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54 **ADSORBENT FOR MUTAGENIC SUBSTANCE CONTAINED IN TOBACCO SMOKE AND USE THEREOF.**

57 An adsorbent for a mutagenic substance contained in tobacco smoke comprises a polysaccharide (e.g. cellulose) or its modified product (e.g., ion-exchanged polysaccharide).

EP 0 246 330 A1

DESCRIPTION

TITLE OF THE INVENTION

Adsorbent for Mutagenic Compounds in Tobacco Smoke,
and Use thereof

TECHNICAL FIELD

5 The present invention relates to an adsorbent for
mutagenic compounds present in tobacco smoke upon
inhalation, a process for using said adsorbent, and a
filter-tip containing said adsorbent.

BACKGROUND ART

10 Various researches have proved that smoking
increases the rate of outbreaks of diseases such as lung
cancer. Further, it has been proved that tobacco smoke
contains many carcinogens, for example, polycyclic
aromatic hydrocarbons such as benz(a)pyrene or
15 benz(a)anthracene, or nitrosoamines.

Therefore, various kinds of cigarettes having low
tar and/or low nicotine contents have been marketed.
However, a study of the mutagenicity of a given amount
of cigarette smoke condensate (CSC) by the Ames-test has
20 revealed a tendency for cigarettes having low tar and/or
low nicotine contents to generally show higher values of
mutagenicity. It is believed, therefore, that there is
no correlation between the amount of CSC and the
rate of outbreaks of cancer. It is known that cigarettes
25 having a lower tar content are not always less dangerous,
and therefore, it is believed that the total mutagenicity
per cigarette should be adopted as a criterion for an
estimation of the rate of outbreaks of cancer due to
cigarettes.

30 Accordingly, an object of the present invention is
to provide an adsorbent for mutagenic compounds, which
is capable of remarkably reducing a total mutagenicity
in tobacco smoke without impairing the inherent flavor
thereof.

35 Other objects of the present invention are to

provide a process for removing mutagenic compounds present in tobacco smoke using the above adsorbent, and a filter-tip for tobacco containing the above adsorbent.

DISCLOSURE OF THE INVENTION

Accordingly, the present invention relates to an adsorbent for mutagenic compounds present in tobacco smoke, comprising a polysaccharide or a modified substance thereof.

Further, the present invention relates to a process for removing mutagenic compounds from tobacco smoke, by adsorbing the mutagenic compounds present in tobacco smoke onto a polysaccharide or a modified substance thereof.

Furthermore, the present invention relates to a filter-tip for tobacco, particularly a filter-tip for a cigarette, containing a polysaccharide or a modified substance thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a graph illustrating a relationship between a tar content and a specific mutagenicity in cigarettes.

Figure 2 illustrates a structure of the filter-tip according to the present invention, and a manner of inserting the same into a cigarette.

Figure 3 is a graph illustrating a relationship between amounts of the adsorbents used of the present invention and total mutagenicity.

Figure 4 is a graph illustrating a relationship between amounts of the adsorbents having various particle sizes and a rate of removing mutagenic compounds.

BEST MODE FOR CARRYING OUT THE INVENTION

The term "polysaccharide" used herein denotes a high-molecular weight compound formed by the condensation of about 7 or more monosaccharide molecules, and includes a simple polysaccharide obtained from identical monosaccharide molecules and a complex polysaccharide obtained from various monosaccharides or derivatives

thereof. The polysaccharides used in the present invention are typically cellulose, agarose, amylose, chitin, chitosan, and derivatives thereof. The polysaccharide is preferably cellulose.

5 In the present invention, the polysaccharide may be used as the adsorbent in any form, such as powders, granules or fibers, although powders are preferable. The particle size of the powder is not critical, but preferably powder having an average particle size of
10 generally 0.1 - 200 μm , more preferably 0.1 - 40 μm , further preferably 1 - 10 μm , is employed.

As polysaccharide powders, those prepared by a mechanical treatment (e.g., grinding or chopping) or a chemical treatment (e.g., acid or alkali treatment) of
15 animal or plant materials are employed. Such powders are, typically, cellulose powder such as cotton powder, pulp powder, flax powder or regenerated cellulosic fiber powder. Further, these powders include crystalline
20 cellulose powder prepared by an acid hydrolysis or cellulase treatment of pulp. As the crystalline cellulose powder, it is preferable to use fine powder having an average particle size of 0.1 - 200 μm , more preferably 1 - 10 μm . When a fine powder having an average particle size of 1 - 10 μm is used, it is
25 possible to maximize an adsorbing action for mutagenic compounds without changing a resistance to the suction of tobacco smoke. Therefore, it is preferable to fractionate crystalline cellulose powders having an average particle size of several microns by a continuous
30 centrifugation. Further, agarose powder (e.g., manufactured by Wako Junyaku Kogyo K.K.), amylose powder (e.g., manufactured by Nakai Kagaku Yakuhin K.K.), chitin powder (e.g., manufactured by Nakai Kagaku Yakuhin K.K.), chitosan powder (e.g., prepared by an
35 alkali treatment of the above chitin powder) and the like may be employed.

The term "modified substance of polysaccharide"

used herein denotes a material prepared by chemically modifying the above polysaccharide, particularly polysaccharide powder. These modified substances include, for example, ion-exchange polysaccharide or hemin-dyed polysaccharide.

The ion-exchange polysaccharide is a polysaccharide, particularly the powder thereof, formed by incorporating therein various dissociating substituents to exhibit the properties of an ion-exchange resin. As typical examples of these ion-exchange polysaccharides, there may be cited, a cellulosic anion exchanger such as aminoethyl(AE)-cellulose, diethylaminoethyl(DEAE)-cellulose, or triethylaminoethyl(TEAE)-cellulose, a cellulosic cation exchanger such as carboxymethyl(CM)-cellulose, phosphate(P)-cellulose, or sulfoethyl(SE)-cellulose, or an agarose ion exchanger.

The hemin-dyed polysaccharide is a polysaccharide, particularly the powder thereof, formed by subjecting the powder to a heat treatment in 2-chloroethyl amine and aqueous solution of NaOH, and thereafter, adding hemin to effect dyeing. These polysaccharides include, for example, hemin-dyed cellulose powder.

The mutagenic compounds present in tobacco smoke can be adsorbed onto the adsorbent according to the present invention, merely by bringing tobacco smoke into contact with the adsorbent. Therefore, the adsorbent of the present invention may be employed in various ways.

For example, the adsorbent of the present invention may be employed in combination with a filter-tip for a cigarette. In this case, the adsorbent of the present invention can be incorporated in an expandable polymer such as polyvinyl alcohol (PVA) or polyvinyl formal (PVF), followed by expansion molding to disperse the adsorbent in the filter-tip. An average particle size of the adsorbent to be incorporated during filter-tip formation is preferably 0.1 - 200 μm , more preferably 1 - 10 μm . Alternatively, the adsorbent can be employed

in embodiments wherein the adsorbents are dispersed between cellulose acetate fibers for a filter-tip or units aggregating the adsorbents are inserted into spaces in cellulose acetates. In the latter case, an
5 average particle size of the adsorbent powder is preferably 50 - 200 μm . Further, the adsorbent may be dispersed in tobacco leaves. Even further, the adsorbent may be used as a component of a filter-tip for removing mutagenic compounds, which filter-tip is incorporated in
10 a holder employed for smoking a cigarette.

An amount of the adsorbent used of the present invention may vary with the conditions of application. When a single kind of the adsorbent is used, the amount is 1 - 500 mg/cigarette. When two or more adsorbents
15 are used in combination, the amount of each adsorbent is 100 μg - 100 mg/cigarette and the total amount is 1 mg-1 g/cigarette.

Example

The following examples illustrate the present
20 invention more concretely.

Method for Determination of Mutagenicity

In the following examples, the mutagenicity was determined according to the following procedure:

A particle phase of cigarette smoke was
25 trapped by a Whatman glass filter GF/C ($\phi 37$ mm) using an automatic suction device. The filter was treated with dimethyl sulfoxide. The resultant eluted substance was designated as a cigarette smoke condensate (CSC) and used in experiments in the form of a DMSO solution.
30 The suction was carried out at a rate of 650 ml/min for 2 seconds at intervals of 20 seconds. The suction was repeated twelve times for one cigarette. A mutagenicity of the resultant CSC (300 μg /plate) was examined by the
Yahagi preincubation method [Takiyahagi, Tanpakushitsu,
35 Kakusan, Koso (Protein, Nucleic Acid and Enzyme), 20, 1178 (1975)] which is a modification of a method of Ames et al [B.N. Ames et al, Mutant Res. 31, 347 (1975)].

As a strain, cells of Salmonella typhimurium TA98 at a logarithmic growth middle phase were employed. When a liver microsome fraction (S9) is added to CSC, a stronger mutagenicity is observed. Thus, 500 μ l of an S9 mixture solution of containing 100 μ l of S9 (30 mg protein/ml), 100 μ l of TA98 strain (1×10^8), and 100 μ l of DMSO solution of CSC (0.3 mg) was used for a plate. The S9 was prepared from an enzyme-induced liver of an SD rat (having a weight of about 100 g) and treated with polychlorinated biphenyl. The amount of CSC was obtained as a weight (mg) per one cigarette by calculation from an absorbance at 260 nm measured by a spectrophotometer as well as by actual determination with an electronic load cell scale (Mettler AE163 [®]).

Accordingly, a total mutagenicity of a cigarette was calculated as follows:

$$T = \frac{S \times CSC}{0.3}$$

wherein T represents a total mutagenicity of a cigarette, S represents the number of reverse colonies (excepting natural revertant colonies) per one plate, and CSC represents the weight (mg) of CSC per one cigarette.

The mutagenicity of CSC increases linearly up to 400 - 500 μ g/plate independently of the kind of cigarette and of the existence or absence of a filter-tip. Therefore, a total mutagenicity was shown on the basis of the numbers of reverse colonies at 300 μ g/plate. A specific mutagenicity was shown on the same basis.

Reference Example: Comparison of Various Cigarette Brands in Specific Mutagenicity

This reference example illustrates that a tar content and mutagenicity of a certain cigarette are not correlated.

According to the above-mentioned method for the determination of mutagenicity, specific mutagenicities of 9 brands of cigarettes commercially available from the Japan Tobacco Inc., and 11 brands of imported

cigarettes were calculated and compared. There were differences of twice or three times the mutagenicity within such brands. Then, the tar contents and mutagenicities of the CSCs' were compared, because the tar content of a cigarette varies with different brands. As shown in Figure 1, the tar content of a cigarette negatively correlates to the mutagenicity thereof. Therefore, it is manifest that a cigarette with a lower tar content is not always less likely to increase outbreaks of cancer.

Figure 1 illustrates a comparison between tar contents and specific mutagenicities in cigarettes. The specific mutagenicities were calculated from the numbers of revertant colonies at 300 µg/plate of the CSC according to the above-mentioned method, and marked with a spot in Figure 1 as the number of revertant colonies per 1 mg of CSC (an average of 20 cigarettes, in each brand). The data of the tar content (mg) of a cigarette was taken from that announced by The Japan Tobacco Inc., in a pamphlet. Figure 1 shows the results obtained from "Mild Seven" (1), "Seven Stars" (2), "Cabin '85 Mild" (3), "Hilite" (4), "Mild Seven Light" (5), "Hope" (6), "Echo" (7), "Peace" (8), and "Mild Seven Select" (9), the above cigarettes (1) to (9) being produced by The Japan Tobacco Inc., and "Lark Mild" by Liggett & Myers (10), "Lark" by Liggett & Myers (11), "Parliament 100's" by Philip Morris (12), "Lark Mild 100's" by Liggett & Myers (13), "Lark 100's" by Liggett & Myers (14), "Salem Light" by R.J. Reynolds (15), "Lark Superlight" by Liggett & Myers (16), "Lucky Strike" by Brown & Williamson (17), "Virginia Slims Lights Menthol" by Philip Morris (18), "More" by R.J. Reynolds (19), and "Carlton" by MFD, Ducham N.C. (20). In Figure 1, white circles o denote cigarettes made in the USA, and black circles ● denote those made in Japan.

Example 1: Comparison of Rates of Removing Mutagenic Compounds by Different Adsorbents

Various adsorbents for mutagenic compounds were inserted in the filter-tip body (1) shown in Figure 2 and then tests were carried out as follows. Note, the filter-tip body (1) comprises portions (2) of filter-tip of cellulose acetate and a space (3) therebetween for insertion of the adsorbent, and is attached to a cigarette body (5) containing tobacco leaves (4).

In order to compare the rates of removing mutagenic compounds by means of cellulose or cellulosic ion exchangers, in the space (3) [6 mm in length] of a filter-tip (1) for the cigarette, 25 mg of Avicel (Asahi Chemical Industry) as the cellulose powder, 25 mg of DEAE-cellulose (DE52: Whatman) as the cellulosic anion exchanger, and 25 mg of CM-cellulose (CM52: Whatman) as the cellulosic cation exchanger, were inserted, respectively. As the cigarette, "Lark" by Ligett & Myers (USA) was employed. The results are summarized in Table 1.

Table 1

Adsorbent	Specific mutagenicity (number of colonies/ 300 μ g of CSC)	Total mutagenicity (number of colonies/cigarette)
Control (Activated carbon 0 mg)	903 \pm 54	38300 \pm 2900
Control (Activated carbon 80 mg)	888 \pm 43	35200 \pm 1380
Avicel 25 mg	776 \pm 70	24600 \pm 2760
DEAE - cellulose 25 mg	774 \pm 69	26000 \pm 3620
CM - cellulose 25 mg	738 \pm 102	19800 \pm 4410

The values of Table 1 are an average \pm standard error of 10 runs.

Example 2: Comparison of Rates of Removing
Mutagenic Compounds in Terms of Different Amounts
Used

In a space having a length of 6 mm and located at
5 the center of the Lark filter-tip, the cellulose powder,
the DEAE-cellulose powder, and the CM-cellulose powder
used in Example 1 were inserted in an amount of 5, 10,
20, 25 or 30 mg. The total mutagenicity of the
cigarettes were compared. The results are summarized in
10 Figure 3. In the graph of Figure 3, a total mutagenicity
obtained when the activated carbon is eliminated from
the space of the Lark filter-tip is regarded as 100%.
An index of a total mutagenicity (%) in an ordinate of
Figure 3 denotes-ratios of total mutagenicities to that
15 of 100%. In Figure 3, the results obtained using as an
adsorbent for mutagenic compounds the cellulose powder,
DEAE-cellulose and CM-cellulose are represented by X(o),
Y(Δ) and Z(\square), respectively.

Example 3: Panel Tests

20 Panel tests were carried out using 5 kinds of
filter-tips employed in Example 1. Evaluation was made
in comparison with Filter-tip No. 5, and determined by
seven grades (much better ... +3; better ... +2; slightly
better ... +1; equal ... 0; slightly worse ... -1;
25 worse ... -2; much worse ... +3). The results from 17
panel members (12 males and 5 females) are summarized in
Table 2. It is apparent therefrom that the filter-tip
according to the present invention has little affect on
the flavor. The adsorbents inserted in the filter-tips
30 listed in Table 2 are as follows:

Filter-tip No. 1 ... 80 mg of activated carbon
(filter-tip conventionally
used by Lark)

Filter-tip No. 2 ... 25 mg Avicel

Filter-tip No. 3 ... 25 mg of CM-cellulose

Filter-tip No. 4 ... 25 mg of DEAE-cellulose

Filter-tip No. 5 ... no activated carbon

(only cellulose acetate
filter-tip)

Table 2

Filter-tip No.	Smell	Taste	Peculiarity	Stimulus	Mildness
1	+2.6	+2.3	+2.4	+1.5	+1.9
2	+1.3	+0.9	+1.9	+2.0	+2.3
3	+0.8	+0.6	+1.0	+1.0	+1.5
4	+0.8	+0.4	+0.5	+1.4	+0.8
5	0	0	0	0	0

Example 4: Comparison of Efficiencies of Removing
Total Mutagenic Compounds by Different Sizes of
Crystalline Cellulose Powders

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A material prepared by incorporating a crystalline cellulose powder (this powder was obtained by treating with diluted sulfuric acid a pulp available from Sanyo-Kokusaku Pulp Co., and had an average particle size of 0.4 μ m, 4 μ m, 40 μ m or 100 μ m) into an expandable polyvinyl alcohol (PVA) during an expanding stage was molded into a cylinder 7 mm in diameter and 10 mm in length. The cylinder was inserted in a holding portion in a commercially available Heminphthalo-filter-tip (manufactured by Advance Co., Ltd.). The total mutagenicity of a cigarette (Mild Seven: Japan Tobacco Inc.) was calculated according to the above-mentioned method. A ratio of removing mutagenic compounds is shown in Figure 4 for each filter-tip.

In Figure 4, A(\bullet), B(o), C(Δ) and D(x) represent crystalline cellulose powders having particle sizes of 0.4 μ m, 4 μ m, 40 μ m, and 100 μ m, respectively, and a

relationship between an amount in weight (g) of crystalline cellulose powder incorporated in 1 liter of expandable polymer (PVA) and a rate of removing mutagenic compounds is shown.

5 Example 5: Reduction of Specific Mutagenicity of
 CSC due to Filter-tip Containing Crystalline
 Cellulose Powder

 An expandable polymer PVA containing 35 g/l of
 crystalline cellulose powder (Avicel SF: Asahi Chemical
10 Industry) having an average particle size of 4 μ m, and a
 cylinder 7 mm in diameter and 10 mm in length was molded
 as in Example 4. The cylinder or PVA filter-tip without
 crystalline cellulose powder was inserted in the holding
 portion of the hemiphthalo filter by Advance Co., Ltd.
15 Smoking tests were performed according to the above-
 mentioned method.

 Specific mutagenicities of CSC were determined for
 ten cigarettes of each of Mild Seven, Long Peace and
 Cabin '85 Mild, made by the Japan Tobacco Inc., and
20 Lark FSK made by Liggett & Myers, according to the
 above-mentioned method. As shown in Table 3, a
 remarkable reduction of specific mutagenicity was
 observed only in the case of the PVA filter-tip
 containing crystalline cellulose powder in each
25 cigarette, and it was proved that such a reduction is
 brought about by a selective adsorption of the adsorbent
 for mutagenic compounds. The values are represented in
 the form of an average \pm standard error. The numerals
 in parentheses in Table 3 are indices, where the index
30 for a cigarette without a filter-tip is 100.

Table 3

	Specific mutagenicity (number of reverse colonies/ CSC 300 µg)			
	Mild Seven	Long Peace	Cabin '85 Mild	Lark FSK
No filter-tip	461 + 33 (100)	330 + 22 (100)	624 + 82 (100)	863 + 28 (100)
PVA filter-tip	508 + 41 (100)	348 + 30 (105)	609 + 40 (98)	847 + 42 (98)
PVA filter-tip containing crystalline cellulose powder	369 + 28 (80)	271 + 28 (82)	485 + 16 (78)	690 + 27 (80)

20 Example 6: Total Efficiency of Removing Total
Mutagenic Compounds and Reduction of Specific
Mutagenicity of CSC by Use of Hemin-Dyed
Polysaccharide

Triethylaminoethyl cellulose (TEAE-cellulose) -
25 prepared by reacting crystalline cellulose powder
(Avicel SF: Asahi Chemical Industry) having an average
particle size of 6 µm with 2-chloroethylamine was dyed
by hemin, and thereafter, unadsorbed hemin was thoroughly
removed to obtain hemin-dyed cellulose powder. 2% by
30 weight of hemin-dyed powder was blended with non-dyed
crystalline cellulose powder to obtain an adsorbent.
The efficiency of removing total mutagenic compounds
and the specific mutagenicity were determined as in
Examples 4 and 5.

35 Provided that an efficiency of removing total
mutagenic compounds in the case of no filter-tip is 0%,
that in the case of the adsorbent consisting only of

non-dyed cellulose powder was 45%, whereas that in the case of the adsorbent containing 2% by weight of hemin-dyed cellulose powder was 55%.

5 Provided that when using a cellulose acetate filter-tip, the reduction rate of the specific mutagenicity is 0%, the rate was 15% with the adsorbent consisting of non-dyed cellulose, but was 30% with the adsorbent containing 2% by weight of hemin-dyed cellulose powder.

10 As shown above, it has been proved that a reduction rate of efficiency of removing total mutagenic compounds and specific mutagenicity can be enhanced by virtue of the hemin-dyeing of the crystalline cellulose powder.

Example 7: Panel Tests

15 Panel tests were carried out using 4 kinds of filter-tips employed in Example 4. Evaluation was made into five grades (very good ... +2; good ... +1; normal ... 0; bad ... -1; very bad ... -2). The results from 34 panel numbers (24 males and 10 females) are
20 summarized in Table 4. It is apparent therefrom that the filter-tip according to the present invention has little effect on the flavor.

The filter-tips were inserted in the holding part of Hemiphthalo filter produced by Advance Co., Ltd.,
25 and then the panel tests were performed. As the cigarette, Mild Seven (Japan Tobacco Inc.) was used.

The adsorbents inserted in Filter-tips Numbers. 1 to 6 are as follows:

- Filter-tip No. 1 ... Control: no filter-tip
- Filter-tip No. 2 ... Control: PVA filter-tip
- Filter-tip No. 3: PVA filter-tip containing crystalline cellulose powder having an average particle size of 0.4 μ m (35 g/l)
- Filter-tip No. 4: PVA filter-tip containing crystalline cellulose

powder having an average
particle size of 4 μm
(35 g/l)

Filter-tip No. 5: PVA filter-tip containing
crystalline cellulose
powder having an average
particle size of 40 μm
(35 g/l)

Filter-tip No. 6: PVA filter-tip containing
crystalline cellulose
powder having an average
particle size of 100 μm
(35 g/l)

Table 4

Filter-tip No.	Smell	Taste	Peculiarity	Stimulus	Mildness
1	+1.5	+1.7	+1.0	+0.8	0
2	+0.5	+1.2	+0.7	+0.5	+0.5
3	-0.2	+1.0	+0.5	-0.6	+1.1
4	+1.2	+1.5	+0.8	0	+1.6
5	+1.0	+0.9	+0.4	-1.2	+0.8
6	+0.2	+1.2	0	-0.3	+0.3

Industrial Applicability

The adsorbent according to the present invention does not diminish the inherent flavor of tobacco, is able to efficiently and selectively adsorb mutagenic compounds present in tobacco smoke and remove the same from tobacco smoke, and greatly contributes to a decrease in the outbreak of diseases such as lung cancer by smoking.

CLAIMS

1. An adsorbent for mutagenic compounds present in tobacco smoke, comprising a polysaccharide or a modified substance thereof.
2. An adsorbent according to claim 1, wherein the
5 polysaccharide or modified substance thereof is powder.
3. An adsorbent according to claim 2, wherein an average particle size of the powder is 0.1 - 200 μm .
4. An adsorbent according to claim 1, wherein the polysaccharide is cellulose.
- 10 5. An adsorbent according to claim 4, wherein the cellulose is crystalline cellulose.
6. An adsorbent according to claim 1, wherein the modified substance is an ion-exchange polysaccharide or hemin-dyed polysaccharide.
- 15 7. A process for removing mutagenic compounds from tobacco smoke, comprising by adsorbing the mutagenic compounds present in the tobacco smoke to a polysaccharide or a modified substance thereof.
8. A filter-tip for tobacco, containing a
20 polysaccharide or a modified substance thereof.
9. A filter-tip according to claim 8, wherein the tobacco is in the form of a cigarette.

Fig. 1

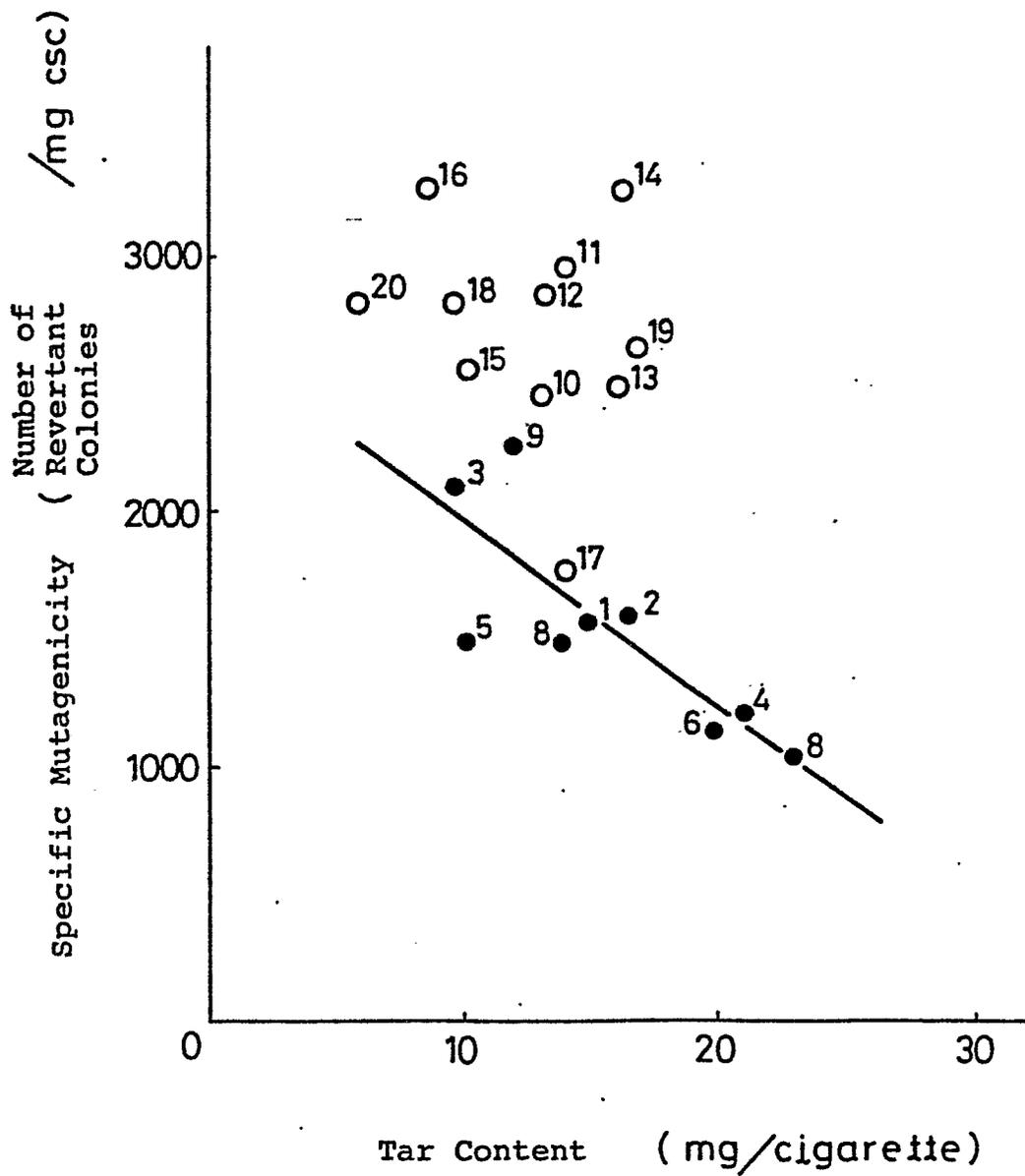


Fig. 2

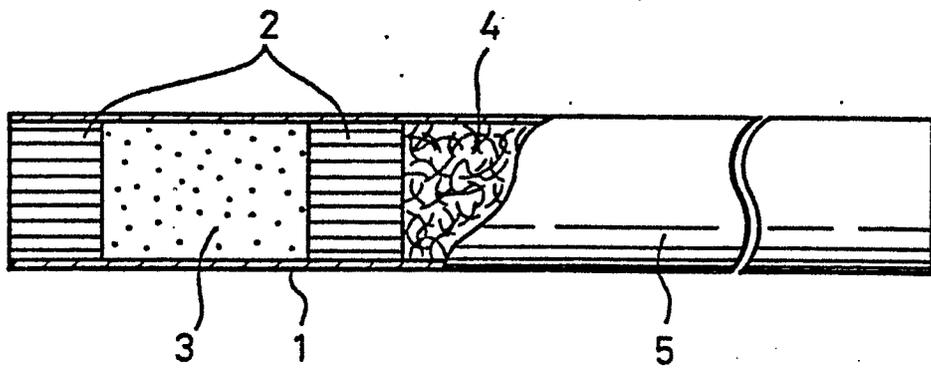
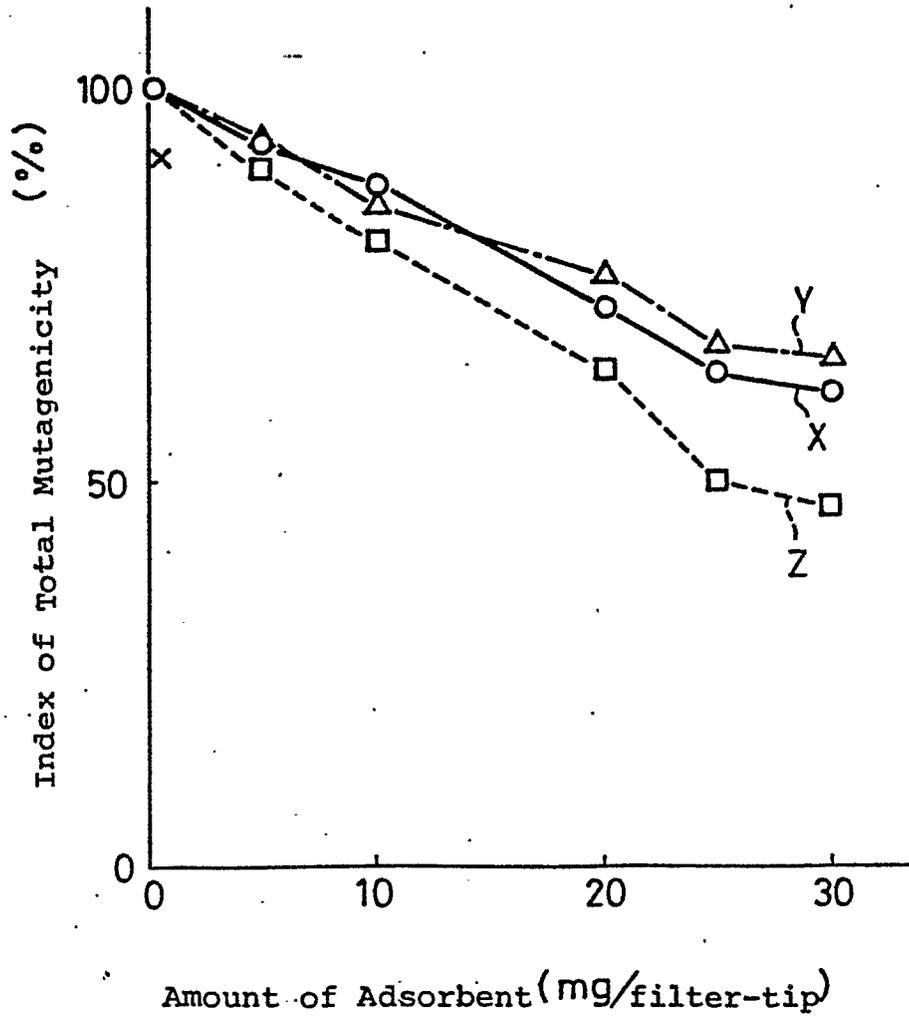
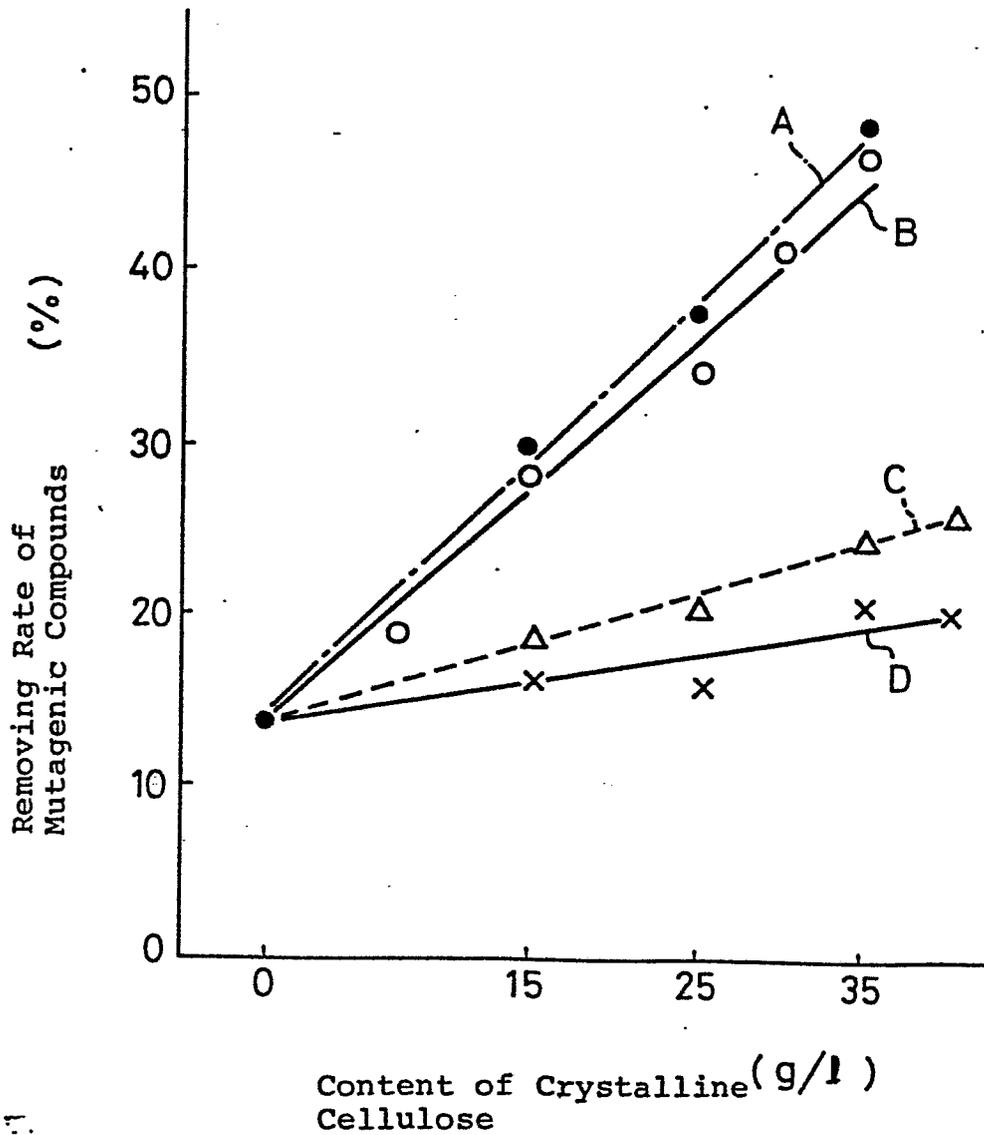


Fig. 3



4/5

Fig. 4



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0246330

List of Reference Numerals and Corresponding Names

- 1 ... filter-tip body
- 2 ... portion of filter-tip of cellulose acetate
- 3 ... space for insertion of adsorbent
- 4 ... tobacco leaves
- 5 ... cigarette body

INTERNATIONAL SEARCH REPORT

International Application No

PCT/JP86/00571

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl ⁴ A24D3/08, 3/12		
II. FIELDS SEARCHED		
Minimum Documentation Searched *		
Classification System	Classification Symbols	
IPC	A24D3/06-3/14, A24F13/06	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched *		
Jitsuyo Shinan Koho	1926 - 1986	
Kokai Jitsuyo Shinan Koho	1971 - 1986	
III. DOCUMENTS CONSIDERED TO BE RELEVANT **		
Category*	Citation of Document ** with indication, where appropriate, of the relevant passages	Relevant to Claim No. *
X	R.E. Kirk, D.F. Othmer Hen "Encyclopedia of Chemical Technology" Vol. 14 (1955) The Interscience Encyclopedia, Inc. (New York), p. 260	1, 4, 7-9
X	JP, A, 54-110,399 (Daicel Kabushiki Kaisha) 29 August 1979 (29. 08. 79) Page 2, lower left column, lines 2 to 10 (Family: none)	1-3, 7-9
X	JP, A, 51-32,799 (Toyo Ink Manufacturing Co., Ltd.) 19 March 1976 (19. 03. 76) (Family: none)	1, 7-9
X	JP, A, 60-110,276 (Toray Industries, Inc.) 15 June 1985 (15. 06. 85) Page 3, lower right column, last line to page 4, upper right column, line 10 (Family: none)	1, 6, 7-9
A	Toxicology, Vol. 15, No.3, (1980),	1
* Special categories of cited documents **		
"A"	document defining the general state of the art which is not considered to be of particular relevance	"T"
"E"	earlier document but published on or after the international filing date	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"X"
"O"	document referring to an oral disclosure, use, exhibition or other means	document of particular relevance the claimed invention cannot be considered novel or cannot be considered to involve an inventive step
"P"	document published prior to the international filing date but later than the priority date claimed	"Y"
		document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents such combination being obvious to a person skilled in the art
		"&"
		document member of the same patent family
IV. CERTIFICATION		
Date of the Actual Completion of the International Search :		Date of Mailing of this International Search Report :
January 20, 1987 (20. 01. 87)		February 2, 1987 (02. 02. 87)
International Searching Authority :		Signature of Authorized Officer :
Japanese Patent Office		