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(54) **CIGARETTE FILTER AND CIGARETTE**

(57) A cigarette filter comprises a filter plug (121) that includes a filter material containing a cellulose acetate fiber tow (122), and filtration rate control particles (124)

dispersed in the tow and selected from cellulose particles, cellulose triacetate particles and a mixture thereof. A cigarette comprises the cigarette filter.

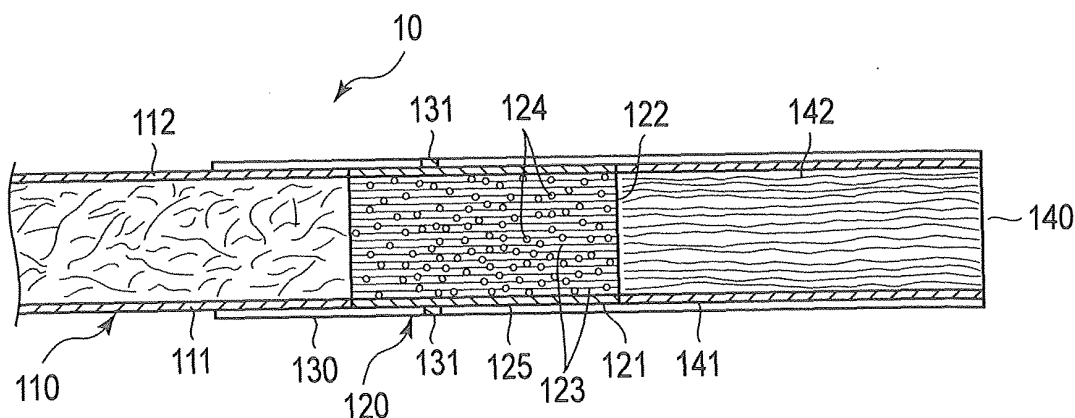


FIG. 1

Description

Technical Field

5 **[0001]** The present invention relates to a cigarette filter and a cigarette comprising the same.

Background Art

10 **[0002]** Many cigarettes comprise filters to remove various components in cigarette mainstream smoke. As the filter, a filter having a cellulose acetate fiber tow as a filter material is widely used.

[0003] The acetate filter is known to have selective filtering characteristics such that the filtration efficiency of semi-volatile components is higher than the filtration efficiency of tar in cigarette mainstream smoke. The semivolatile component is a component present in both the particulate and vapor phases of cigarette mainstream smoke, and includes a nitrogen-containing compound, ketones, and phenols. These semivolatile components have an effect on cigarette smoking taste, and thus it may be desired that the components are not significantly removed by the filter.

15 **[0004]** Patent Document 1 discloses a tobacco smoke filter which is substantially formed of cellulose acetate micro-filaments having an average diameter of 20 to 250 μm as a tobacco smoke filter for removing harmful components from tobacco smoke. Patent Document 1 also discloses that the microfilaments are mixed with a normal cellulose acetate fiber tow. However, Patent Document 1 does not teach of the semivolatile components, though it discloses that the filter is excellent in removal efficiency of tar in tobacco smoke.

Prior Art Document

Patent Document

25 **[0005]** Patent Document 1: Jpn. Pat. No. 3939823

Summary of Invention

30 Problem to be solved by the Invention

[0006] An object of the present invention is to provide a cigarette filter which does not significantly remove a semivolatile component in cigarette mainstream smoke and a cigarette comprising the same.

35 Means for solving the Problem

[0007] In order to solve the above problem, according to the first aspect of the present invention, there is provided a cigarette filter comprising a filter plug that includes a filter material containing:

40 a cellulose acetate fiber tow, and
filtration rate control particles dispersed in the tow and selected from cellulose particles, cellulose triacetate particles and a mixture thereof.

45 **[0008]** According to the second aspect of the present invention, there is provided a filter-tipped cigarette comprising:
a cigarette rod; and
the cigarette filter of the present invention which is attached to the end of the cigarette rod.

Effects of the Invention

50 **[0009]** The cigarette filter of the present invention does not significantly remove the semivolatile component in cigarette mainstream smoke.

Brief Description of Drawings

55 **[0010]**

FIG. 1 is an enlarged schematic view showing a part of a cigarette comprising a filter according to one embodiment

of the present invention.

FIG. 2 shows graphs showing relationships between the draw resistance of the control filter and each of the permeability of tar, nicotine, and typical semivolatile components in mainstream smoke.

FIG. 3 shows graphs showing relationships between the draw resistance of the filter according to the present invention and each of the permeability of tar, nicotine, and typical semivolatile components in mainstream smoke.

FIG. 4 is a graph showing a relationship between the selective filtration coefficient S_x and the draw resistance of the filter according to the present invention together with that of the control filter.

FIG. 5 is a graph showing a relationship between a plasticizer (triacetin) to be added to a filter material and the permeability of typical semivolatile components.

FIG. 6 is a graph showing a relationship between the draw resistance of the particle-containing filter and the total outer peripheral surface area of the cellulose acetate fiber.

FIG. 7 is a graph showing a relationship between the total outer peripheral surface area of the cellulose acetate fiber forming a filter plug and the permeability of typical semivolatile components.

FIG. 8A is a graph showing the permeability of typical semivolatile components of the filter which is obtained by adding cellulose triacetate particles to a cellulose acetate fiber having a total outer peripheral surface area of 223 cm² on average.

FIG. 8B is a graph showing the permeability of typical semivolatile components of the filter which is obtained by adding cellulose triacetate particles to a cellulose acetate fiber having a total outer peripheral surface area of 255 cm² on average.

FIG. 8C is a graph showing the permeability of typical semivolatile components of the filter which is obtained by adding cellulose particles to a cellulose acetate fiber having a total outer peripheral surface area of 206 cm² on average.

FIG. 9A is a schematic view showing a structure of the filter containing filtration rate control particles used in Example 7.

FIG. 9B is a schematic view showing a structure of the control filter used in Example 7.

FIG. 10 is a graph showing influences of the addition of an additive to the filtration rate control particles on the permeability of typical semivolatile components of the filter.

Mode for Carrying Out the Invention

[0011] Hereinafter, several embodiments of the present invention will be described in detail.

[0012] The cigarette filter of the present invention comprises a filter plug which includes a filter material containing a cellulose acetate fiber tow. Filtration rate control particles are dispersed in the cellulose acetate fiber tow. The term "dispersed" used herein generally means that the filtration rate control particles are almost uniformly distributed over the entire inside of the cellulose acetate fiber tow (refer to FIG. 1), and the distribution may be weighted toward the side of a cigarette mouthpiece or the side of a cigarette rod. The filtration rate control particles play a role in controlling to reduce the filter-filtration rate of the semivolatile components in cigarette mainstream smoke. The filtration rate control particles are selected from cellulose particles, cellulose triacetate particles, and a mixture thereof.

[0013] Cellulose triacetate particles have an average acetyl substitution degree of 2.76 to 3.00, preferably an average acetyl substitution degree of 2.8 to 3.0, according to the definition of Japan Chemical Fibers Association. The average acetyl substitution degree may be measured in accordance with the titration method: ASTM D871-96. The acetyl substitution degree of the cellulose acetate which is determined by the measuring method shows a normal distribution. Accordingly, it is defined as the "average acetyl substitution degree".

[0014] The cellulose acetate fibers may be bound with a plasticizer such as triacetin to form a tow. The cellulose acetate fibers are extended in parallel to one another over the total length of the filter.

[0015] The cellulose acetate fibers forming the cellulose acetate fiber tow may be cellulose acetate fibers to be used for normal cigarette filters. The cellulose acetate fibers may have a single fineness of 1.5 to 8 deniers and have a sectional shape, such as a circular shape, an oval shape, a Y-shape, an X-shape or an I-shape. The cellulose acetate fibers may be formed of cellulose acetate having an acetyl substitution degree of 2.4 to 2.5 (diacetate). The total fineness of the cellulose acetate fiber tow may be normally from 15000 to 50000 deniers. The cellulose acetate fiber tow is labeled as 1.9Y44000. This means that the single fineness is 1.9 deniers, the fiber cross section has a Y-shape, and the total fineness is 44000 deniers, as well known by those skilled in the art. In the present specification, the unit of the single fineness "denier" represents a weight of a piece of fiber per 9000 m (g/9000 m), and the unit of the total fineness "denier" represents a weight of all pieces of fiber per 9000 m (g/9000 m).

[0016] The cellulose particles hardly adsorb the semivolatile components in cigarette mainstream smoke and hardly adsorb menthol either (refer to Examples 1 and 2 below). Further, the cellulose triacetate particles hardly adsorb the semivolatile components in cigarette mainstream smoke and hardly adsorb menthol either (refer to Examples 1 and 2 below). As described above, the filtration rate control particles hardly adsorb menthol. Therefore, in the case where a cigarette filter of the present invention is applied to a menthol cigarette, there is a low possibility that menthol is significantly

adsorbed by the filter after production of the cigarette up to when it is smoked by a smoker, and the menthol content in mainstream smoke is hard to be decreased during smoking of the cigarette.

[0017] The filtration rate control particles have a granular shape. The average sphere equivalent diameter of the filtration rate control particles is preferably from 100 to 1000 μm , more preferably greater than 250 μm , from the viewpoints of the hardness and draw resistance of the filter, the filtration performance, and the easiness of production of the filter. When producing a filter containing particles having an average sphere equivalent diameter of 100 to 1000 μm , a normal charcoal filter production machine may be directly used (in that case, needless to say, the filtration rate control particles are used in place of charcoal particles). The average sphere equivalent diameter may be obtained by measuring the particle size distribution using a particle size distribution measurement device and calculating the 50% median size of the sphere equivalent diameter, as described in the following examples. The BET specific surface area of the filtration rate control particles is preferably less than 5 m^2/g . The BET specific surface area may be determined according to a well-known BET method.

[0018] The filtration rate control particles may be obtained with a compression type granulating machine. Specifically, they may be obtained with the compression type granulating machine in the following manner. First, the material of cellulose particles or cellulose triacetate particles is ground into powder. The resulting ground product and various additives are mixed with a precision mixer. Thereafter, the resulting mixture is compaction-molded using a dry granulator while applying a pressure with a roller, and a molded product (e.g., a plate-shaped product) is obtained. Subsequently, the molded product is crushed with a dry particle-size selector. At this time, it is roughly crushed at the first stage, and then it may be crushed into a desired particle size at the second stage. The resulting crushed product is passed through a sieving machine to screen granules having a predetermined particle size. As a result, the filtration rate control particles are prepared. Thus, the filtration rate control particles obtained with the compression type granulating machine are excellent in terms of high yield and fewer problems due to the mixing of long fiber during winding of the filter.

[0019] Regarding the filtration rate control particles, the cellulose triacetate particles may be obtained by grinding cellulose triacetate flakes and classifying them. Alternatively, the cellulose triacetate particles may be obtained by granulating cellulose triacetate flakes with a well-known granulating machine such as of a tumbling type, an extrusion type, a fluid-bed type, a stirring type or a compression type. Further, the cellulose particles are commercially available.

[0020] Preferably, the filtration rate control particles account for 1.5 to 30% by volume of the volume of the filter containing the filtration rate control particles. Further, when considering the production of the filter, if the volume ratio of the added particles increases, the production tends to be difficult. Thus, in order to satisfy the results of the filtration rate control and the sensory evaluation without having any effect of the particles on the production of the filter, the filtration rate control particles more preferably account for 1.5 to 16% by volume of the volume of the filter containing the filtration rate control particles (refer to Examples 3 and 4 below). Such ratio of the filtration rate control particles can achieve a draw resistance of 35 mm H_2O to 180 mm H_2O , which is considered to be suitable for the draw resistance of a filter having a circumference of 24.5 mm and a length of 25 mm. A volume V of the filter may be determined by the equation $V = \pi r^2 L$, where r represents a radius of the filter and L represents a length of the filter (in this regard, the thickness of the filter wrapping paper is of a level small enough to be ignored). The addition weight and the apparent density obtained using a mercury porosimeter were used to calculate the volume of the particles.

[0021] If the filtration rate control particles are added to the cellulose acetate fiber tow, the hardness of the resulting filter plug increases. Therefore, triacetin as the plasticizer does not need to be added. Even when triacetin is added as the plasticizer, the addition amount of triacetin as the plasticizer may be decreased. For example, when the filtration rate control particles are added to the cellulose acetate fiber tow at the above ratio, a sufficient hardness of the filter plug is obtained by adding triacetin in an amount of 3% by weight or less based on the cellulose acetate fiber tow or not adding (refer to Example 1 below). In this regard, the hardness of the filter plug may be expressed as the amount of strain of the filter plug when an indenter having a diameter of 12 mm is pressed against the filter plug under a 300 g loading for 10 seconds. As the amount of strain is smaller, the filter plug is harder.

[0022] Besides the addition of the filtration rate control particles to the filter, the addition amount of the plasticizer is decreased or the plasticizer is not added to the filter. As a result, the permeability of semivolatile components can be further improved (refer to Example 6 below).

[0023] Further, if the filtration rate control particles are added to the cellulose acetate fiber tow, the total outer peripheral surface area of the cellulose acetate fiber can be decreased by 10% or more (usually, 30% or less), as compared to the case where the filtration rate control particles are not added. As a result, the permeability of semivolatile components is further improved (refer to Examples 4 and 5 below).

[0024] Further, if a filter obtained by adding the filtration rate control particles to the cellulose acetate fiber tow is used for the cigarette, the smoking flavor can be changed as compared with the case where a filter not containing the filtration rate control particles is used for the cigarette (refer to Example 3 below).

[0025] In order to obtain a more preferable cigarette smoking flavor, a small amount of an additive may be added to the filtration rate control particles. Example 7 below demonstrates that even if an additive contributing to cigarette smoking flavor is added to the filtration rate control particles, no influence is given to the selective permeation of the semivolatile

components. The additive may be a component for smoking flavor (e.g., a flavorant) or a component having an effect on the smoking flavor (e.g., a humectant, amino acid, polysaccharide or dietary fiber). Both the components are collectively referred to as a "component contributing to smoking flavor". The addition amount of the component contributing to smoking flavor is preferably 10% by weight or less, and more preferably 5% by weight or less, based on the total weight of the particles (the total weight of the filtration rate control particles and the component contributing to smoking flavor). Examples of the component contributing to smoking flavor include the following flavorants, humectants, amino acids, polysaccharides, and dietary fibers.

[0026] The flavorants may be synthetic flavorants, natural flavorants, essential oils, and the like. Further, they may be used regardless of lipophilicity or hydrophilicity. Examples of lipophilic flavorants include vanillin, ethyl vanillin, guarlinalool, thymol, methyl salicylate, linalool, eugenol, menthol, clove, anise, cinnamon, bergamot oil, geranium, lemon oil, spearmint, and ginger. Examples of hydrophilic flavorants include glycerin, propylene glycol, ethyl acetate, and isoamyl alcohol.

[0027] Examples of humectants include:

polyols including:

diols [e.g., an alkanediol (e.g., a C₂₋₁₀ alkanediol such as ethylene glycol, propylene glycol, trimethylene glycol, 1,3-butanediol, 1,4-butanediol, 1,5-pentanediol or hexylene glycol, preferably a C₂₋₈ alkanediol, more preferably a C₂₋₆ alkanediol, particularly a C₂₋₄ alkanediol), and polyalkylene glycol (e.g., diethylene glycol, dipropylene glycol, triethylene glycol or tripropylene glycol)],

triols [e.g., an alkane triol (e.g., a C₃₋₁₀ alkane triol such as glycerin or 1,2,6-hexanetriol, preferably a C₃₋₆ alkane triol, more preferably a C₃₋₄ alkane triol)], and

polyols having a tetrafunctional or higher functionality [e.g., a polymer of a polyol (e.g., alkane triols) having a trifunctional or higher functionality (e.g., polyglycerol such as diglycerol or triglycerol)]; and

derivatives of these polyols [e.g., dialkylene glycol monoalkyl ether (e.g., methylcarbitol and ethylcarbitol), and (poly)alkylene glycol monoacylate (e.g., ethylene glycol monoacetate)].

[0028] Examples of amino acids include amino acids and salts thereof (amino acid salts). The amino acid may be any of a neutral amino acid (monoamino monocarboxylic acid etc.), an acidic amino acid (monoamino dicarboxylic acid etc.), and a basic amino acid (diaminomono-carboxylic acid etc.), or may be a sulfur-containing amino acid. The amino acid may be an α -amino acid, β -amino acid, γ -amino acid, or the like. Particularly, it may be an α -amino acid. The amino acid may be either an optically active form (D-form, L-form, etc.) or a racemate. Further, examples of amino acids include polyamino acids having a low polymerization degree (e.g., a polymerization degree of 2 to 9, preferably a polymerization degree of 2 to 5, more preferably a polymerization degree of 2 to 3). The amino acid may have a substituent or may be an amino acid derivative in which at least a part of the carboxyl group(s) or the amino group(s) is derivatized. For example, at least a part of the carboxyl group(s) in the amino acid may be a derivatized carboxyl group (e.g., an amide group).

[0029] Examples of a typical amino acid include:

an aliphatic amino acid [e.g., an aliphatic monoamino carboxylic acid such as glycine, alanine, isoleucine, leucine, valine, threonine, serine, asparagine, aminosuccinic acid, cysteine, methionine, glutamine or glutamic acid (e.g., an amino-C₂₋₂₀-alkane carboxylic acid, preferably an amino-C₂₋₁₂-alkane carboxylic acid, more preferably an amino-C₂₋₈-alkane carboxylic acid), an aliphatic polyaminocarboxylic acid such as lysine, hydroxylysine, arginine or cystine (e.g., a polyamino-C₂₋₂₀-alkane carboxylic acid, preferably a polyamino-C₂₋₁₂-alkane carboxylic acid)];

an aromatic amino acid (e.g., an aryl-C₂₋₂₀-alkane carboxylic acid such as phenylalanine or tyrosine, preferably a C₆₋₁₀-aryl-C₂₋₁₂-alkane carboxylic acid);

a heterocyclic amino acid (e.g., tryptophan, histidine, proline or 4-hydroxyproline); and

a polypeptide obtained by the polymerization of these amino acids at a low polymerization degree (e.g., at a polymerization degree of 9 or less) (e.g., glycyl glycine, glutamyl glycine, glycyl glycyl glycine, and glycyl proline). Further,

examples of amino acid salts include a metal salt [e.g., an alkali metal salt (e.g., a sodium salt such as sodium glutamate)], a hydrochloride (e.g., arginine hydrochloride), and a salt of amino acids (e.g., a salt of lysine with glutamic acid).

[0030] Other examples of components contributing to smoking flavor include food additives such as xylitol and mannitol; polymers such as lignin; and polysaccharides or dietary fibers such as cellulose, carboxymethyl cellulose, hydroxypropyl methylcellulose, chitin, starch, glycogen, guar gum, glucomannan, sodium alginate, agarose, chitosan, pectin, carrageenan, and xanthan gum.

[0031] Further, a dye may be added to the filtration rate control particles. As the dye, for example, a natural dye

extracted from gardenia, safflower, turmeric, annatto, red pepper, paprika, red yeast rice, red cabbage, cacao or the like may be added. The dye may be added in an amount of 0.1 to 5% by weight based on the total weight of the particles (total weight of the filtration rate control particles and the dye). Preferably, it may be added in an amount of 1% by weight or less. The filtration rate control particles containing the dye may have various colors depending on the color of the dye. When the dye is added to the filtration rate control particles and a transparent tipping paper is used to produce a filter, the filtration rate control particles (granules) with which the filter is loaded can be confirmed from the outside. It is known that human feelings can be influenced by color. Accordingly, it is expected that a new feeling is provided to the smoking taste of the cigarette by the color of the dye. Further, when the color of the filtration rate control particles is different from that of the filter fiber, as with charcoal filter, it is possible to easily distinguish between the filtration rate control particles and the filter fiber in the quality inspection in manufacturing the filter.

[0032] The filter of the present invention may be attached to one end of a cigarette rod singly or in combination with another filter plug. The latter example is shown in FIG. 1.

[0033] FIG. 1 is a schematic view of a cigarette 10 comprising the filter according to one embodiment of the present invention. The cigarette 10 comprises a cigarette rod 110 and a filter 120 which is provided at an end in the axial direction of the cigarette rod 110 in such a manner that the end surface of the filter comes in contact with the end surface of the cigarette rod. The cigarette rod 110 includes a tobacco filler 112 such as tobacco shreds wrapped in a cigarette paper 111. The filter 120 comprises a filter plug 121 of the present invention which includes a tow 122 of a lot of cellulose acetate fibers 123 which are disposed along the axial direction of the filter 120 and may be bundled with a plasticizer such as triacetin. Each of the cellulose acetate fibers 123 is extended over the total length of the filter plug 121. The filtration rate control particles 124 are dispersed in the cellulose acetate fiber tow 122. The filter plug 121 is wrapped in a filter wrapping paper 125. The cigarette rod 110 and the filter 120 are connected with a tipping paper 130, similarly to a normal filter-tipped cigarette. A plurality of ventilation holes 131 may be punched in the tipping paper 130 in one or more rows in the circumferential direction of the filter. A so-called acetate plain filter plug 140 of a cellulose acetate fiber tow 142 wrapped in a filter wrapping paper 141 may be attached to the posterior end of the filter 120 including the filtration rate control particles (to the direction of smoke inhalation). In this case, the filter plug 140 is also wrapped in the tipping paper 130.

Examples

[0034] Hereinafter, the present invention will be described with reference to the examples.

Example 1

<Preparation of filtration rate control particles>

1. Cellulose triacetate particles

[0035] Cellulose triacetate flakes (average acetyl substitution degree: 2.86) were purchased from Daicel Chemical Industries, Ltd. The acetyl substitution degree of the cellulose triacetate flakes was measured in accordance with the titration method: ASTM D871-96. Then, the above-mentioned acetyl substitution degree was confirmed. Subsequently, the cellulose triacetate flakes were ground with a coffee mill (MK-52M manufactured by Matsushita Electric Industrial Co., Ltd.). The ground product was classified through a sieve with an electromagnetic sieve shaker (AS200 control, manufactured by Retsch) to obtain particles at mesh intervals of 300 to 710 μm . As for the particle size distribution, the 50% median size of the sphere equivalent diameter was calculated as the average particle size using a digital-image-analysis type particle size distribution meter (manufactured by Retsch, (sold at HORIBA, Ltd.)). Regarding the resulting particles, the average sphere equivalent diameter was 550 μm , the bulk density was 0.54 g/cc, the apparent density obtained by using a mercury porosimeter was 0.71 g/cc, and the BET specific surface area obtained by nitrogen desorption method was 4.6 m^2/g .

2. Cellulose particles

[0036] Cellulose particles were cellulose beads obtained by refining and dissolving wood and forming the resulting viscose into a granular and porous form, and those marketed under the trademark of Viscop Pearl in Rengo Co., Ltd. were used. Regarding the used particles, the average sphere equivalent diameter was 400 μm , the bulk density was 0.20 g/cc, the apparent density obtained by using a mercury porosimeter was 0.34 g/cc, and the BET specific surface area was below the detection limit.

<Production of filter plug>

[0037] The cellulose particles or cellulose triacetate particles were added to an acetate filter containing triacetin in the same manner as a generally known method for producing a charcoal filter. Further, plain acetate filters not containing filtration rate control particles were also produced. As a filter wrapping paper for each filter plug, one having a basis weight of 24.0 ± 1.5 g/m², a thickness of 60 ± 5 μ m, and an air permeability of $10,000 \pm 1,800$ Coresta unit was used. The diameter of each of the filter plugs was 7.7 mm and the length was 120 mm. The draw resistance of each of the filters was measured according to ISO6565: 2002.

[0038] The hardness of the resulting filter plugs was measured as the amount of strain of the filter plug when an indenter having a diameter of 12 mm was pressed against the filter plug under 300 g loading for 10 seconds.

[0039] The specification and hardness of the resulting filter plugs are shown in Tables 1A to 1C below.

[Table 1A]

Table 1A: Filters containing cellulose triacetate particles					
Filter Nos.	Specification of cellulose acetate fiber tow	Amount of triacetin (% by weight)	Draw resistance per length of 120 mm (mmH ₂ O)	Addition amount of particles per filter length of 10 mm (mg/10mm)	Filter hardness (mm)
A-1	3.5Y/35000	6	418	35	0.71
A-2	5.9Y/35000	6	319	35	0.54
A-3	2.2Y/35000	6	570	35	0.82
A-4	3.5Y/35000	9	396	35	0.63
A-5	3.5Y/35000	0	420	35	1.2
A-6	5.9Y/35000	6	423	70	0.42

[Table 1B]

Table 1B: Filters containing cellulose particles					
Filter Nos.	Specification of cellulose acetate fiber tow	Amount of triacetin (% by weight)	Draw resistance per length of 120 mm (mmH ₂ O)	Addition amount of particles per filter length of 10 mm (mg/10mm)	Filter hardness (mm)
B-1	5.9Y/35000	6	429	35	0.52
B-2	5.5Y/31000	6	337	35	0.77
B-3	2.2Y/35000	6	763	35	0.77
B-4	5.9Y/35000	9	432	35	0.48
B-5	5.9Y/35000	0	439	35	0.97
B-6	2.5Y/35000	6	430	10	0.97

[Table 1C]

Table 1C: Acetate plain filters					
Filter Nos.	Specification of cellulose acetate fiber tow	Amount of triacetin (% by weight)	Draw resistance per length of 120 mm (mmH ₂ O)	Addition amount of particles per filter length of 10 mm (mg/10mm)	Filter hardness (mm)
AF-1	2.2Y/35000	0	470	-	1.6
AF-2	5.9Y/35000	0	197	-	1.6
AF-3	2.2Y/35000	6	433	-	1.0
AF-4	5.5Y/31000	6	169	-	1.1

[0040] The hardness of the filters (A-1 to A-6) containing cellulose triacetate particles was from 0.42 to 1.2 (mm). The hardness of the filters (B-1 to B-6) containing cellulose particles was from 0.48 to 0.97 (mm). On the other hand, in the acetate plain filters, the hardness of the acetate plain filters (AF-1 and AF-2) not containing triacetin was 1.6 (mm). Therefore, it was found that the hardness of the filters is increased by the inclusion of the particles in the filters containing cellulose particles and the filters containing cellulose triacetate particles. When the addition amount of the particles is increased, the hardness of the filters can be guaranteed regardless of the addition of triacetin.

<Production of cigarette sample>

[0041] The filter of the commercially available filter-tipped cigarette "Mild Seven Aqua Squash Menthol" was removed. The resultant cigarette rod was connected to a filter for evaluation obtained by cutting the above-produced filter plug into various lengths and placing it in a paper tube (outer diameter: 7.7 mm) with an adhesive tape to produce a cigarette sample. The length and draw resistance of each of the cut filters are shown in Tables 2A to 2C below. It took a month to perform the smoking test after the production of the filters.

[Table 2A]

Table 2A: Cut filters containing cellulose triacetate particles			
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Marks of cigarettes comprising cut filters
A-1	20	72	CA-1
A-2	20	53	CA-2
A-3	20	93	CA-3
A-4	20	69	CA-4
A-5	20	71	CA-5
A-6	20	70	CA-6

[Table 2B]

Table 2B: Cut filters containing cellulose particles			
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Marks of cigarettes comprising cut filters
B-1	20	72	CB-1
B-2	20	56	CB-2
B-3	20	129	CB-3
B-4	20	73	CB-4
B-5	20	75	CB-5
B-6	20	71	CB-6

[Table 2C]

Table 2C: Cut filters containing acetate plain filters			
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Marks of cigarettes comprising cut filters
AF-3	10	38	CAF-3-1
AF-3	15	55	CAF-3-2
AF-3	20	69	CAF-3-3
AF-3	30	104	CAF-3-4

(continued)

Table 2C: Cut filters containing acetate plain filters

Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Marks of cigarettes comprising cut filters
AF-3	40	133	CAF-3-5
AF-4	10	14	CAF-4-1
AF-4	15	22	CAF-4-2
AF-4	20	28	CAF-4-3
AF-4	30	38	CAF-4-4
AF-4	40	55	CAF-4-5

<Smoking test>

[0042] Ten cigarette samples produced in the above process (ventilation holes were closed with an adhesive tape) were automatically smoked using an automatic smoking machine (RM20D, manufactured by Borgwaldt KC Inc.) under the following conditions: puff volume: 35.0 mL/2 sec, puff duration: 2 sec/puff, puff interval: 1 puff/min. The particulate matter in cigarette smoke was collected with a Cambridge filter (CM-133, manufactured by Borgwaldt KC Inc.). The smoke passed through the Cambridge filter was collected on 10 mL of methanol cooled to 70°C with a coolant of dry ice and isopropanol.

[0043] The Cambridge filter having the particulate matter collected, 10 mL of a methanol solution containing the cigarette smoke collected, and 1 mL of an internal standard solution (d-32 pentadecane: 0.05 mg/mL, d-1-ethanol: 150 mL/L, anethole: 2 mL/L, 1,3-butanediol: 4 mL/L) were added to a serum bottle, which was shaken for 30 minutes. After shaking, the supernatant liquid was collected and used as a sample for analysis. The above operation was also performed on the cigarette rod (control cigarette) obtained by removing the filter of the commercially available filter-tipped cigarette "Mild Seven Aqua Squash Menthol".

<Analysis of tar, nicotine, and semivolatile components>

[0044] The sample for analysis was analyzed by gas chromatography-mass spectrometry (GC-MSD). Agilent 7890A (Agilent Technologies Inc.) was used for GC, and Agilent 5975C (Agilent Technologies Inc.) was used for MSD.

[0045] The peak area of each component (standardized by the internal standard) in the chromatogram obtained by the analysis was compared to the peak area of each component in the chromatogram regarding the control cigarette. The permeability "1-E_x" of each smoke component through each filter was calculated using the following formula.

[Numerical Formula 1]

$$1 - E_x = 1 - \frac{(A_{x,in} - A_{x,out})}{A_{x,in}}$$

[0046] In the above formula, "A_{x, in}" and "A_{x, out}" represent values obtained by standardizing the peak areas of a component "x" in smoke of the control cigarette and each of the filter-tipped cigarette sample, respectively, by the internal standard. "E_x" represents a filtration rate of the component "x".

[0047] As for the semivolatile components, 3-furaldehyde, 2-acetyl furan, and furfural were selected as typical semivolatile components. The average permeability of the components was calculated, and the selective filtration performance of the semivolatile components was evaluated.

[0048] Regarding the cigarettes CAF-3-1 to CAF-3-5 and CAF-4-1 to CAF-4-5, the draw resistance of each filter is represented on the horizontal axis, and the logarithm values of the permeability of tar, nicotine, and the typical semivolatile components are plotted on the vertical axis. These are shown in FIG. 2. FIG. 2(A) shows the permeability of tar, FIG. 2(B) shows the permeability of nicotine, and FIG. 2(C) shows the permeability of the typical semivolatile components.

In FIGS. 2(A) to 2(C), the circles relate to CAF-3-1 to CAF-3-5, and the triangles relate to CAF-4-1 to CAF-4-5.

[0049] Regarding the tar (FIG. 2(A)) and the nicotine (FIG. 2(B)), the permeability can be linearly approximated regardless of the kind of the cellulose acetate tow fiber. Regarding the typical semivolatile components (FIG. 2(C)), the permeability has a different slope depending on the kind of the tow fiber (the diameter of the fiber). This shows that the permeability of tar and nicotine is regulated by only the draw resistance of each filter; however, the permeability behavior of the typical semivolatile components varies depending on the kind of the tow. Since the tar and nicotine are basically particulate phase components, the filtration efficiency may be represented as a function of the draw resistance of each filter. On the other hand, the semivolatile components are distributed to both the vapor phase and the particulate phase, and thus the permeability behavior is influenced by filtration of the particulate phase component and absorption of the vapor phase component to the fiber. As a result, it is not regulated by only the draw resistance.

[0050] Subsequently, the results of the cigarettes CA-1 to CA-5 and CB-1 to CB-5 are shown in FIG. 3. FIG. 3(A) shows the permeability of tar, FIG. 3(B) shows the permeability of nicotine, and FIG. 3(C) shows the permeability of the typical semivolatile components. In FIGS. 3(A) and 3(B), a line 'a' relates to CAF-3-1 to CAF-3-3 and CAF-4-4 to CAF-4-5, a line 'b' relates to the cigarettes CA-1 to CA-5, and a line 'c' relates to the cigarettes CB-1 to CB-5. In FIG. 3(C), a line 'a' relates to CAF-3-1 to CAF-3-5 and a line 'b' relates to CAF-4-1 to CAF-4-5.

[0051] The results shown in FIGS. 3(A) and 3(B) show that the filter containing the filtration rate control particles of the present invention has a high permeability of tar and nicotine as compared to that of the acetate plain filter, in other words, it has a low filtration rate of tar and nicotine. From the results shown in FIG. 3(C), it is found that the filter containing the filtration rate control particles of the present invention has a high permeability of the semivolatile components as compared to the acetate plain filter. That is, the filtration rate control particles of the present invention allow the permeation of the semivolatile components to be significantly improved. On the other hand, the present inventors have demonstrated that the filter containing the cellulose diacetate particles (average acetyl substitution degree: 2.4 to 2.5) has a function in filtrating the semivolatile components and hardly allows the semivolatile components to be permeated as compared to the acetate plain filter.

[0052] From the results shown in FIG. 3(A), the filter containing the filtration rate control particles of the present invention has a different filtration rate of tar from that of the acetate plain filter. In order to reveal the filtering characteristics of the semivolatile components in comparison with tar, the selective filtration coefficient S_x was calculated by the following formula as an indicator showing the component selectivity in filtration of the typical semivolatile components.

[Numerical Formula 2]

$$S_x = \frac{(1 - E_{TPM})}{(1 - E_x)}$$

[0053] In the above formula, E_{TPM} represents the filtration rate of crude tar (the total particulate matter).

[0054] The selective filtration coefficient S_x thus calculated was plotted against the filter draw resistance. The results are shown in FIG. 4. In FIG. 4, a line 'a' relates to the cigarettes CAF-4-1 to CAF-4-5, and a line 'b' relates to CAF-3-1 to CAF-3-5. The results shown in FIG. 4 show the following. When comparing the filters having the same single fineness, the selective filtration coefficient of the filter containing the filtration rate control particles of the present invention is decreased as compared to that of the acetate plain filter in any case of filter draw resistance. Even if the filtration rate of tar is taken into consideration, the semivolatile components are permeated selectively.

[0055] Subsequently, in order to examine the effect of the plasticizer (triacetin (GTA)) added to the filter material on the permeability of the typical semivolatile components, as for the cigarette CA-1 (triacetin: 6% by weight), the cigarette CA-4 (triacetin: 9% by weight), the cigarette CA-5 (triacetin: 0%), the cigarette CB-1 (triacetin: 6% by weight), the cigarette CB-4 (triacetin: 9% by weight), and the cigarette CB-5 (triacetin: 0%), the selective filtration coefficient S_x of the typical semivolatile components was plotted against the draw resistance. The results are shown in FIG. 5. As shown in FIG. 5, if the addition amount of triacetin is small, it acts in the direction where the semivolatile components are permeated. If the addition amount of triacetin is large, it acts in the direction where the semivolatile components are filtrated. The permeability of the typical semivolatile components can also be controlled by the amount of triacetin. This is because the vapor of the semivolatile components is absorbed (adsorbed) into triacetin on the surface of the cellulose acetate fiber. It is considered that if the addition amount of triacetin is decreased, the adsorption amount of the semivolatile components decreases. In the filter containing the filtration rate control particles of the present invention, even if the addition amount of triacetin is decreased, the filter hardness can be guaranteed. Therefore, a filter which allows the semivolatile components to be more selectively permeated can be achieved by decreasing the addition amount of triacetin.

[0056] When the above results are summarized, it was found that the filter containing the filtration rate control particles of the present invention is effective in the selective permeation of the semivolatile components in view of the design of the filter and the easiness in production, although the selective permeation of the semivolatile components can also be controlled by the diameter of cellulose acetate fibers forming a tow and the amount of triacetin.

Example 2

[0057] When the filter of the present invention is intended to be used for a menthol tobacco product, if the adsorption of menthol to the filtration rate control particles of the present invention is continued, the amount of menthol in mainstream smoke is decreased. Therefore, the adsorption amount of menthol to the filtration rate control particles of the present invention was examined.

[0058] Specifically, an acetate plain filter (cellulose acetate fiber tow: 1.9Y44000, weight: 30 mg, filter length: 5 mm) was attached to an end of a cigarette rod (circumference: 24.9 mm, length: 59 mm) having 640 mg of tobacco shreds containing 0.59% by weight of menthol. A mentholated acetate filter (cellulose acetate fiber tow: 2.5Y35000, weight: 90 mg, filter length: 15 mm) in which a menthol-containing string was threaded through the center of the filter was arranged at a distance of 5 mm from the acetate plain filter. The cavity between both the filters was filled with the filtration rate control particles of the present invention (cellulose particles or cellulose triacetate particles prepared in Example 1). The content of menthol in the mentholated acetate filter was 1.99% by weight. A cigarette whose cavity is filled with cellulose particles as the filtration rate control particles is designated as a cigarette A, while a cigarette whose cavity is filled with cellulose triacetate particles as the filtration rate control particles is designated as a cigarette B.

[0059] The cigarettes thus produced were placed in a glass bottle and sealed, followed by storage at 50°C for two weeks. The cigarette samples after the storage were taken out from the glass bottle. Then, the content of menthol in each of tobacco shreds, acetate plain filters, filtration rate control particles, and mentholated acetate filters was quantified. The content of menthol was quantified by performing methanol-extraction of the samples to be measured (tobacco shreds, acetate plain filters, filtration rate control particles or mentholated acetate filters) and analyzing the methanol-extract with a gas chromatograph (6890 series, manufactured by HEWLETT PACKARD). In the preliminary test, it was confirmed that menthol was extracted with methanol.

[0060] The content of menthol in each of tobacco shreds, acetate plain filters, filtration rate control particles, and mentholated acetate filters is shown in Table 3 below.

[Table 3]

Table 3: Content of menthol (unit: % by weight)					
		Tobacco shreds	Acetate plain filter	Filtration rate control particles	Mentholated acetate filters
Before storage		0.59	0	0	1.99
After storage	Cigarette A	0.56	2.03	Cellulose particles 0.20	1.97
	Cigarette B	0.43	2.30	Cellulose acetate particles 0.22	2.23

[0061] From the results shown in Table 3, it is found that the filtration rate control particles of the present invention (cellulose particles and cellulose triacetate particles) hardly adsorb menthol. Therefore, even if the filter of the present invention containing the filtration rate control particles is used for a menthol tobacco product, the undesired adsorption of menthol to the filtration rate control particles is hardly caused, and thus the menthol smoking taste can be sufficiently experienced.

Example 3

[0062] Filter-tipped cigarettes having the structure shown in FIG. 1 were produced. The tipping paper having ventilation holes punched and a filter were removed from the commercially available filter-tipped cigarette "Mild Seven Aqua Squash Menthol 7 Box", and the obtained cigarette rod was used as the cigarette rod 110 (content of menthol in tobacco shreds: 0.55% by weight). Each of the filters A-1 and A-6 shown in Table 1A, the filters B-1 and B-6 shown in Table 1B, and the filter AF-3 shown in Table 1C was cut into a length of 10 mm so that the filter draw resistance is 35 ± 2 mm H₂O so as to almost constantly keep the amounts of tar, nicotine, and menthol in cigarette mainstream smoke, and the obtained filter plugs were used as the filter plug 121. The acetate plain filter 140 (cellulose acetate fiber tow: 5.0Y35000 (containing 6.9% by weight of triacetin); filter length: 17 mm, menthol content: 2.22% by weight) was attached to the rear end of the

filter plug 121. The filter plugs 121 and 140 were connected to the cigarette rod using the tipping paper. It took three months to perform the smoking test after the production of the filters. For confirmation, the specification of the filter plug 121 is shown in Table 4 below.

[Table 4]

Table 4: Cut filters			
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Marks of cigarettes comprising cut filters
A-1	10	35±2	CA-1-2
A-6	10	35±2	CA-6-2
B-1	10	35±2	CB-1-2
B-6	10	35±2	CB-6-2
AF-3	10	35±2	CAF-3-1-2

[0063] Among the cigarettes comprising the filters shown in Table 4, five each of the cigarettes (CA-6-2 and CB-1-2) containing a large amount of the filtration rate control particles in the filters thereof and five of the control cigarette (CAF-3-1-2) were smoked without closing the ventilation holes under the smoking conditions described in Example 1. The amounts of tar, nicotine, and menthol in mainstream smoke were measured and the puff number was also measured. The measurement results (averages) of the amounts of tar, nicotine, and menthol per cigarette as well as the puff number are shown in Table 5 below.

[Table 5]

Table 5: Amounts of tar, nicotine and menthol in mainstream smoke per cigarette, as well as puff number				
Cigarette	Tar (mg)	Nicotine (mg)	Menthol (mg)	Puff number
CAF-3-1-2	7.9	0.55	0.40	6.9
CB-1-2	8.8	0.62	0.45	6.9
CA-6-2	8.9	0.64	0.45	6.9

[0064] Subsequently, nine evaluation panelists evaluated the smoking taste of the cigarettes. The evaluation panelists felt the difference in smoking taste between the cigarettes CB-1-2 and CB-6-2 and the control cigarette CAF-3-1-2. As for the characteristics of the smoking flavor of the cigarettes CB-1-2 and CB-6-2, they detected menthol strongly and clearly.

[0065] They also felt the difference in smoking taste between the cigarettes CA-1-2 and CA-6-2 and the control cigarette CAF-3-1-2. As for the characteristics of the smoking flavor of the cigarettes CA-1-2 and CA-6-2, they detected menthol strongly and clearly. Further, as for the smoking flavor of the cigarettes CA-1-2 and CA-6-2, they sharply detected the smoking flavor, hardly detected a left bitter taste, and detected a strong tobacco-like flavor. After smoking, they lost the taste immediately and hardly had a strong aftertaste.

Example 4

[0066] Acetate plain filter plugs of various draw resistances (filter length: 10 mm; draw resistance: 14 to 58 mm H₂O) and filter plugs containing the filtration rate control particles of the present invention shown in Table 6 below were produced, and the draw resistance thereof was measured. Regarding the cellulose acetate fiber tows in the produced filters, the total of the outer peripheral surface areas of each single fiber (the total outer peripheral surface area) was calculated in the following manner based on the article (Kazuo Maeda "Research on Development of Tobacco Smoke Filter"), Central Research Institute, Japan Tobacco Inc., December 1983, pages 27 to 30).

<Total outer peripheral surface area of cellulose acetate>

[0067]

(1) The circle equivalent diameter of the single fiber is calculated from the following equation.

$$\text{Circle equivalent diameter} = 11.91 \times (\text{single fineness} / \text{fiber density})^{1/2}$$

In the above equation, the fiber density is 1.32 g/cm³.

(2) Subsequently, the shape coefficient of the cross section of the single fiber is calculated from the following equation.

$$\text{Shape coefficient} = \frac{\text{actual fiber outer peripheral length}}{(4 \times \pi \times \text{actual cross-sectional area})^{1/2}}$$

In the above equation, the actual fiber outer peripheral length and the actual cross-sectional area are actually measured from a photomicrograph of the cross section of the single fiber.

(3) Subsequently, the outer peripheral length of the single fiber is calculated from the following equation.

$$\text{Outer peripheral length of single fiber} = \pi \times \text{circle equivalent diameter} \times (\text{shape coefficient})^{1/2}$$

(4) Finally, the total outer peripheral surface area is determined from the following equation.

$$\text{Total outer peripheral surface area} = \text{outer peripheral length of single fiber} \times \text{number of single fiber} \times \text{fiber length}$$

[0068] In the above equation, the fiber length is calculated by removing the amount of triacetin from the actually measured weight of the acetate tow to obtain a net weight of the acetate tow and dividing the net weight of the acetate tow by a weight obtained by converting the total fineness into a weight per filter length.

Table 6: Specification of filters									
Filtration rate control particles	Particle size (μm)	Addition amount of particles per length of 10 cm (mg/10cm)	Specification of cellulose acetate fiber tow	Total outer peripheral surface area of cellulose acetate fiber per filter length of 10 cm (cm ² /10cm)	Addition amount of triacetin (% by weight)	Filter length (mm)	Fiber length (mm)	Draw resistance (mmH ₂ O)	percentage of particles to volume of filters (% by volume)
Cellulose particle	400	35	5.5Y31000	80	6	10	11.5	28	22.3
	400	35	5.9Y35000	87	6	10	11.8	36	22.3
	400	35	2.2Y35000	123	6	10	11.4	65	22.3
	400	35	5.9Y35000	87	0	10	12.2	38	22.3
	400	25	5.9Y35000	87	6	10	11.9	28	15.9
	400	25	5.9Y35000	87	0	10	12.0	28	15.9
	400	25	5.9Y35000	87	3	10	11.9	28	15.9
	700	25	5.9Y35000	87	6	10	12.1	27	15.9
	400	35	5.9Y35000	87	6	10	12.8	27	10.7
	400	35	3.5Y35000	108	6	10	12.2	36	10.7
Cellulose acetate particle	400	35	2.2Y35000	124	6	10	11.6	46	10.7
	400	70	5.9Y35000	87	6	10	12.3	35	21.3
	400	35	3.5Y35000	108	0	10	12.3	35	10.7
	400	35	5.0Y35000	92	6	10	12.1	26	10.7
	400	35	5.0Y35000	92	0	10	12.1	26	10.7
	400	35	5.0Y35000	92	3	10	12.0	26	10.7
	400	35	5.0Y35000	92	3	10	12.0	26	10.7
	400	35	5.0Y35000	92	3	10	12.0	26	10.7

[0069] The results are shown in Table 6.

[0070] Regarding the results shown in Table 6, the draw resistance of each filter is plotted against the total outer peripheral surface area of each cellulose acetate fiber, and the results are shown in FIG. 6. In FIG. 6, the black rhombuses relate to acetate plain filter plugs, the white triangles relate to filter plugs containing cellulose particles, and the white squares relate to filter plugs containing cellulose triacetate particles. FIG. 6 shows that, in any case of the draw resistance, the filter plugs containing the filtration rate control particles of the present invention have a smaller total outer peripheral surface area of the cellulose acetate fiber as compared to that of the acetate plain filter plugs. In the filter plugs containing the filtration rate control particles of the present invention, a reduction rate of the total outer peripheral surface area of the cellulose acetate fiber is about from 10 to 30%. Therefore, in the case where the filtration rate control particles of the present invention are added, a cellulose acetate fiber tow having a smaller total outer peripheral surface area can be used as compared to the case where the particles are not added, in producing filter plugs having the same draw resistance.

Example 5

[0071] Regarding the cigarettes CA-1 to CA-6, CB-1 to CB-6, CAF-3-1 to CAF-3-5 and CAF-4-1 to CAF-4-5 (refer to Tables 2A to 2C), and the cigarettes comprising the filter plugs shown in Tables 7A and 7B below, the total outer peripheral surface area of the cellulose acetate fiber and the permeability and selective permeation coefficient of the typical semi-volatile components were calculated. The results are shown in Tables 8A and 8B below.

[Table 7A]

Table 7A: Filters containing cellulose particles (average particle size: 700 μm)						
Filter Nos.	Specification of cellulose acetate fiber tow	Amount of triacetin (% by weight)	Filter length (mm)	Draw resistance (mmH_2O)	Addition amount of particles per filter length of 10 mm ($\text{mg}/10\text{mm}$)	Marks of cigarettes comprising corresponding filters
B2-1	5.9Y/35000	6	20	72	35	CB2-1
B2-2	2.2Y/35000	6	20	117	35	CB2-2
B2-3	5.9Y/35000	9	20	71	35	CB2-3
B2-4	5.9Y/35000	0	20	74	35	CB2-4
B2-5	2.5Y/35000	6	20	70	10	CB2-5

[Table 7B]

Table 7B: Acetate plain filters			
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH_2O)	Marks of cigarettes comprising cut filters
AF-1	10	40	CAF-1-1
AF-1	15	58	CAF-1-2
AF-1	20	76	CAF-1-3
AF-1	30	112	CAF-1-4
AF-1	40	146	CAF-1-5
AF-2	10	18	CAF-2-1
AF-2	15	26	CAF-2-2
AF-2	20	35	CAF-2-3
AF-2	30	53	CAF-2-4
AF-2	40	68	CAF-2-5

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[Table 8A]

Table 8A			
Cigarette	Total outer peripheral surface area of cellulose acetate fiber (cm ²)	Permeability of typical semivolatile components	Selective permeation coefficient of typical semivolatile components
CA-1	214	0.17	3.78
CA-2	174	0.17	4.57
CA-3	247	0.10	5.60
CA-4	214	0.09	7.30
CA-5	214	0.28	2.38
CA-6	174	0.15	4.43
CB-1	174	0.21	3.42
CB-2	160	0.16	4.84
CB-3	247	0.08	6.87
CB-4	174	0.09	7.32
CB-5	174	0.47	1.51
CB-6	245	0.13	4.64
CB2-1	174	0.18	3.84
CB2-2	247	0.13	4.08
CB2-3	174	0.15	4.69
CB2-4	174	0.45	1.56
CB2-5	245	0.17	3.63

[Table 8B]

Table 8B			
Cigarette	Total outer peripheral surface area of cellulose acetate fiber (cm ²)	Permeability of typical semivolatile components	Selective permeation coefficient of typical semivolatile components
CAF-3-1	124	0.35	2.25
CAF-3-2	186	0.20	3.34
CAF-3-3	247	0.15	3.56
CAF-3-4	371	0.04	11.39
CAF-3-5	495	0.03	12.03
CAF-4-1	80	0.51	1.80
CAF-4-2	120	0.37	2.32
CAF-4-3	160	0.23	3.44
CAF-4-4	240	0.11	6.06
CAF-4-5	320	0.06	9.69
CAF-1-1	124	0.58	1.34
CAF-1-2	186	0.40	1.76

(continued)

Table 8B

Cigarette	Total outer peripheral surface area of cellulose acetate fiber (cm ²)	Permeability of typical semivolatile components	Selective permeation coefficient of typical semivolatile components
CAF-1-3	247	0.30	2.05
CAF-1-4	371	0.19	2.49
CAF-1-5	495	0.09	3.77
CAF-2-1	87	0.68	1.29
CAF-2-2	131	0.61	1.40
CAF-2-3	174	0.54	1.49
CAF-2-4	262	0.37	1.88
CAF-2-5	349	0.25	2.28

[0072] Regarding the results shown in Tables 8A and 8B, the permeability of the typical semivolatile components is plotted against the total outer peripheral surface area of the cellulose acetate fiber, and the results are shown in FIG. 7. In FIG. 7, the white triangles relate to the cigarettes CB-5 and CB2-4, the white squares relate to the cigarette CA-5, the white circles relate to the cigarettes CAF-1-1 to CAF-1-5, and the white rhombuses relate to the cigarettes CAF-2-1 to CAF-2-5. The plasticizer is not contained in the filter plugs of any of the above-mentioned cigarettes. In FIG. 7, the black triangles relate to the cigarettes CB-1, CB-2, CB-3, CB-6, CB2-1, CB2-2, and CB2-5, the black squares relate to the cigarettes CA-1, CA-2, CA-3, and CA-6, the black circles relate to the cigarettes CAF-3-1 to CAF-3-5, and the black rhombuses relate to the cigarettes CAF-4-1 to CAF-4-5. The plasticizer is contained in the filter plugs of all the above-mentioned cigarettes.

[0073] From FIG. 7, it is found that the permeability of the typical semivolatile components in each of the acetate plain filter plugs is largely dependent on the addition amount of the plasticizer (triacetin); however, in the case of the same amount of the plasticizer, the permeability of the typical semivolatile components is determined by the total outer peripheral surface area of the cellulose acetate fiber. Further, it is found that the particle size of cellulose particles has no influence on the filtering characteristics of the semivolatile components. These facts are attributed to the fact that cellulose particles and cellulose triacetate particles are not easily dissolved in an acetone solvent and hardly adsorb menthol or triacetin, and thus they have a low adsorbing ability for semivolatile components having a polarity close to that of menthol or triacetin.

[0074] FIG. 7 shows that the selective filtering characteristics of the semivolatile components can be controlled by changing the kind of the cellulose acetate fiber tow or the addition amount of the plasticizer. However, since a certain level of hardness of the filter needs to be guaranteed and the draw resistance needs to be controlled when setting the amounts of tar and nicotine in mainstream smoke, it is not possible to freely combine the kind of the cellulose acetate fiber tow and the addition amount of the plasticizer for cigarette products. However, if the filtration rate control particles (cellulose particles and/or cellulose triacetate particles) are added to a filter according to the present invention, the constant hardness of the filter can be maintained by the presence of the particles even if the addition amount of triacetin is decreased, and the draw resistance can be controlled by the addition of the particles in order to adjust the amount of tar/nicotine in mainstream smoke to a desired value. Therefore, the cellulose acetate fiber tow regarded as unusable in the prior art (i.e., a cellulose acetate fiber tow whose total outer peripheral surface area is small) can be used according to the present invention. In other words, generally, as the total outer peripheral surface area of the cellulose acetate fiber becomes smaller, the draw resistance of the filter plug tends to decrease. Thus, a cellulose acetate fiber tow having the draw resistance lower than that of the cellulose acetate fiber tow of the conventional cigarette filter can be used. As a result, it is possible to develop a filter having characteristics in which the semivolatile components are permeated selectively as compared to tar.

[0075] As described above, it is found that, if the filtration rate control particles (cellulose particles and/or cellulose triacetate particles) are added to a filter according to the present invention, the control width of the permeation of the semivolatile components can be extended, which has been limited in the conventional acetate plain filter in view of product design or production. Thus, the present invention is effective in providing new tobacco smoking flavor or new menthol tobacco smoking flavor.

Example 6

[0076] The cellulose powder (FMC Biopolymer, trade name: Endurance MCC) and the cellulose triacetate powder (Daicel Chemical Industries, Ltd., trade name: Acetate Flake DS2.9 LT-55 (TAC)) were used as raw materials. Each powder was pressurized at 20 Mpa for 10 minutes using a tablet molding machine (manufactured by Jasco Corporation) and a hydraulic hand pump (manufactured by Riken Seiki Co., Ltd.) to obtain a plate-shaped molded product. Subsequently, the resulting plate-shaped molded product was ground with a coffee mill (MK-52M manufactured by Matsushita Electric Industrial Co., Ltd.). The ground product was classified through a sieve with an electromagnetic sieve shaker (AS200 control, manufactured by Retsch) to produce filtration rate control particles at mesh intervals of 300 to 710 μm . The resulting filtration rate control particles were used to produce filters containing cellulose triacetate particles and filters containing cellulose particles according to the same procedure as Example 1. The draw resistance and hardness of the produced filters were measured according to the same procedure as Example 1.

[0077] The specification and hardness of the resulting filter plugs are shown in Tables 9A and 9B below.

[Table 9A]

Table 9A: Filters containing cellulose triacetate particles					
Filter Nos.	Specification of cellulose acetate fiber tow	Addition amount of triacetin (% by weight)	Draw resistance per length of 120 mm (mmH_2O)	Addition amount of particles per filter length of 10 mm ($\text{mg}/10\text{mm}$)	Filter hardness (mm)
A-7	5.0Y35000	6	311	35	0.54
A-8	5.0Y35000	3	307	35	0.70
A-9	5.0Y35000	0	314	35	1.2
A-10	3.5Y35000	6	327	29	0.75
A-11	3.5Y35000	3	332	29	1.0
A-12	3.5Y35000	1	322	29	1.3

[Table 9B]

Table 9B: Filters containing cellulose particles					
Filter Nos.	Specification of cellulose acetate fiber tow	Addition amount of triacetin (% by weight)	Draw resistance per length of 120 mm (mmH_2O)	Addition amount of particles per filter length of 10 mm ($\text{mg}/10\text{mm}$)	Filter hardness (mm)
B-7	5.9Y35000	6	339	25	0.52
B-8	5.9Y35000	3	334	25	0.7
B-9	5.9Y35000	0	331	25	1.0
B-10	5.0Y35000	6	343	21	0.65
B-11	5.0Y35000	1	321	21	1.1

[0078] In Tables 9A and 9B, the addition amount of triacetin is expressed in percent by weight based on the cellulose acetate fiber tow.

[0079] The hardness of the filters (A-7 to A-12) containing cellulose triacetate particles was from 0.54 to 1.3 (mm). The hardness of the filters (B-7 to B-12) containing cellulose particles was from 0.52 to 1.1 (mm). On the other hand, in the acetate plain filters, the hardness of the acetate plain filters (AF-1 and AF-2) not containing triacetin was 1.6 (mm). Therefore, it was found that the hardness of the filters is increased by the inclusion of the particles in the filters containing cellulose particles and the filters containing cellulose triacetate particles. When the addition amount of the particles is increased, the hardness of the filters can be guaranteed even if the addition amount of triacetin is decreased. Specifically, it was found that when 3% by weight or less of triacetin was added or even when triacetin was not added, the sufficient hardness of the filter plugs was obtained.

<Production of cigarette samples>

[0080] The filter of commercially available filter-tipped cigarette "Seven Stars Solid Menthol" was removed. The resultant cigarette rod was connected to a filter for evaluation obtained by cutting the above-produced filter plug into various lengths and placing it in a paper tube (outer diameter: 7.7 mm) with an adhesive tape to produce a cigarette sample. It took two months to perform the smoking test after the production of the filters. The length and draw resistance of each of the cut filters as well as the total outer peripheral surface area of the cellulose acetate fiber are shown in Tables 10A and 10B below.

[Table 10A]

Table 10A: Cut filters containing cellulose triacetate particles				
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Total outer peripheral surface area of cellulose acetate fiber (cm ²)	Marks of cigarettes comprising cut filters
A-7	20	54	223	CA-7
A-8	20	53	222	CA-8
A-9	20	54	224	CA-9
A-10	20	53	255	CA-10
A-11	20	54	256	CA-11
A-12	20	56	255	CA-12

[Table 10B]

Table 10B: Cut filters containing cellulose particles				
Filter plug Nos. (before cutting)	Filter length after cutting (mm)	Draw resistance (mmH ₂ O)	Total outer peripheral surface area of cellulose acetate fiber (cm ²)	Marks of cigarettes comprising cut filters
B-7	20	56	202	CB-7
B-8	20	57	202	CB-8
B-9	20	59	204	CB-9
B-10	20	62	207	CB-10
B-11	20	58	214	CB-11

[0081] The produced cigarettes CA-7 to CA-12 and CB-7 to CB-11 were subjected to the smoking test according to the same procedure as Example 1. The smoking test was also performed on the cigarette rod (control cigarette) obtained by removing the filter of the commercially available filter-tipped cigarette "Seven Stars Solid Menthol".

[0082] The sample for analysis obtained in the smoking test was used to analyze tar, nicotine, and the semivolatile components according to the same procedure as Example 1. The permeability (%) and the selective filtration coefficient (%) were calculated according to the same numerical formulae described in Example 1.

[0083] The permeability and selective filtration coefficient of the typical semivolatile components of the cigarettes CA-7 to CA-12 and CB-7 to CB-11 are shown in Tables 11A to 11C below with respect to each total outer peripheral surface area of the cellulose acetate fiber.

[Table 11A]

Table 11A: Permeability and selective filtration coefficient of typical semivolatile components			
Filter	CA-7	CA-8	CA-9
Permeability (%)	20	28	32
Selective permeation coefficient (%)	316	244	193

[Table 11B]

Table 11B: Permeability and selective filtration coefficient of typical semivolatile components			
Filter	CA-10	CA-11	CA-12
Permeability (%)	27	30	35
Selective permeation coefficient (%)	252	220	192

[Table 11C]

Table 11C: Permeability and selective filtration coefficient of typical semivolatile components					
Filter	CB-7	CB-8	CB-9	CB-10	CB-11
Permeability (%) Selective permeation coefficient (%)	26 285	31 221	44 159	28 256	42 162

[0084] The permeability of the typical semivolatile components of the cigarettes CA-7 to CA-12 and CB-7 to CB-11 is shown in FIGS. 8A to 8C with respect to each total outer peripheral surface area of the cellulose acetate fiber.

[0085] FIG. 8A shows the permeability of the typical semivolatile components of the filters containing cellulose triacetate particles in the case where the total outer peripheral surface area of the cellulose acetate fiber is 223 cm² (the specification of the fiber tow: 5.0Y35000). FIG. 8B shows the permeability of the typical semivolatile components of the filters containing cellulose triacetate particles in the case where the total outer peripheral surface area of the cellulose acetate fiber is 255 cm² (the specification of the fiber tow: 3.5Y35000). FIG. 8C shows the permeability of the typical semivolatile components of the filters containing cellulose particles in the case where the total outer peripheral surface area of the cellulose acetate fiber is 206 cm² (the specification of the fiber tow: 5.9Y35000 and 5.0Y35000).

[0086] From the results of Tables 11A to 11C and FIGS. 8A to 8C, it is confirmed that when the filters having almost the same total outer peripheral surface area of the cellulose acetate fiber are compared to one another, the permeability of the typical semivolatile components is high in the filters having a small addition amount of the plasticizer. This is because the amount of adsorption of the semivolatile components to the surface of the cellulose acetate fiber is increased by the plasticizer. Therefore, the permeation of the semivolatile components can be further improved by decreasing the addition amount of the plasticizer or not adding the plasticizer, in addition to adding the filtration rate control particles (i.e., cellulose triacetate particles or cellulose particles) to a filter.

Example 7

[0087] In order to examine the influences of the additives on the filtration performance of the filtration rate control particles, cellulose particles and the cellulose particles containing the additives described in Table 12 below were prepared. The cellulose particles were prepared by the same procedure as Example 6. The cellulose particles containing the additives were prepared by mixing the additives and cellulose powder according to the same procedure as Example 6. The additives were added in amounts described in Table 12. In Table 12, the "addition amount" is represented by a ratio (% by weight) of the additives to the total weight of the filtration rate control particles and the additives. The filtration rate control particles not containing the additives were also prepared for comparison.

[Table 12]

Table 12: Additive-added filtration rate control particles		
Filtration rate control particle Nos.	Additives	Addition amount (%)
A-1	-	-
B-1	L-arginine	5
B-2	L-alanine	5
B-3	L-glutamic acid	5
B-4	Lignin	10
B-5	Powder sugar	2

(continued)

Table 12: Additive-added filtration rate control particles		
Filtration rate control particle Nos.	Additives	Addition amount (%)
B-6	Pectin	10
B-7	Sorbitol	5

<Production of filter plug>

[0088] In order to evaluate the filtration performance of the filtration rate control particles, the filtration rate control particles A-1 and B-1 to B-7 were used to produce filter plugs F-A-1 and F-B-1 to F-B-7. As shown in FIG. 9A, the filtration rate control particles were arranged in a paper tube having a length of 25 mm. Acetate filters (5 mm) were placed at the front side (the side of a tobacco rod) and the rear side (the side of a filter mouthpiece) of the filtration rate control particles so as to cover the filtration rate control particles. Thus, filters filled with the filtration rate control particles were produced. The addition amounts of the filtration rate control particles are shown in Table 13. As shown in FIG. 9B, a filter plug F-C-1 having two acetate filters (5 mm) arranged in a paper tube having a length of 25 mm was produced as a control. The specification of each of the filters is shown in Table 13.

[Table 13]

Table 13: Specification of filters				
Filter Nos.	Filtration rate control particle Nos.	Addition amount (mg)	Draw resistance of filter (mmH ₂ O)	Draw resistance of filtration rate control particles (mmH ₂ O)
F-A-1	A-1	100	68	54
F-B-1	B-1	100	55	41
F-B-2	B-2	100	53	39
F-B-3	B-3	100	51	37
F-B-4	B-4	100	50	36
F-B-5	B-5	100	64	50
F-B-6	B-6	100	69	55
F-B-7	B-7	100	53	39
F-C-1	-	-	14	-

<Production of test cigarette>

[0089] The commercially available filter-tipped cigarettes "Seven Stars Solid Menthol" were used for the preparation of test cigarettes. The filter of the commercially available cigarettes was removed. The resultant cigarette rod was connected to each of the filters (F-A-1, F-B-1 to F-B-7, and F-C-1) to produce test cigarettes. In this regard, the filter ventilation was set to 0. The cigarette prepared by using the filter F-C-1 is a control cigarette.

<Smoking test>

[0090] The produced cigarettes were subjected to the smoking test according to the same procedure as Example 1. The sample for analysis obtained in the smoking test was used to analyze tar, nicotine, and the semivolatile components according to the same procedure as Example 1. The permeability (%) and the selective filtration coefficient (%) were calculated according to the same numerical formulae described in Example 1.

[0091] The permeability and selective filtration coefficient of the typical semivolatile components of the cigarettes obtained by using the filters F-A-1 and F-B-1 to F-B-7 are shown in Table 14 below.

[Table 14]

Table 14: Permeability and selective filtration coefficient of typical semivolatile components								
Filter	F-A-1	F-B-1	F-B-2	F-B-3	F-B-4	F-B-5	F-B-6	F-B-7
Permeability (%)	68	67	73	72	74	69	68	72
Selective permeation coefficient (%)	98	105	98	100	101	100	98	99

[0092] The permeability of the typical semivolatile components of the cigarettes obtained by using the filters F-A-1 and F-B-1 to F-B-7 is shown in FIG. 10.

[0093] From the results of Table 14 and FIG. 10, it can be confirmed that the permeability of the semivolatile components by the filtration rate control particles contained in each filter is constant regardless of the presence or absence of the additive or the kind of the additive. From this result, the following could be confirmed. Even if the additive contributing to cigarette smoking flavor is added to the filtration rate control particles, a small amount thereof does not have any influence on the filtering characteristics of the filtration rate control particles.

List of Reference Signs

[0094] 10 ... cigarette, 110 ... cigarette rod, 111 ... cigarette paper, 112 ... tobacco filler, 120 ... filter, 121 ... filter plug, 122 ... cellulose acetate fiber tow, 123 ... cellulose acetate fiber, 124 ... filtration rate control particles, 125 ... filter wrapping paper, 130 ... tipping paper, 131 ... ventilation hole, 140 ... acetate plain filter plug, 141 ... filter wrapping paper, 142 ... cellulose acetate fiber tow

Claims

1. A cigarette filter comprising a filter plug that includes a filter material containing:
 - a cellulose acetate fiber tow, and
 - filtration rate control particles dispersed in the tow and selected from cellulose particles, cellulose triacetate particles and a mixture thereof.
2. The cigarette filter according to claim 1, **characterized in that** the cellulose triacetate particles have an average acetyl substitution degree of 2.8 to 3.0.
3. The cigarette filter according to claim 1 or 2, **characterized in that** the filtration rate control particles account for 1.5 to 30% by volume of the total volume of the filter plug.
4. The cigarette filter according to any one of claims 1 to 3, **characterized in that** the filtration rate control particles account for 1.5 to 16% by volume of the total volume of the filter plug.
5. The cigarette filter according to any one of claims 1 to 4, **characterized in that** the total outer peripheral surface area of the cellulose acetate fiber forming the tow is decreased by 10% or more, as compared to the total outer peripheral surface area of the cellulose acetate fiber forming the corresponding filter plug not containing the filtration rate control particles.
6. The cigarette filter according to any one of claims 1 to 5, **characterized in that** the filtration rate control particles are obtained with a compression type granulating machine.
7. The cigarette filter according to any one of claims 1 to 6, **characterized in that** the filtration rate control particles have a sphere equivalent diameter of 100 μm to 1000 μm .
8. The cigarette filter according to any one of claims 1 to 7, **characterized in that** a plasticizer is added to the cellulose acetate fiber tow in an amount of 3% by weight or less based on the cellulose acetate fiber tow, or not added.
9. The cigarette filter according to any one of claims 1 to 8, **characterized in that** the filtration rate control particles contain a component contributing to smoking flavor, and the component contributing to smoking flavor is added in

an amount of 10% by weight or less based on the total amount of the particles.

10. The cigarette filter according to any one of claims 1 to 8, **characterized in that** the filtration rate control particles contain a dye.

11. A filter-tipped cigarette comprising:

a cigarette rod; and

the cigarette filter according to any one of claims 1 to 10 which is attached to the end of the cigarette rod.

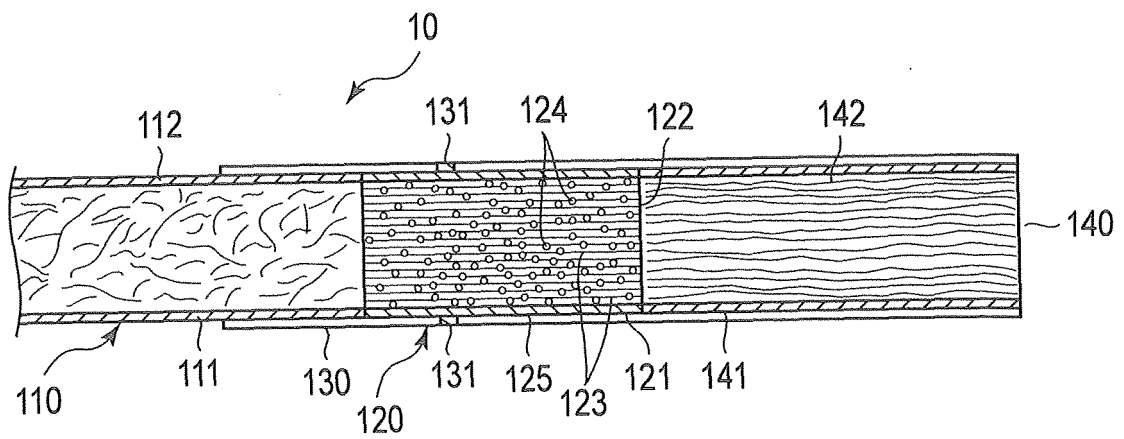


FIG. 1

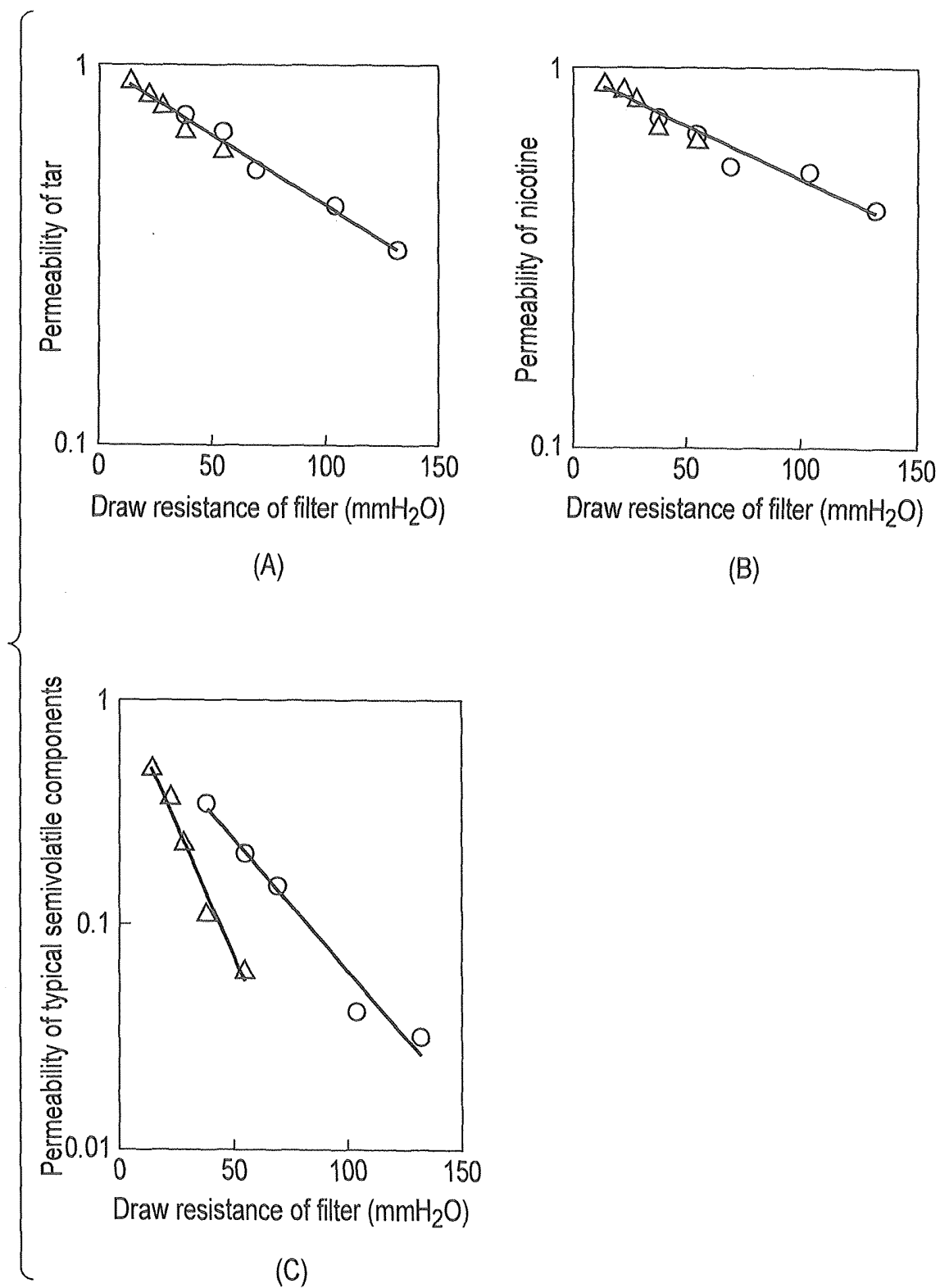


FIG. 2

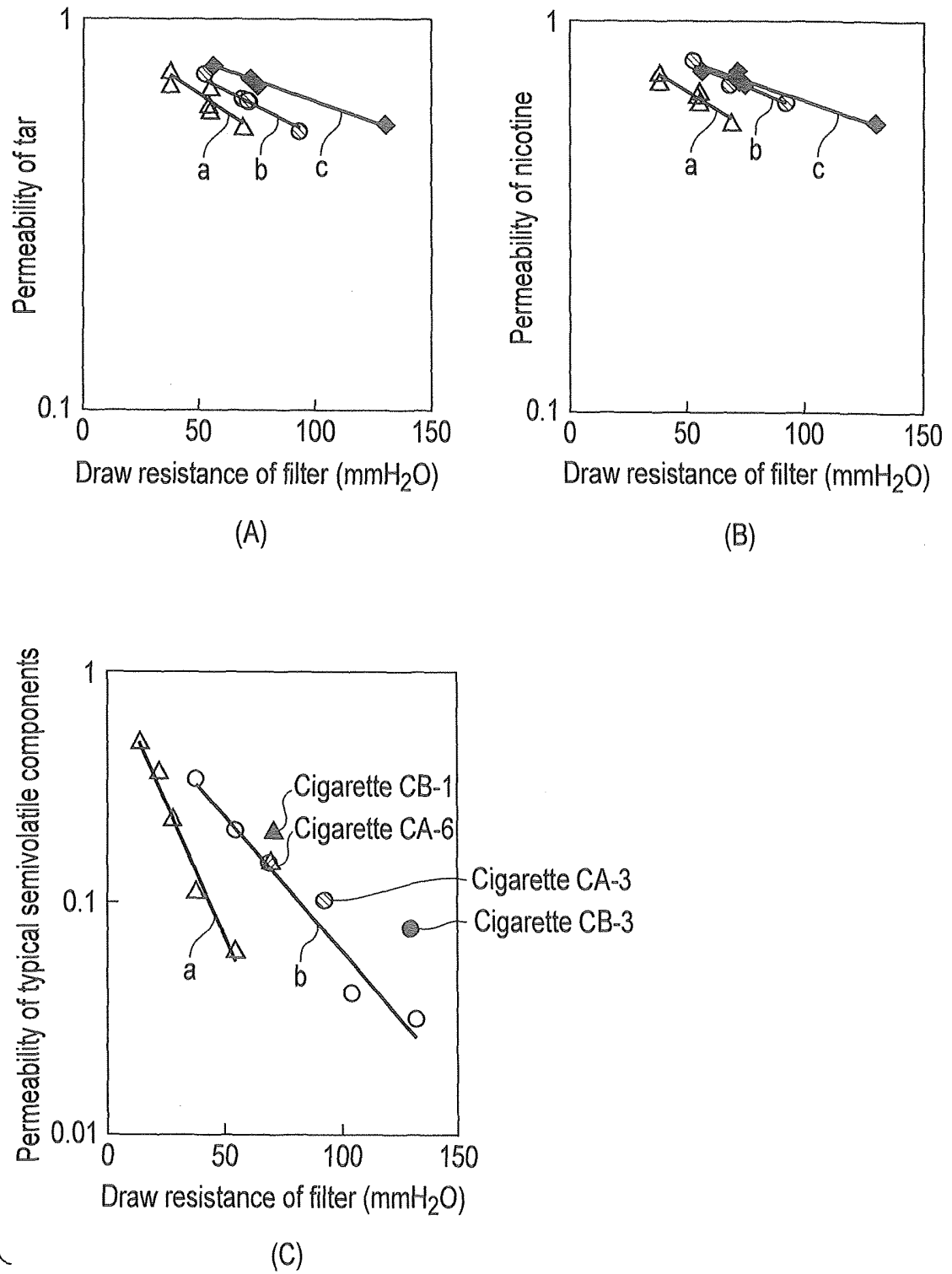


FIG. 3

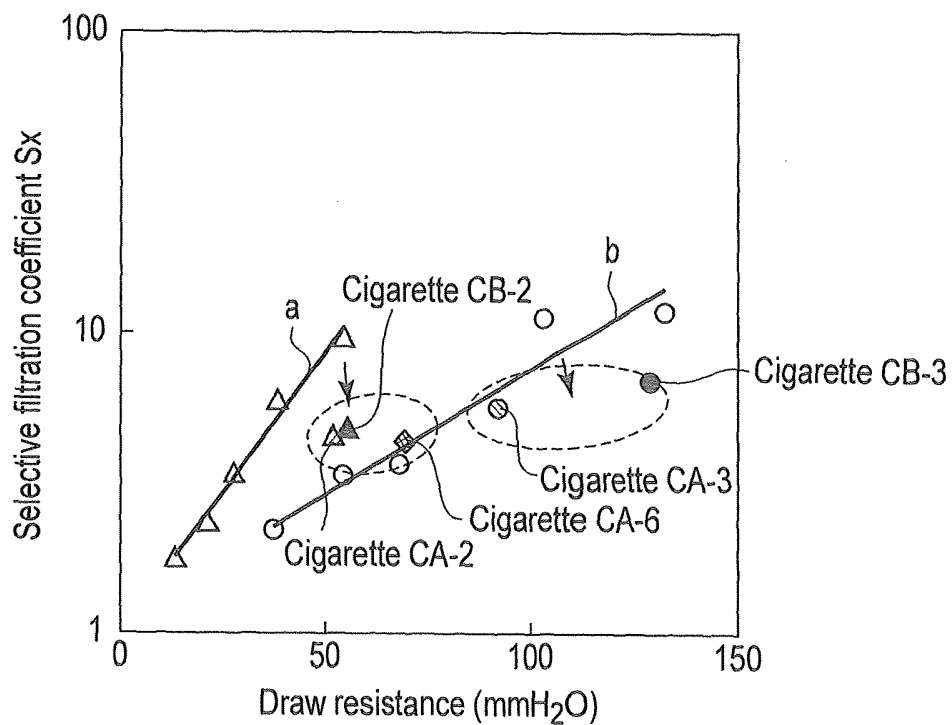


FIG. 4

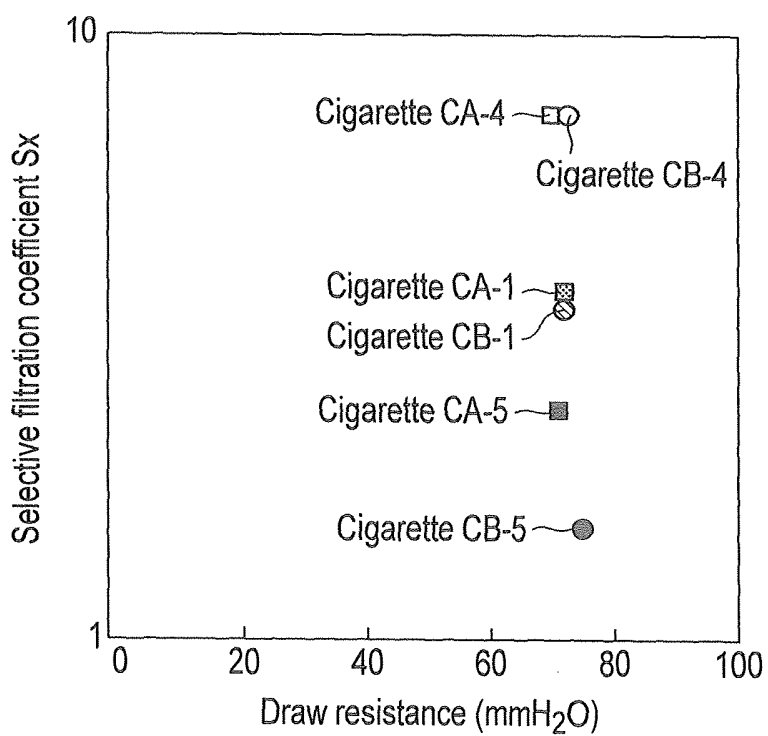


FIG. 5

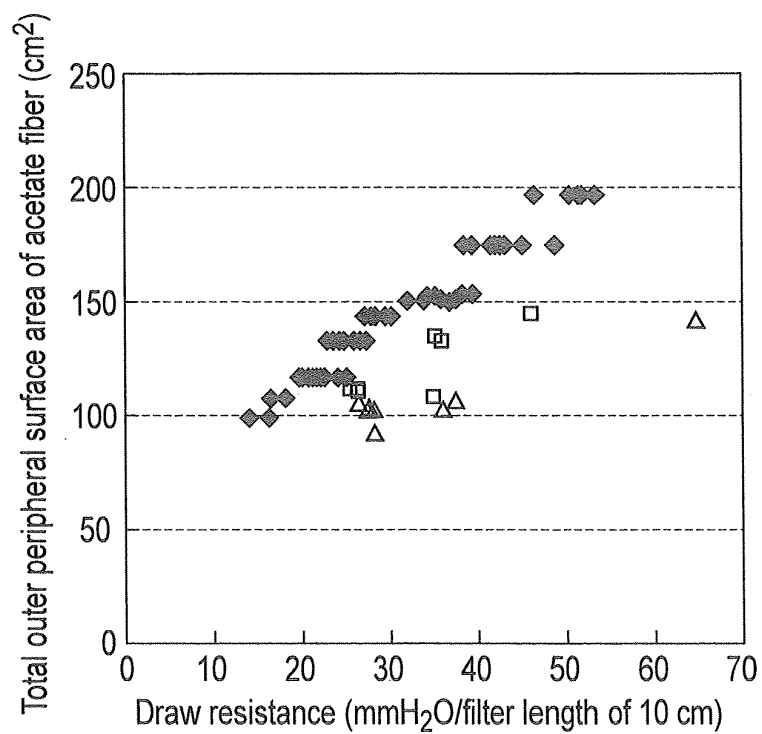


FIG. 6

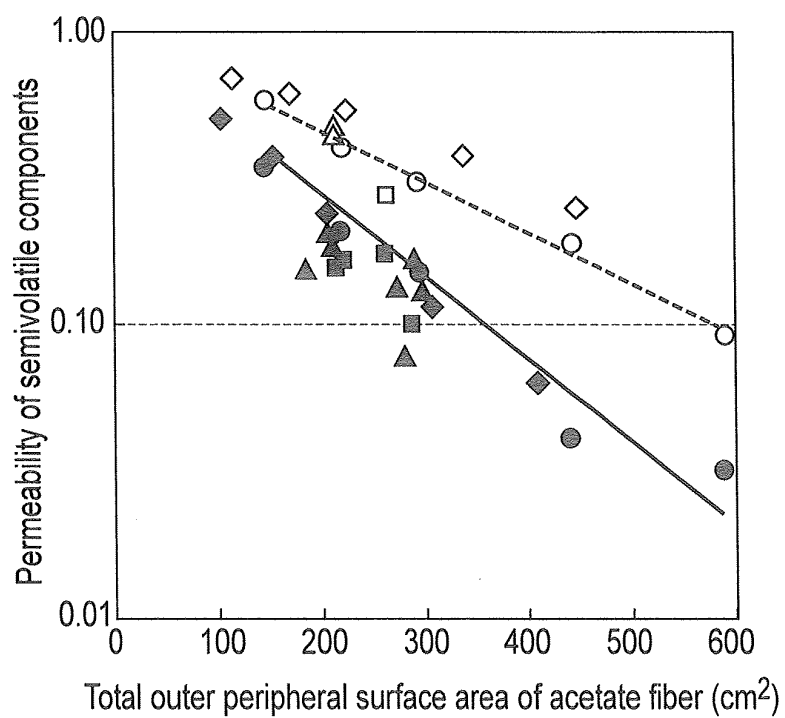


FIG. 7

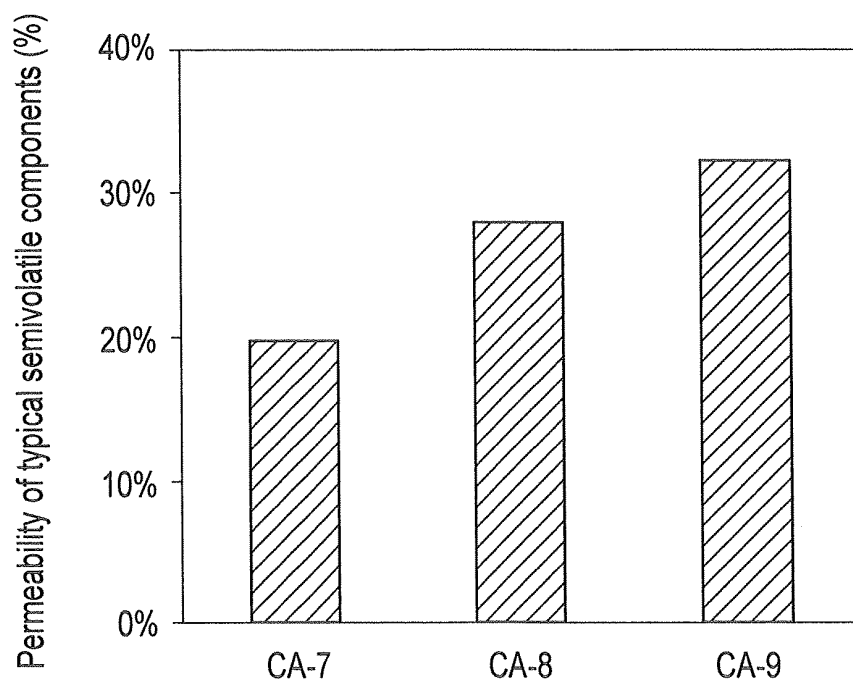


FIG. 8A

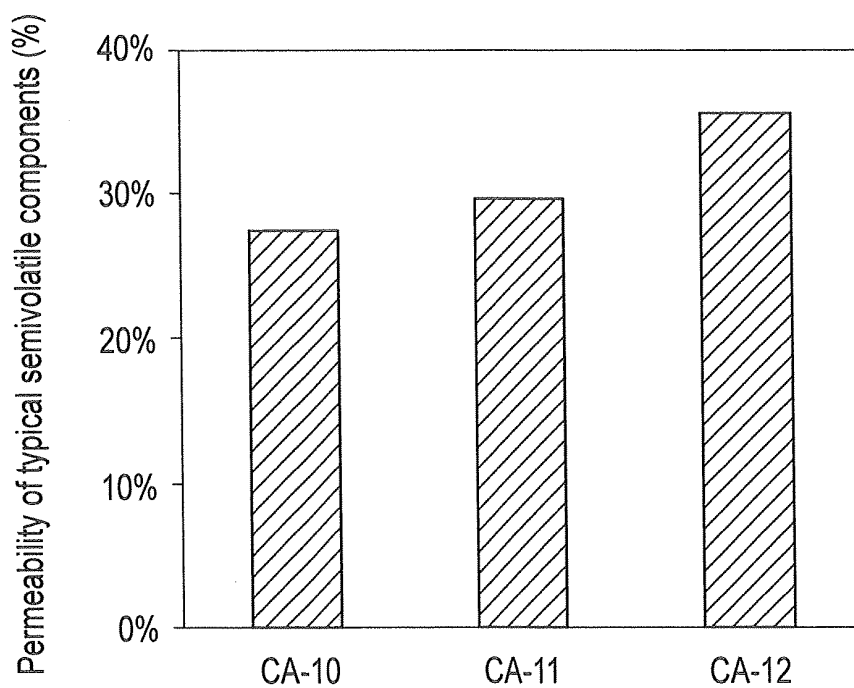


FIG. 8B

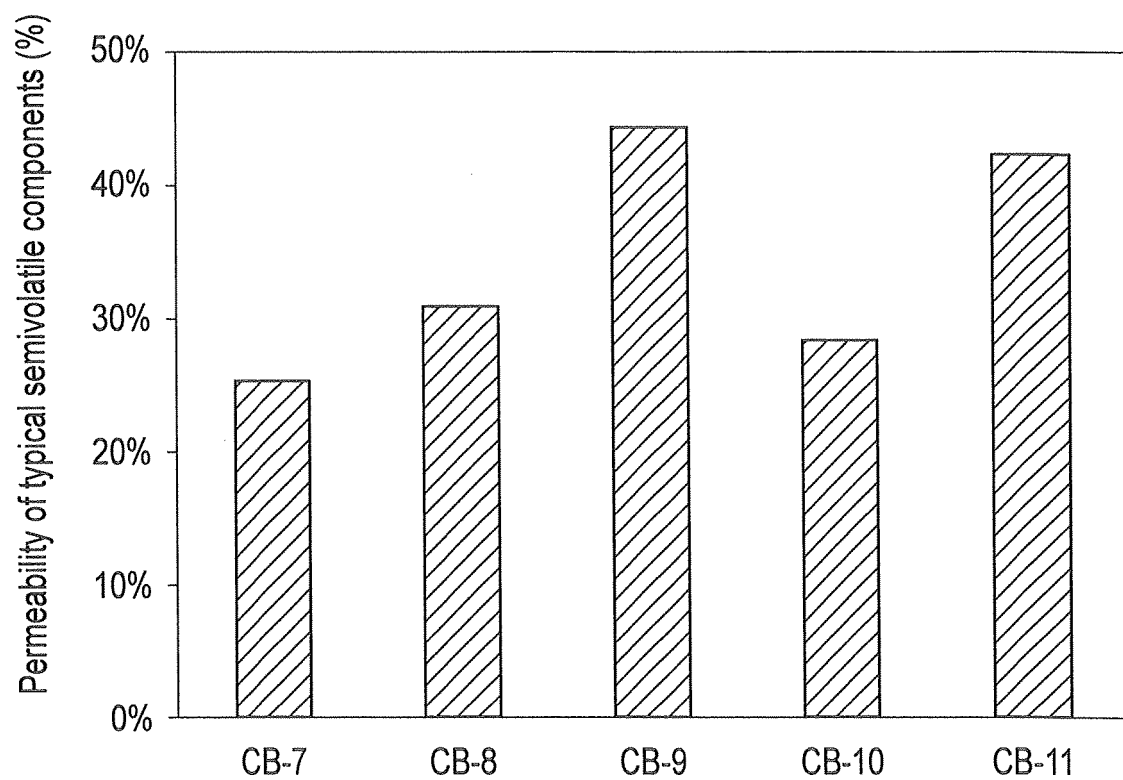


FIG. 8C

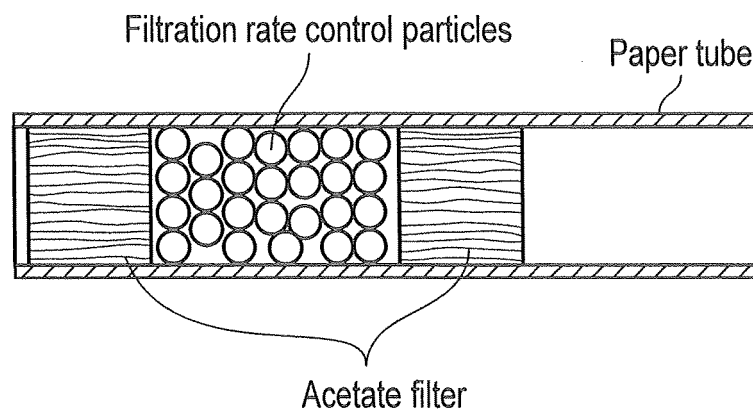


FIG. 9A

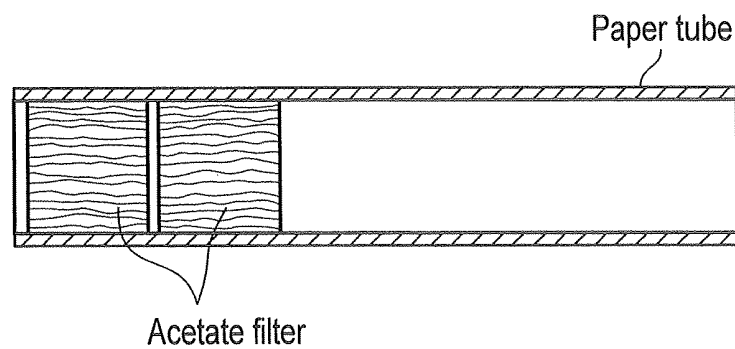


FIG. 9B

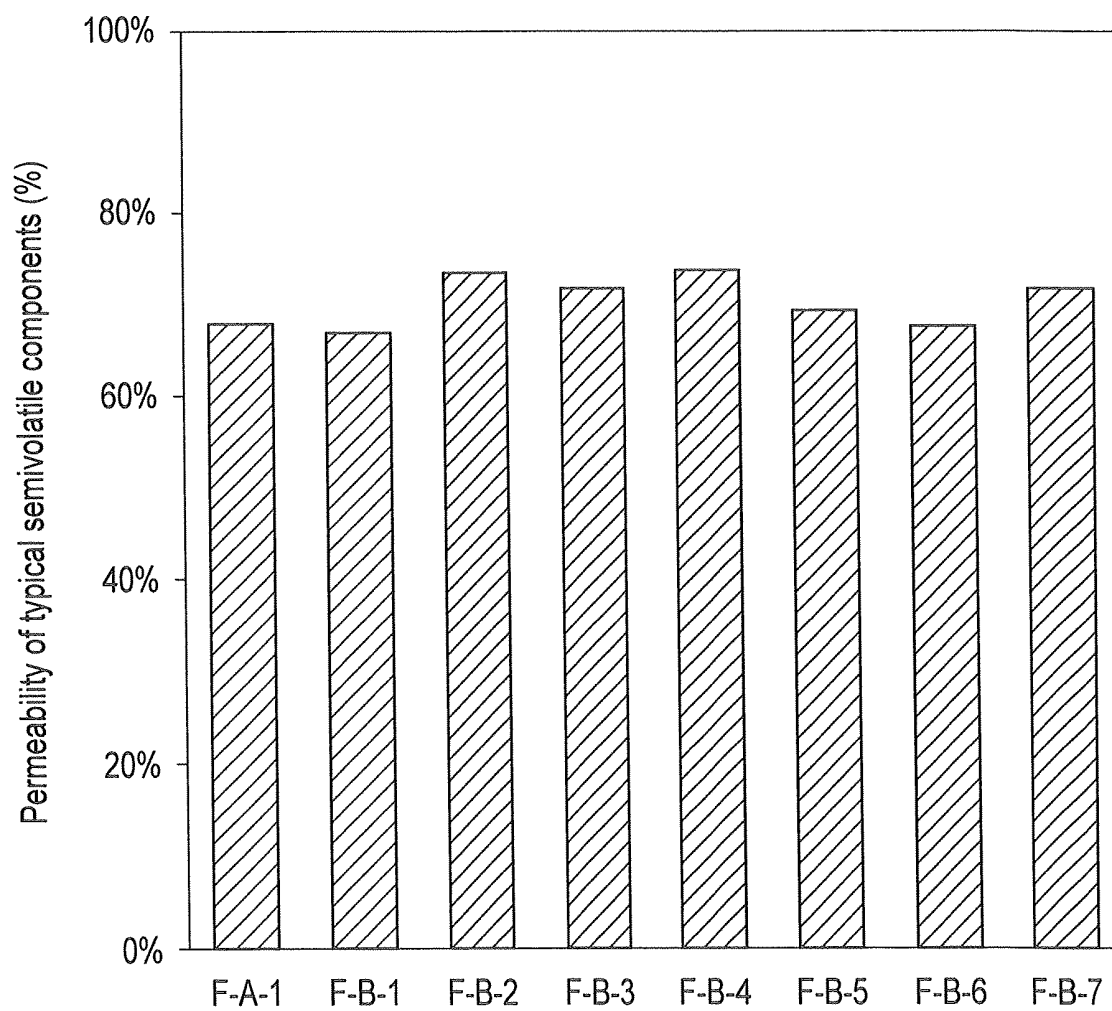


FIG. 10

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2012/079108

A. CLASSIFICATION OF SUBJECT MATTER

A24D3/10 (2006.01) i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A24D3/10

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2013

Kokai Jitsuyo Shinan Koho 1971-2013 Toroku Jitsuyo Shinan Koho 1994-2013

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	JP 63-74477 A (Advance Co., Ltd.), 04 April 1988 (04.04.1988), page 1, lower right column, line 12 to page 2, upper right column, line 12 & EP 246330 A1 & WO 1987/002869 A1	1, 3-5, 7, 11 6, 8-10
X Y	JP 2005-522208 A (ROZIM, Peter), 28 July 2005 (28.07.2005), page 3, lines 30 to 39; page 5, lines 2 to 13; drawings & US 2005/0161055 A1 & EP 1494553 A1 & WO 2003/086117 A1 & HU 201257 A & CA 2482157 A1 & CN 1625350 A & AU 2002307875 A	1, 3-5, 7, 11 6, 8-10

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

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Date of the actual completion of the international search
05 February, 2013 (05.02.13)Date of mailing of the international search report
12 February, 2013 (12.02.13)Name and mailing address of the ISA/
Japanese Patent Office

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2012/079108

C (Continuation).	DOCUMENTS CONSIDERED TO BE RELEVANT	
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	JP 2011-177698 A (Daicel Chemical Industries, Ltd.), 15 September 2011 (15.09.2011), page 2, lines 41 to 43; page 6, line 26 to page 7, line 39; page 10, lines 15 to 21 (Family: none)	1-5, 7, 11 6, 8-10
Y	WO 2003/56947 A1 (Japan Tobacco Inc.), 17 July 2003 (17.07.2003), page 11, line 1 to page 12, line 1; fig. 1 & US 2004/0237983 A1 & EP 1470761 A1 & CA 2472385 A1 & AU 2003202479 A & UA 77052 C & CN 1612701 A & RU 2004124060 A & CN 102028307 A & TWB 001324049	6
Y	JP 8-47385 A (Daicel Chemical Industries, Ltd.), 20 February 1996 (20.02.1996), column 3, lines 38 to 49; column 4, line 15 to column 6, line 44; column 8, lines 24 to 28 & US 5711322 A & US 5967149 A & EP 641525 A3 & DE 69414938 D & ES 2129094 T & AT 173889 T & DK 641525 T & HK 1011915 A & KR 10-0188815 B & CN 1108514 A & CA 2131236 A1	8
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Form PCT/ISA/210 (continuation of second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2012/079108

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 59-55180 A (Asahi Chemical Industry Co., Ltd.), 30 March 1984 (30.03.1984), entire text (Family: none)	1-11

Form PCT/ISA/210 (continuation of second sheet) (July 2009)

REFERENCES CITED IN THE DESCRIPTION

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- **KAZUO MAEDA.** Research on Development of Tobacco Smoke Filter. Central Research Institute, Japan Tobacco Inc, December 1983, 27-30 [0066]