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(54) **FILTER FOR TOBACCO SMOKE**
FILTER FÜR TABAKRAUCH
FILTRE POUR FUMEE DE TABAC

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- **STN INTERNATIONAL, Derwent Information LTD, WPIDS Accession No. 91-041054, TDK CORP, "Tobacco Filter Partic. Removing Harmful Aldehyde(s)-Contg. Zeolite as Adsorbent"; & JP,A,02 308 784, 21-12-90, (9106).**

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Description

[0001] The present invention relates to a tobacco smoke filter and primarily to filters of this kind that include zeolite as a filter material. The invention relates more particularly to a tobacco smoke filter that includes certain specific types of zeolite as a filter material.

[0002] In the composition of tobacco products, including smoking products as well as products intended for other forms of consumption, it has long been endeavored to reduce to the greatest possible extent the amounts of undesirable substances that can be transmitted to the consumer. One problem in this regard is that of combining this endeavour with the endeavour to produce products that will be found acceptable by the consumers, since many of the components that are natural to the tobacco or that are formed as the tobacco burns contribute to the flavour and aroma of the product.

[0003] It has long been endeavored to reduce the concentrations of undesirable substances in the main smokestream generated by smoking articles, and then particularly when smoking cigarettes, with the aid of filters at the mouth end of the smoking article. Many different filter materials and different filter constructions are known to the art from the literature concerned with such products.

[0004] Fibrous cellulose-based material, such as crêpe paper, different forms of regenerated cellulose and, above all, cellulose acetate fibres are examples of those filter materials most used at present. The prime purpose of such fibrous filter materials is to remove part of the aerosol particles, "tar", from the smoke. Cigarette filters may also include different additives whose purpose is to capture gaseous smoke components. The most common of these additives is activated carbon, in different forms. Also known to the art are cigarette filters which include a combination of activated carbon and cellulose filter material, one such known filter being marketed under the registered trademark CURZEL®.

[0005] Another material that has been proposed for use in cigarette filters is comprised of zeolite. For instance, the U.S. Patent Specification US-A-2,839,065 published on June 17, 1958, describes a tobacco smoke filter which includes a "molecular sieve", and three types of zeolite are mentioned by way of example, to-wit: Sodium Zeolite X, Sodium Zeolite A and Calcium Zeolite A. However, no clear explanation is given with regard to the dependency of the filter effect on the properties of the zeolites. In actual fact, this publication gives no clear values with regard to the filter effect. The publication therefore gives no assistance to one skilled in this art in optimizing filter properties by selection of a zeolite that has specific properties.

[0006] Furthermore, US-A-2,839,065 solely describes the removal of aerosol particles from tobacco smoke and gives no indication that gaseous components can also be removed.

[0007] GB-A-2,122,473 also describes the use of zeolite in cigarette filters. In this case, however, the zeolite is merely intended to provide a carrier for smoke-modifying agents, and then particularly menthol. Nothing is disclosed with regard to the filtering properties of the zeolite itself.

[0008] US-A-4,668,648, granted on May 26, 1987, describes magnesium-silicate bound zeolites which according to this publication can be used in cigarette filters. It is clear from the adsorption properties shown that the zeolites used in this case are of the hydrophilic type, because their ability to adsorb water is much higher than the ability of adsorbing organic compounds.

[0009] JP 63248380 and JP 02308784 also describe the use of zeolite in tobacco smoke filters. No clear description of the properties of the zeolites is given.

[0010] All of the aforementioned publications are based on the concept of using standard zeolites, i.e. hydrophilic zeolites, which have an extremely high water adsorption capacity. This adsorption essentially eliminates the possibility of adsorbing organic molecules effectively.

[0011] In distinction to the aforesaid publications, the present invention proposes the use of so-called hydrophobic zeolites, which have quite different adsorption properties. This is explained in more detail below.

[0012] In accordance with the present invention, it has now been found that it is possible to produce tobacco smoke filters, and then particularly cigarette filters, which have filter properties that, in certain respects, are superior to the filter properties of the best of the known filters that include conventional filter materials. The invention is based on the development of zeolites that have specific properties and on the selection of such zeolites on the basis of their ability to eliminate undesirable compounds from tobacco smoke. These zeolites have been found to provide a surprisingly good filter effect.

[0013] In accordance with the invention, there is provided a tobacco smoke filter which is characterized in that the filter material includes at least one hydrophobic zeolite. The zeolites used are preferably of the faujasite type and are here designated ST 100, ST 101, ST 102, ST 103 and ST 104. Of these zeolites, ST 103 and ST 104 are particularly preferred because of their very specific selectivity and therewith their superior filter properties. More specific characteristic data relating to these zeolites will be given in the following.

[0014] The zeolites used in accordance with the invention will suitably have a pore size of 0.5-1 nm (5-10 Å), particularly 0.6-0.8 nm (6-8 Å). The zeolites will preferably have a particlesize of 0.2-1 mm, particularly about 0.5 mm.

[0015] Most zeolites constitute crystalline, hydrated metal-aluminium silicates with the simplest chemical formula

$NaAlSiO_4.H_2O$. The $AlSiO_4$ part builds up an infinite three-dimensional network, where the aluminium atoms and silicon atoms (the central atoms) are coordinated tetrahedrally by oxygen atoms. Each oxygen atom is bound to two central atoms, wherein all such atoms are coupled in space. This part of the zeolite structure is normally referred to as the skeleton and forms an infinite anionic space network. The chemical bonds in this network are covalent and relatively strong.

[0016] There exist a large number of different zeolites with different chemical compositions and differently arranged spatial network structures. However, a common feature of all these zeolites is that the space network contains channels and cavities of different configurations, in which space is available for other ions and molecules.

[0017] Positive ions are always present in the same numbers as there are aluminium atoms in the space network, these positive ions functioning as electric charge equalizers. These positive ions, in turn, surround themselves with water molecules which fill up the remaining cavities in the space network, to a greater or lesser extent. The cavity molecules are bound relatively weakly to the space network and can be expelled from the zeolite in different ways, without influencing the network. For instance, the water molecules can be removed by heating. It is normally said that the zeolite is activated.

[0018] A zeolite which includes a high percentage of aluminium atoms, and therewith a high concentration of positive ions in the cavities, has a high affinity to the adsorption of water molecules and is referred to as hydrophilic, whereas a zeolite with a low aluminium content, or no aluminium content at all, has a low affinity to water and is referred to as being hydrophobic.

[0019] Both organic molecules and water vapour are present in the gaseous substances in tobacco smoke. When a hydrophilic zeolite is used in a filter, the filter will adsorb essentially only water vapour, and has an extremely limited effect on the organic molecules. On the other hand, when using a hydrophobic zeolite which has a very low water affinity, considerable adsorption of the organic substances can be expected.

[0020] The adsorption ability of the zeolite also differs considerably with regard to the organic substances. The properties that control this adsorption ability are dependent on the properties of the organic molecules and also on the properties of the hydrophobic zeolite chosen. The decisive factors with regard to the zeolite are primarily its polarity and the dimensions of the pore system. It is possible to choose different zeolites having different pore systems and different degrees of hydrophobization in achieving desired adsorption in a molecule mixture that consists of many different types of molecules having different physical properties, such as size, shape, polarity and chemical composition.

[0021] It is also possible to use all types of hydrophobic zeolites and mixtures thereof to control and achieve desired adsorption properties in the endeavour to reduce the concentration of undesirable substances in tobacco smoke. The present invention is based on investigations concerning the following types of zeolites:

Internal Designation	Zeolite type Standard	Ratio SiO_2/Al_2O_3	a_0 (nm)	Pore volume ml/g	Bulk density g/ml
ST 100	FAU	5.6	2.451	0.22	0.65
ST 101	FAU	5.7	2.453	0.25	0.54
ST 102	FAU	6.0	2.436	0.23	0.62
ST 103	FAU	14	2.427	0.24	0.55
ST 104	FAU	440	2.425	0.28	0.49

[0022] The aluminum atoms leave the spatial network structure in the de-aluminating process, although some aluminum atoms can still remain in-the pores and cavities of the zeolite. This means that the a_0 values describe the hydrophobicity more effectively than the mole ratio SiO_2/Al_2O_3. A completely dealuminated zeolite will have the following parameters:

Y.Deal. FAU Al-free 2.423 0.28

[0023] All of the zeolites tested are of synthetic origin, and the series ST 100 to ST 104 are of the faujasite-Y-type. It will be understood, however, that the invention is not restricted solely to the use of faujasite-type zeolites, and that these zeolites are given merely by way of example. Generally speaking, any zeolite that has hydrophobic properties can be used in the inventive filter.

[0024] It is particularly preferred to use zeolites in which the SiO_2/Al_2O_3 ratio is greater than 5.5. It has been found that increasing values of the SiO_2/Al_2O_3 ratio increase the hydrophobicity and reduce the water affinity. The cell constant decreases, as does also the polarity. This means that the usefulness of the zeolites as tobacco smoke filter mate-

rial increases with increasing values of said ratio.

[0025] The pore diameter will suitably lie in the range of from 0.5 to 1 nm (5 to 10 Å), and preferably from 0.6-0.8 nm (6-8 Å).

[0026] The zeolite materials are prepared for use in the inventive filter, by agglomerating said materials to a granular form, with the aid of a suitable binder. There is preferably used about 25 parts by weight binder to 100 parts by weight zeolite material and the grains produced will have a diameter of 0.2-1 mm, preferably about 0.5 mm, as previously mentioned.

[0027] The inventive filter may include one or more of the aforesaid zeolites as a the sole gas phase adsorbent. However, a better filter effect can be achieved when the zeolites are used together with other, known filter materials.

[0028] The zeolites can be combined with other gas-phase active filter materials in different ways.

[0029] For instance, the different materials may be simply mixed together and the mixture applied to a filter space comprised of two sections of particle-filter with an intermediate chamber at the mouth end of a cigarette. Alternatively in another embodiment, the filter may be divided into several compartments which may contain different filter materials or different mixtures of filter materials.

[0030] The filter effect of an inventive cigarette filter containing zeolite has been investigated with zeolites in chamber filters. Comparison tests were also run with prior art filters. The reduction in the concentrations of gas-phase components in the smoke and the reduction of the toxic effects of the gas phase, as measured in the V79 system, were determined in the tests. The tests are described in more detail below.

[0031] In the chemical gas-phase analysis, the concentrations of a series of different gas-phase components (classified according to functional groups) were determined quantitatively by means of high-resolution gas chromatography in a conventional manner. The percentile reduction in the concentration of gas-phase components in the smoke produced from a test filter cigarette in relation to the concentration of these components in smoke produced from a reference cigarette was calculated. The reductions in concentration can be given for each chemical group per se or as a mean value for all groups.

[0032] The cytotoxity was determined in a known way, by measuring the ability of the gas phase to inhibit the colony forming ability of V79 cells from the lungs of Chinese hamsters (Jenssen, D. in G.J. Kilbey, M. Legator, W. Nichols and C.Ramel (Eds): Handbook of Mutagenicity Test Procedures, Elsevier Science Publishers BV, Amsterdam (1984), pp. 269-290). The results are given as an IC-50% value, i.e. the number of "puffs" on a cigarette which inhibits colony formation by 50%.

[0033] When testing the filter properties of a number of zeolites, there was used a type of American blend cigarette having a chamber filter which included 150 mg of the tested zeolite. The same cigarette was used as a reference cigarette, but with the filter chamber empty. The tests were carried out in a standard smoke machine in accordance with CORESTA standards.

[0034] The following results were obtained:

Chemical Gas-Phase Analysis					
Zeolite	ST 100	ST 101	ST 102	ST 103	ST 104
Reduction of gas-phase components, %	28	25	37	81	83

Cytotoxity Test	
Filter	IC-50% (number of puffs)
Empty chamber	3
ST 100, 150 mg	8
ST 101, 150 mg	6
ST 102, 150 mg	7
ST 103, 150 mg	23

(continued)

Cytotoxity Test	
Filter	IC-50% (number of puffs)
ST 104, 150 mg	29
Highly activated carbon, 50 mg	13

[0035] It will be seen from the Tables that all of the zeolite materials have a pronounced filter effect, and that the types ST 103 and ST 104 provide the absolute best results.

[0036] The filter properties of the zeolite types ST 103 and ST 104 were compared with the filter properties of the known filter CURZEL® in a further test, this known filter comprising a combination of cellulose acetate fibres and activated carbon. The cigarettes used in the test contained tobacco of the American blend type and the filters used were double filters containing 8 mm acetate and 12 mm acetate+zeolite (40 mg). All filters were ventilated to 51%. The reference cigarette included a filter containing only cellulose acetate fibres.

[0037] The following Table shows the reduction in different groups of gas-phase components in the smoke with regard to the two zeolite-filter cigarettes in relation to the reduction obtained with the CURZEL® filter.

Chemical group	ST 103	ST 104
Saturated hydrocarbons	0.57	1.32
Non-saturated hydrocarbons	0.22	0.98
Aromatic hydrocarbons	0.68	1.27
Aldehydes	0.47	1.16
Alcohols	0.21	0.79
Nitriles	0.62	1.32
N-heterocyclics	0.47	0.86
Ketones	0.93	1.38
Mean value	0.56	1.20

[0038] It will be seen from the results listed in the Table that from the aspect of elimination, the inventive zeolite-containing filters are superior in some respects to the CURZEL® filter, which has hitherto been considered to be one of the most effective filters on the market.

[0039] Sensory tests also showed that the inventive zeolite-containing filter results in no loss in flavour in comparison with the known CURZEL® filter. On the contrary, a test smoking panel reported in its sensory evaluation an improvement in flavour and a reduction in irritation levels, which is in accord with the reduction in the concentrations of irritants shown by gas chromatography.

[0040] With the exception of the inclusion of zeolites of specific types, the smoking article and the filters have a construction typical in the tobacco industry. The tobacco material used and the casing material for the tobacco strand and the filter do not differ from those used conventionally hitherto.

[0041] The filters may also be ventilated, so as to dilute the smoke with secondary air. Ventilation will often result in a further decrease in the concentrations of undesirable smoke components. The degree of ventilation may be up to 80%, determined in a manner conventional in the tobacco industry. Ventilation can be achieved in any one of several different ways known in the art, for instance by perforating the filter casing.

[0042] The invention thus enables the manufacture of a tobacco smoke filter whose filter properties are superior to those filters that have hitherto been considered the best filters to be had commercially. It has also been possible to control the aroma and biological qualities by using different types of zeolites. These control possibilities become still more extensive when using zeolite together with other filter materials, and then particularly carbon, as the differences in adsorption properties between the different materials can then be used in different filter material combinations. Neither has the improved filter effect resulted in undesirable losses or in an impairment of the aroma and flavour of the smoking article when smoked.

[0043] The inventive filter has been described in the aforegoing primarily with reference to cigarettes. The person skilled in this art, however, will understand that the invention is not limited to this combination. For instance, an inventive filter can be inserted into a holder for cigarettes, cigar-cigarettes or cigars, or into the stem of a tobacco pipe, therewith obtaining selective and favourable filter effects in all instances. Other variants and modifications of the invention are also possible without departing from the inventive concept as defined in the following Claims.

Claims

1. A tobacco smoke filter, **characterized** in that the filter includes as filter material at least one zeolite that has hydrophobic properties, meaning that the zeolite has a low affinity for water.

2. A filter according to Claim 1, **characterized** in that the hydrophobic zeolite or zeolites has/have a mole ratio SiO_2/Al_2O_3 which is greater than 5.5.

3. A filter according to Claim 1 or Claim 2, **characterized** in that the filter includes at least one zeolite of at least one of the types ST 100, ST 101, ST 102, ST 103 and ST 104.

4. A filter according to any one of Claims 1-3, **characterized** in that the zeolite or the zeolites has/have a pore size of 0.5-1 nm (5-10 Å), preferably 0.6-0.8 nm (6-8 Å).

5. A filter according to any one of Claims 1-4, **characterized** in that the zeolite or zeolites is/are used in the form of agglomerated grains having a particle size of 0.2-1 mm, preferably about 0.5 mm.

6. A filter according to any one of Claims 1-5, **characterized** in that the filter also includes one or more other, known filter materials.

7. A filter according to any one of Claims 1-6, **characterized** in that the filter is divided into several compartments through which smoke is caused to flow successively, wherein at least one of the compartments includes at least one zeolite.

8. A filter according to any one of Claims 1-7, **characterized** in that the filter is mounted on a cigarette.

9. A filter according to Claim 8, **characterized** in that the filter is ventilated.

10. A filter according to any one of Claims 1-7, **characterized** in that the filter is arranged to be fitted to a holder for cigarettes, cigar-cigarettes or cigars or in the stem of a tobacco pipe.

Patentansprüche

1. Filter für Tabakrauch, **dadurch gekennzeichnet**, daß der Filter als Filtermaterial zumindest ein Zeolith beinhaltet, das hydrophobe Eigenschaften aufweist, gleichbedeutend damit, daß das Zeolith eine niedrige Affinität für Wasser aufweist.

2. Filter nach Anspruch 1, **dadurch gekennzeichnet**, daß das hydrophobe Zeolith oder die hydrophoben Zeolithe ein Mol-Verhältnis SiO_2/Al_2O_3 hat bzw. haben, das größer als 5,5 ist.

3. Filter nach Anspruch 1 oder 2, **dadurch gekennzeichnet**, daß der Filter zumindest ein Zeolith aus zumindest einer der Arten ST 100, ST 101, ST 102, ST 103 und ST 104 beinhaltet.

4. Filter nach einem der Ansprüche 1 bis 3, **dadurch gekennzeichnet**, daß der Zeolith oder die Zeolithe eine Porengröße von 0,5 nm - 1 nm (5 -10 A), vorzugsweise 0,6 nm - 0,8 nm (6 - 8 A) aufweist bzw. aufweisen.

5. Filter nach einem der Ansprüche 1 bis 4, **dadurch gekennzeichnet**, daß der Zeolith oder die Zeolithe in Form von agglomerierten Körnern verwendet wird bzw. werden, die eine Teilchengröße von 0,2 mm - 1mm, vorzugsweise ungefähr 0,5 mm, aufweisen.

6. Filter nach einem der Ansprüche 1 bis 5, **dadurch gekennzeichnet**, daß der Filter auch ein oder mehrere andere bekannte Filtermaterialien beinhaltet.

7. Filter nach einem der Ansprüche 1 bis 6, **dadurch gekennzeichnet**, daß der Filter in mehrere Zellen unterteilt ist, welche von Rauch nacheinander durchströmt werden, wobei zumindest eine der Zellen zumindest ein Zeolith beinhaltet.

8. Filter nach einem der Ansprüche 1 bis 7, **dadurch gekennzeichnet**, daß der Filter auf einer Zigarette befestigt ist.

9. Filter nach Anspruch 8, **dadurch gekennzeichnet**, daß der Filter belüftet ist.

10. Filter nach einem der Ansprüche 1 bis 7, **dadurch gekennzeichnet**, daß der Filter so ausgebildet ist, daß er in einem Halter für Zigaretten, Zigarren-Zigaretten oder Zigarren oder in dem Stiel einer Tabakpfeife einpaßbar ist.

Revendications

1. Filtre pour fumée de tabac, caractérisé en ce qu'il comprend, comme matériau filtrant, au moins une zéolithe qui présente des propriétés hydrophobes, ce qui signifie que la zéolithe présente une faible affinité pour l'eau.

2. Filtre selon la revendication 1, caractérisé en ce que la ou les zéolithe(s) présente(nt) un rapport molaire SiO_2/Al_2O_3 qui est supérieur à 5,5.

3. Filtre selon la revendication 1 ou 2, caractérisé en ce qu'il comprend au moins une zéolithe d'au moins l'un des types ST 100, ST 101, ST 102, ST 103 et ST 104.

4. Filtre selon l'une quelconque des revendications 1 à 3, caractérisé en ce que la ou les zéolithe(s) présente(nt) une taille de pores de 0,5 à 1 nm (5 à 10 Å), de préférence de 0,6 à 0,8 nm (6 à 8 Å).

5. Filtre selon l'une quelconque des revendications 1 à 4, caractérisé en ce que lion utilise la ou les zéolithe(s) sous la forme de grains agglomérés présentant une taille de particules de 0,2 à 1 mm, de préférence d'environ 0,5 mm.

6. Filtre selon l'une quelconque des revendications 1 à 5, caractérisé en ce qu'il comprend aussi un ou plusieurs autres matériaux de filtre connus.

7. Filtre selon l'une quelconque des revendications 1 à 6, caractérisé en ce qu'il est divisé en plusieurs compartiments à travers lesquels on fait passer successivement la fumée, dans lequel au moins l'un des compartiments comprend au moins une zéolithe.

8. Filtre selon l'une quelconque des revendications 1 à 7, caractérisé en ce qu'il est installé sur une cigarette.

9. Filtre selon la revendication 8, caractérisé en ce que le filtre est aéré.

10. Filtre selon l'une quelconque des revendications 1 à 7, caractérisé en ce qu'il est disposé de manière à s'adapter à un porte-cigarette, un porte-cigare/cigarette ou un porte-cigare, ou dans le tuyau d'une pipe à tabac.