 4	12.1	2.5	0.53	9.1
Average	12.4	2.7	0.53	9.2

Average number of puffs 7.8.

Pressure drops in inches of water ---------- 9.3

Percentage reduction:

Tar ---------- 56

Nicotine ---------- 60

TEST 10

[Absorbent CA-8 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	11.7	2.3	0.50	8.9
Run 2	13.6	2.5	0.54	10.6
Run 3	13.5	1.8	0.51	11.2
Run 4	13.2	2.3	0.60	10.3
Average	13.0	2.2	0.54	10.3

Average number of puffs 8.1.

Pressure drops in inches of water ---------- 8.9

Percentage reduction:

Tar ---------- 51

Nicotine ---------- 59

TEST 11

[Absorbent HA-1-3 (300 milligrams per filter)]

Control no filter	Total particulate matter	Mg. in smoke of 1 cigarette		
		Moisture	Nicotine	Tar
Run 1	9.6	0.9	0.63	8.1
Run 2	11.6	1.1	0.57	9.9
Run 3	10.7	1.1	0.65	8.9
Run 4	11.0	1.0	0.53	9.5
Average	10.7	1.0	0.60	9.1

Average number of puffs 8.2.

Pressure drops in inches of water ---------- 9.9

Percentage reduction:

Tar ---------- 57

Nicotine ---------- 55

TEST 12

[Absorbent HA-2 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	15.5	1.9	0.66	12.9
Run 2	13.4	1.8	0.63	11.0
Run 3	13.9	1.7	0.67	11.5
Run 4	14.5	1.3	0.57	12.6
Average	14.3	1.7	0.63	12.0

Average number of puffs 8.0.

Pressure drops in inches of water ---------- 7.6

Percentage reduction:

Tar ---------- 43

Nicotine ---------- 53

TEST 13

[Absorbent HA-3 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	13.9	1.3	0.83	11.8
Run 2	12.0	1.2	0.95	9.8
Run 3	12.6	1.8	0.77	10.0
Run 4	12.9	1.4	0.85	10.6
Average	12.9	1.4	0.85	10.6

Average number of puffs 8.2.

Pressure drops in inches of water ---------- 8.5

Percentage reduction:

Tar ---------- 50

Nicotine ---------- 36

TEST 14

[Absorbent HA-4 (300 milligrams per filter)]

Control no filter	Total particulate matter	Mg. in smoke of 1 cigarette		
		Moisture	Nicotine	Tar
Run 1	13.0	1.5	0.64	10.9
Run 2	12.1	1.5	0.59	10.0
Run 3	12.8	1.3	0.65	10.8
Run 4	12.1	1.3	0.65	10.0
Average	12.5	1.4	0.63	10.5

Average number of puffs 7.9.

Pressure drops in inches of water ---------- 8.8
Percentage reduction:
Tar ---------- 50
Nicotine ---------- 53

TEST 15

[Absorbent HA-5 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	7.9	0.2	0.37	7.5
Run 2	7.5	0.6	0.37	6.7
Run 3	8.0	0.9	0.41	6.3
Run 4	8.1	0.4	0.34	7.4
Average	7.9	0.5	0.37	7.0

Average number of puffs 8.0.

Pressure drops in inches of water ---------- 10.9
Percentage reduction:
Tar ---------- 75
Nicotine ---------- 72

TEST 16

[Absorbent HA-6 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	16.6	4.2	0.67	11.7
Run 2	18.6	4.3	0.60	13.7
Run 3	17.0	3.5	0.73	12.8
Run 4	17.8	4.0	0.68	13.1
Average	17.5	4.0	0.67	12.8

Average number of puffs 8.3.

Pressure drops in inches of water ---------- 6.5
Percentage reduction:
Tar ---------- 39
Nicotine ---------- 50

TEST 17

[Absorbent HA-9 (300 milligrams per filter)]

Control no filter	Total particulate matter	Moisture	Nicotine	Tar
Run 1	6.8	1.3	0.36	5.1
Run 2	7.1	1.5	0.42	5.2
Run 3	5.9	1.1	0.34	4.5
Run 4	6.9	1.8	0.33	4.8
Average	6.7	1.4	0.36	4.9

Average number of puffs 7.8.

Pressure drops in inches of water ---------- 11.2
Percentage reduction:
Tar ---------- 77
Nicotine ---------- 73

What is claimed is:

1. A filter for tobacco smoke comprising a non-tobacco filter plug shaped to fit a tobacco smoke passage, including granules of porous hydroxy apatite.

2. A filter of claim 1 wherein the hydroxy apatite is calcium hydroxy apatite.

3. A filter of claim 1 wherein the hydroxy apatite component has a particle size distribution of from about 5 to about 2,000μ, and a B.E.T. surface area greater than 50 m.^{2}/gm.

4. A filter of claim 1 wherein the hydroxy apatite component has a particle size distribution of from about 100 to about 850μ, and a B.E.T. surface area greater than 50 m.^{2}/gm.

5. A filter of claim 1 wherein the hydroxy apatite component is present in an amount of from about 1.0% to about 80% by weight based on the weight of the tobacco to be smoked.

6. A filter of claim 1 wherein the calcium hydroxy apatite component is in the form of a pellet.

7. The filter of claim 1 wherein the calcium hydroxy apatite component is utilized in combination with a fibrous material.

8. The filter of claim 7 wherein the fibrous material is cellulose acetate.

9. The filter of claim 1 wherein the porous hydroxy apatite is utilized in combination with active carbon.

10. A non-tobacco filter shaped to fit a tobacco smoke passage comprising porous hydroxy apatite having a particle size of from about 5 to about 2,000μ, and a B.E.T. surface area greater than 50 m.^{2}/gm. utilized in an amount of from 100 to 400 milligrams.

11. The method of filtering tobacco smoke to remove acidic and particulate materials comprising passing said tobacco smoke through a non-tobacco filter plug including granules of porous hydroxy apatite.

12. The method of claim 11 wherein the porous hydroxy apatite is calcium hydroxy apatite having a particle size of from about 5 to about 2,000μ, and a B.E.T. surface area of greater than 50 m.^{2}/gm.

No references cited.

MELVIN D. REIN, Primary Examiner

U.S. Cl. X.R.

131—10.7, 266

PO-1050
(5/69)

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,464,423 Dated September 2, 1969

Inventor(s) George I. Klein

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 44, "$Ca(OH_2$" (both occurrences) should read --$Ca(OH)_2$--

Column 3, line 3, "maner" should read --manner--

Column 3, line 17, "whil" should read --while--

Column 3, line 45, "maner" should reach --manner--

Column 3, line 48, "Cigarete" should read --Cigarette--

Column 4, line 13, "Eethane" should read --Ethane--

Column 5, line 64, "a" should read --at--

Column 6, line 41, "Ml." should read --Mol--

Column 6, between lines 52 and 53, the title --Sample Designatio has been omitted

The numerals "5" and "6" designating the column numbers appear t the second occurrence of "5" and "6" should read --7-- and --

Column 9, line 53, "Run 3" should read --Run 2--

Column 9, line 44, the phrase --greater than-- has been omitted before "13.0"

Column 10, line 73, "10.0" should read --10.1--

Column 11, line 11, "7.5" should read -- 7.3 --

Column 11, line 12, "6.7" should read -- 6.5 --

Column 11, line 13, "6.3" should read -- 6.7 --

SIGNED AND
SEALED
MAY 5 1970

(SEAL)
Attest:

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Commissioner of Patents