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(54) **CIGARETTE FILTER**

(57) The present invention relates to a multi-component filter comprising a bed of adsorbent, wherein the bed of adsorbent comprises a ground herbal mixture of angelica root and valerian impregnated with honey and vinegar. The invention also relates to a cigarette comprising such a filter.

**EP 3 042 575 A1**

## Description

**[0001]** The present invention relates to filters suitable for cigarettes to filter harmful substances from tobacco smoke.

**[0002]** It has been known for a long time that smoking of tobacco products and in particular smoking of cigarettes is addictive and harmful to the health of human beings. Cigarette smoke, i.e., the smoke generated when a smoker lights a cigarette and draws mainstream smoke from the lit end of the cigarette, contains a large number of toxic substances, in particular nicotine, tar, carbon monoxide, and combustion products from additives.

**[0003]** Nicotine is the major psychoactive constituent of tobacco. Nicotine is addictive and contributes to high blood pressure, but it is not carcinogenic. Tar, however, is carcinogenic and causes the well known "smokers cough". Carbon monoxide displaces the oxygen in blood which results in a decrease of the transport of oxygen and it deteriorates the general condition of the smoker.

**[0004]** Additives which are added to the tobacco by the cigarette manufacturers make cigarette smoke more attractive or even more addictive (i.e., they facilitate the absorption of nicotine) and regulate the moisture content of tobacco. These additives may well constitute 10% of the contemporary cigarette. Examples of substances which are often added are glycerin, sugars, licorice cocoa, menthol, vanilla, and cellulose. When additives are burned new combustion products are generated which may be toxic or pharmacologically active. Known carcinogenic substances in tobacco smoke include benzene, nitrosamines, and formaldehyde.

**[0005]** Manufacturers of cigarettes also put on the market brands of cigarettes which contain less nicotine and less tar, but the concentration of other harmful substances is comparable to that in a traditional cigarette. Moreover, it actually appears that these cigarettes which previously had also been referred to as "light" or "mild" do not yield health benefits, because they do not change the amount of nicotine needed. Smokers of these types of cigarettes will change their habits and smoke in a different way: they smoke more cigarettes, take more puffs per minute, inhale more deeply, they smoke their cigarettes till the very end or pinch off the filter. Therefore, the amount of nicotine and tar inhaled by the smoker will remain substantially the same as compared to smoking a "regular" or traditional cigarette.

**[0006]** Through the years one has tried in many ways to make smoking less attractive in order to help people to get rid of nicotine addiction and to live a healthier life. For example, several nicotine substitutes have been developed to cut back the need for nicotine by decreasing the dosage. In the substitute products nicotine may be present in different amounts and strengths. Examples of such substitutes include the electronic cigarette or "e-cigarette", nicotine patch, inhaler, and nicotine gum. Also, there are herbal cigarettes on the market which contain neither tobacco nor nicotine.

**[0007]** For further background information reference may be made inter alia to the following websites: [www.rokenin-fo.nl/publiek/wetbeleid/tabaksindustrie](http://www.rokenin-fo.nl/publiek/wetbeleid/tabaksindustrie), [www.jellinek.nl/vraagantwoord](http://www.jellinek.nl/vraagantwoord) and [nl.wikipedia.org/wiki/sigarettenrook](http://nl.wikipedia.org/wiki/sigarettenrook).

**[0008]** Cigarettes generally comprise a tobacco rod of shredded tobacco, usually in a cut filler form, surrounded by a paper wrapper ("tapping paper") and a cylindrical filter aligned in an end-to-end relationship with the tobacco rod. Typically, the filter includes a plug of cellulose acetate tow attached to the tobacco rod by tipping paper, also known as "plug wrap". Ventilation of mainstream smoke is achieved with a row or rows of perforations about a location along the filter. Such ventilation provides dilution of drawn mainstream smoke with ambient air to reduce the delivery of tar. The filter plug usually comprises one or more adsorbent materials, such as activated carbon, silica gel, sepiolite, alumina, an ion exchanger, and the like, for the removal of at least one smoke constituent. The filter may also include one or more flavour and/or fragrance dispensing components. Certain cigarettes incorporate a filter element having multiple segments and one of those segments may comprise particles of activated charcoal.

**[0009]** WO 2008/150130 discloses a multi-component cigarette filter comprising a herb flavour filter unit filled with natural herb plant material selected from peppermint, spearmint, coffee, pineapple, chamomile, orange, eucalyptus, thyme, geranium, jasmine, rosemary, lavender, lemongrass, pine needle, clover, sage, taxol, bergamot, basil, valerian, hyssop, tea tree, myrrh and juniper, and mixtures thereof. The herb material may be used in the form of herb granules which may contain a coating made of various sugars.

**[0010]** JP H08-266261 discloses a multi-component cigarette filter comprising crushed plant fibres selected from a large group including *Angelica keiskei* Koidz.

**[0011]** WO 2005/041151 relates to visual content indicators of a tobacco product and discloses inter alia a list of more than 600 additives which are legally permitted to be added to tobacco products. This list includes angelica, valerian, vinegar and honey, but there is no teaching or suggestion whatsoever to combine these specific additives for use in a multi-component cigarette filter.

**[0012]** Although the prior art already discloses a plurality of adsorbing materials for cigarette filters which notably aim to remove tar and other harmful components from (burning) cigarettes while preserving the smoking pleasure, there is still a need for alternative substances and combinations thereof which on the one hand are meant to reduce the harmful health aspects caused by smoking cigarettes and on the other hand aim to provide new flavours and/or fragrances for cigarettes which are attractive for cigarette smokers.

**[0013]** According to the present invention it has been found that this objective can be achieved by a multi-component filter comprising a bed of adsorbent, wherein the adsorbent comprises a herbal mixture of angelica root and valerian,

that is impregnated with honey and vinegar.

**[0014]** The ground and impregnated herbal mixture of plant according to the invention preferably forms a segment of the multi-component filter, which preferably but not necessarily is adjacent at either end to a filter segment of conventional filter material.

**[0015]** Preferably, the ground and impregnated herbal mixture of plant origin is admixed with a substrate that adds to a pressure drop across the filter. This substrate may be made of conventional smoke filtering material, for example finely chopped lint filaments or staple fibers, or activated carbon in particulate form. The combination of the herbal ingredients with a substrate has the advantage that the mixture remains light and thus provides a good ventilation through the filter. Furthermore, the presence of additional smoke filtering material will improve the characteristics of the filter to remove harmful substances. If desired, the substrate may also comprise particles which have little or no filtering effect.

**[0016]** The ratio of the ground and impregnated herbal mixture of plant origin according to the invention and the substrate in the filter segment preferably is in the order of 90 wt.% of the impregnated herbal mixture and 10 wt.% of substrate.

**[0017]** The composition of the herbal mixture according to the invention can be varied within relatively wide limits, but preferably is in the range from 20 to 40 wt.%, more particularly about 30 wt.% of angelica root and from 20 to 40 wt.%, more particularly about 30 wt.% of valerian.

**[0018]** The two herbs, which are commercially available, are coarsely chopped and then mixed. Subsequently, the herbal mixture is immersed in a mixture of about 15 wt.% of vinegar and about 15 wt.% of honey and then dried. If desired, the dried mixture is mixed with about 10 wt.% of a substrate, such as indicated above.

**[0019]** As used herein, the term "vinegar" is meant to indicate any solution from 4 to 15 wt.% acetic acid in water. Suitable forms of vinegar for the purpose of the present invention are, for example, table vinegar and wine vinegar. The term "honey" is meant to indicate any commercially available honey and may be applied for the purpose of the present invention according to the discretion of the manufacturer and/or the user.

**[0020]** The weight of the impregnated and dried herbal mixture according to the invention in the filter is in the order of 20-180 mg, more preferably 30-100 mg and most preferably about 50 mg. The impregnated and dried herbal mixture thus prepared is then put into a rod using a conventional mechanical apparatus and optionally provided at either end with a segment of conventional filter material. The whole is wrapped with paper tapping which preferably has a thin plastic foil on the inside in order to prevent percolation of the honey and vinegar from the herb mixture.

**[0021]** The multi-component filter according to the invention may be manufactured in various ways. First of all, the production of the filter may be part of a continuous production line, as is the case in cigarette factories. Continuous production lines for this purpose are generally known from the production of regular cigarette filters. Another possibility is to produce the herbal filters manually, to combine these filters with commercially available filter housings and to insert a tobacco rod into the filter housing by known means.

**[0022]** The properties of the individual components of the ground and impregnated herbal mixture which forms the core of the present invention are known per se, but they exhibit in this unique combination a surprising and unexpected beneficial effect with regard to the removal of harmful substances, in particular gas phase constituents from the inhaled smoke of burning cigarettes.

**[0023]** Angelica and valerian ("*Valeriana officinalis*") are already known to be purifying means against the toxic, or at least harmful or undesired substances in tobacco, see Al tadawi bi al'shaab, Dr. Amin Rawayha, dar el Qalam, Beirut - Lebanon, 7th edition, 1983, pp. 124, 284.

**[0024]** Vinegar is not only detoxifying, but also antiseptic and restorative, see e.g. [www.leerwiki.nl](http://www.leerwiki.nl).

**[0025]** Honey has an antibacterial and antifungal effect, it protects against cancer and cardiovascular disease, increases endurance and sports performance, regulates blood sugar levels, it is a probiotic and keeps the intestinal flora in balance.

**[0026]** The invention further relates to a cigarette comprising a tobacco rod and the multi-component filter, as defined and described above.

**[0027]** Such a cigarette can be manufactured in various ways which are described in detail in the literature. A person skilled in the art of cigarette manufacturing will have no problems in producing filter cigarettes comprising a multi-component filter according to the present invention.

**[0028]** The invention is further illustrated and explained with reference to the accompanying drawings, wherein:

Figure 1 is a schematic side view of a preferred embodiment of a filter cigarette according to the invention;

Figure 2 is a side view of a cigarette filter according to the invention;

Figure 3 is a cigarette filter as shown in Figure 2, with a schematic representation of the associated tubing of the tobacco rod;

Figure 4 is a microscopic photograph which shows the harmful substances adsorbed by the herbal filter of the invention;

Figure 5 is a microscopic photograph of the top of the upper section of a filter according to Figure 1;

Figure 6 shows a microscopic photograph of the middle section of a filter according to Figure 1.

Figure 7a is a photograph of the unused filter material on an aluminium stub; the areas investigated are within the two circles;

Figure 7b shows two photographs of unused filter material measured at two different positions showing the morphology in SEM-EDX settings;

Figure 7c shows two photographs of unused filter material measured at two different positions evidencing the presence of different elements in colour differences (here different shades of grey);

Figure 8a is a photograph of a slice (tobacco side) of the used filter material on an aluminium stub; the areas investigated are within the two circles;

Figure 8b shows two photographs of used smart filter (tobacco side) measured at two different positions showing the morphology in SEM-EDX settings;

Figure 8c shows two photographs of used smart filter (tobacco side) measured at two different positions evidencing the presence of different elements in colour differences (here different shades of grey);

Figure 9a is a photograph of a slice (mouth side) of the used filter material on an aluminium stub; the areas investigated are within the two circles;

Figure 9b shows two photographs of used smart filter (mouth side) measured at two different positions showing the morphology in SEM-EDX settings;

Figure 9c shows two photographs of used smart filter (mouth side) measured at two different positions evidencing the presence of different elements in colour differences (here different shades of grey).

**[0029]** As shown in Figure 1, the multi-component filter comprises three sections, a lower segment (a) adjacent to the tobacco rod which is made of conventional filter material, a middle segment (b), which comprises the impregnated herbal mixture according to the invention, and the upper segment (c), which is the same as the bottom segment (a) made of conventional filter material. Segment (a), if present, preferably has a length of approximately 3 mm, segment (b) preferably has a length of about 14 mm and segment (c), if present, preferably has a length of about 7 mm.

**[0030]** The multi-component filter according to the invention which comprises a segment with the above-defined impregnated plant herbal mixture, does not differ on the outside from a state of the art cigarette filter, as can be seen in Figure 2 and Figure 3. Also, the taste and the softness of the filter of the invention between the lips is substantially the same for smokers as compared to a conventional filter cigarette. However, the difference internally between the two filters is significant, since the herb filter of the present invention showed improved filtering properties, which will much benefit to the smoker's health.

**[0031]** Microscopic inspection shows the adsorbed amount of harmful substances when using the herbal filter (see Figure 4). This test shows the positive experience of the herbal filter by smokers in addition to the effect against the harmful substances, because they have the feeling of smoking a "normal" cigarette.

**[0032]** The microscopic inspection also shows that the upper and the middle section of the filter have adsorbed about 80% of the harmful gaseous constituents released from the tobacco (see Figure 4).

**[0033]** In Figure 5 it can be seen that the top of the upper section of the filter is not discoloured, which means that the smoker inhaled hardly any harmful substances.

**[0034]** Figure 6 shows the middle section of the filter, where it can be seen that the impregnated herbal mixture according to the invention has adsorbed the harmful substances.

**[0035]** In order to further assess the filtering potential of the cigarette filters according to the present invention, filters were analyzed using the SEM-EDX technique.

**[0036]** A batch of herb filter material was prepared by grinding 3 g angelica and 3 g of valerian. To this mixture 1,5 of honey and 1,5 g of vinegar were added and the blending was continued for 10 min. at room temperature using a pounder. 1,5 g of active coal was then added and the blending was continued for another 20 min. The mixture was then further dried, if necessary, until it has a suitable moisture content of about 5-10%. 50 mg of the mixture is then filled within the filter paper in the middle section of a filter such that the total filter has a length of 24 mm of which 2 x 8 mm of cellulose acetate at either side of the impregnated herbal mixture and 8 mm of the herb mixture, whereas the length of the sleeve filled with tobacco is 61 mm. Thus, the total length of the filter cigarette is 85 mm. The total weight of the filter cigarette is 155 mg, of which 65 mg is contributed by the filter, viz. 50 mg by the impregnated herb mixture and 15 mg by the remaining part of the filter. The filters so produced are hereinafter referred to as "smart filter" and were tested as explained below. Used smart filters in this test were obtained by smoking the filter cigarettes in question by one and the same volunteer in a similar way until 3 mm from the filter and cutting off the burning tobacco..

#### Analytical method

**[0037]** In the Scanning Electron Microscope (SEM), a source of electrons is focused *in vacuo* into a fine probe that is rastered over the surface of the specimen. A detector follows simultaneously the electron beam and collects a reasonable

fraction of the emitted electrons. In this way the whole specimen is scanned. Three types of images are produced by the SEM: secondary electron images, backscattered electron images, and elemental X-ray maps. The primary and secondary electrons are used for topographical information. By using signal modulation and amplification an image is built, which "looks" just like the object. With an X-ray detector information about the elemental composition in the near surface region is obtained.

### Equipment

**[0038]** The analysis was performed with a Hitachi TM-3030 electron microscope showing sample surface morphology in contrast differences. The magnifying range is 15x to 30.000x. The maximum magnification is feasible for ideal samples. The sample must be electrically conductive by itself or coated with an ultrathin layer of electrically-conducting material, commonly gold, deposited on the sample by low vacuum sputter coating.

**[0039]** X-ray microanalysis was performed for determination of the chemical composition of solid samples using an Oxford SwiftED3000 energy dispersive X-ray (EDX) spectrometer system to detect and analyze all elements simultaneously starting from carbon and heavier elements. In this way elemental information from a sample of only a few particles could be obtained and it provided relative detection limits in the order of a tenth of mass percent. A typical penetrating depth is about 1  $\mu\text{m}$  depending on the electron beam and the sample material.

### **Results & Discussion:**

**[0040]** A piece of each sample was placed on an aluminium sample stub and secured with conducting copper tape. In this way the amount of carbon measured will represent purely the carbon of the sample, rather than when working with the commonly used carbon tape to hold the samples in position. All samples were pre-treated in a separate vacuum oven for 5 minutes to prevent contamination of the electron microscope.

**[0041]** For the used smart filter, two slices were made at both ends of the filter, one at the tobacco side and one at the mouth side. Unused smart filter material was also investigated in order to obtain a reference measurement.

**[0042]** Thus, three samples were investigated and the surface of each sample was measured at two different locations. The measured areas are shown in Figures 1a to 3b and the corresponding quantitative elemental results are shown in Tables 1 to 3. For sake of clarity, elements at a concentration < 0.5 wt.% have not been included in the Tables.

**Table 1.** Quantitative summary of the elements encountered in the unused smart filter material by SEM-EDX investigation at two different locations.

Element name	Symbol	Element concentration 1 wt. %	Element concentration 2 wt. %
Carbon	C	44,73	44,98
Oxygen	O	54,38	54,16
Potassium	K	0,89	0,86

**Table 2.** Quantitative summary of the elements encountered in the used smart filter at the tobacco side by SEM-EDX investigation at two different locations.

Element name	Symbol	Element concentration 1 wt. %	Element concentration 2 wt. %
Carbon	C	46,29	47,60
Oxygen	O	52,85	51,55
Potassium	K	0,86	0,86

**Table 3.** Quantitative summary of the elements encountered in the used smart filter at the mouth side by SEM-EDX investigation in two different locations.

Element name	Symbol	Element concentration 1 wt. %	Element concentration 2 wt. %
Carbon	C	63,34	65,63

(continued)

Element name	Symbol	Element concentration 1 wt. %	Element concentration 2 wt. %
Oxygen	O	36,09	34,37
Potassium	K	0,58	0,0

**[0043]** All results were obtained with a 50x magnification. The obtained elemental concentration was gathered by measuring 5 minutes at each location. In this way a substantial amount of signal was gathered and the element concentration could be calculated accurately.

**[0044]** The carbon concentration of the unused smart filter material, obtained at two different locations, was 44.73% and 44.98%, respectively, indicating a perfect repeatability of technique and method for this sample. Also the measured concentrations of the other detected elements (oxygen and potassium) in Table 1 confirm the perfect repeatability. Further elements were not identified to a substantial degree (i.e. several tenths of %).

**[0045]** The used smart filter at the tobacco side was also measured at two different locations and this resulted in a carbon concentration of 46.29% and 47.60%, respectively. Based on the repeatability of the results as evidenced above and presuming that the bare filter material has the same composition in the used and unused smart filter, it can be concluded that a somewhat increased carbon concentration at this side of the used smart filter was present compared to the unused filter material. The amount of carbon increased about 5% compared with the unused smart filter material which is to be attributed to some deposition of carbon-containing species.

**[0046]** When investigating the smart filter at the mouth side, an even more distinct difference in elemental species was observed. When focussing in the region where the black deposits were found which are evidenced as smooth surfaces in Figure 3b, it became clear that these parts were rich in carbon and much poorer in oxygen, which is likely related to the presence of tar-like substances. Table 3 illustrates that the carbon concentration overall is in the order of 64 wt.% whereas oxygen is now much lower at approx. 35 wt.%; clearly an increased concentration of carbon-containing species. Based on the colour differences shown in Figure 3c, it can be concluded that the smooth surface is even richer in carbon than the afore-mentioned values.

**[0047]** Further analysis of the results obtained with the SEM-EDX method as described above leads to the conclusion that the smart filter can filter between 85 and 90% of the harmful substances in cigarette smoke. This conclusion is based on the following calculation.

**[0048]** The difference between the averages of absorbed harmful substances contained in the used and unused smart filter (see Tables 1 and 3) in wt% is shown in Table 5 below:

Table 5

Carbon	64.485 - 44.855	19.63 %
Oxygen	54.27 - 35.23	19.04 %
Potassium	0.875 - 0.29	0.585 %
Total weight in % of absorbed harmful substances		39.255 %

**[0049]** The weight of the smart filter with the herbal mixture is 50 mg. Thus, the amount of harmful substances which was adsorbed by the smart filter is  $39.255 \times 50 \text{ mg} = 19.63 \text{ mg}$ . A cigarette may contain about 23 milligrams of various substances, so called PM2.5 (Particles smaller than 2.5 micrometers); see. <http://tobaccosmoke.exposurescience.org/abcs-of-shs/the-cigarette-is-a-major-source-of-pollution>; [http://en.wikipedia.org/wiki/List\\_of\\_cigarette\\_smoke\\_carcinogens](http://en.wikipedia.org/wiki/List_of_cigarette_smoke_carcinogens); The Chemical Constituents in Cigarettes and Cigarette Smoke: Priorities for Harm Reduction, A Report to the New Zealand Ministry of Health, Jefferson Fowles, Michael Bates, Dominique Noiton, March 2000, pp 16 - 18

**[0050]** The potentially harmful compounds in cigarette smoke are shown in Table 6 below:

Table 6

Component	% per cigarette	mg per cigarette
Tier	40,2	9,246
CO / Gas phase	48,3	11,109

(continued)

Component	% per cigarette	mg per cigarette
Nicotine	4	0,920
Organic vapour phase	7,4	1,702
<b>Total</b>	<b>100</b>	<b>22,977</b>

**[0051]** Our assessment showed that 19.63 mg of the harmful substances were adsorbed by the smart filter, which is  $19,63/23 \times 100\% = \text{about } 85\%$ .

**[0052]** It will be clear to a person skilled in the art that modifications and adaptations can be made to the impregnated herb filter described herein without deviating from the spirit of the invention. These modifications and adaptations therefore all come within the scope of this invention which is determined by the following claims.

### Claims

1. A multi-component filter suitable for use in smoking articles, in particular cigarettes, comprising a bed of adsorbent, wherein the bed of adsorbent comprises a herbal mixture of angelica root and valerian impregnated with honey and vinegar.
2. The multi-component filter of claim 1, wherein the impregnated herbal mixture forms a segment of the multi-component filter which is adjacent at either end to a filter segment of conventional filter material.
3. The multi-component filter of claim 1 or 2, wherein the impregnated herbal mixture is admixed with conventional smoke filtering material, for example, chopped lint filaments, staple fibers, or activated carbon.
4. The multi-component filter of claim 1 or 2, wherein the impregnated herbal mixture is admixed with particles which have little or no filtering effect.
5. The multi-component filter of any one of claims 1 to 4, wherein the composition of the herbal mixture comprises from about 20 to about 40 wt.%, more particularly about 30 wt.% of angelica root, and from about 20 to about 40 wt.%, more particularly about 30 wt.% of valerian.
6. The multi-component filter of claim 5, wherein the herbal mixture is immersed in a mixture of about 15 wt.% of vinegar and about 15 wt.% of honey and then dried.
7. The multi-component filter of any one of claims 1 to 6, wherein the ratio of the impregnated herbal mixture and the substrate in the filter segment is about 90 wt.% of the impregnated herbal mixture and 10 wt.% of substrate.
8. The multi-component filter of any one of claims 1 to 7, wherein the weight of the impregnated and dried herbal mixture in the entire filter is 20-180 mg, more preferably 30-100 mg and most preferably about 50 mg.
9. Cigarette comprising a tobacco rod and the multi-component filter as claimed in any one of claims 1-8.

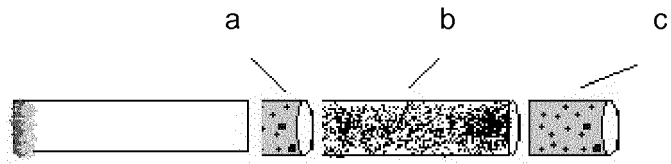


Fig. 1

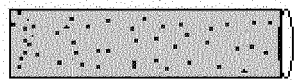


Fig. 2

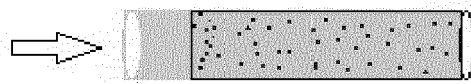


Fig. 3



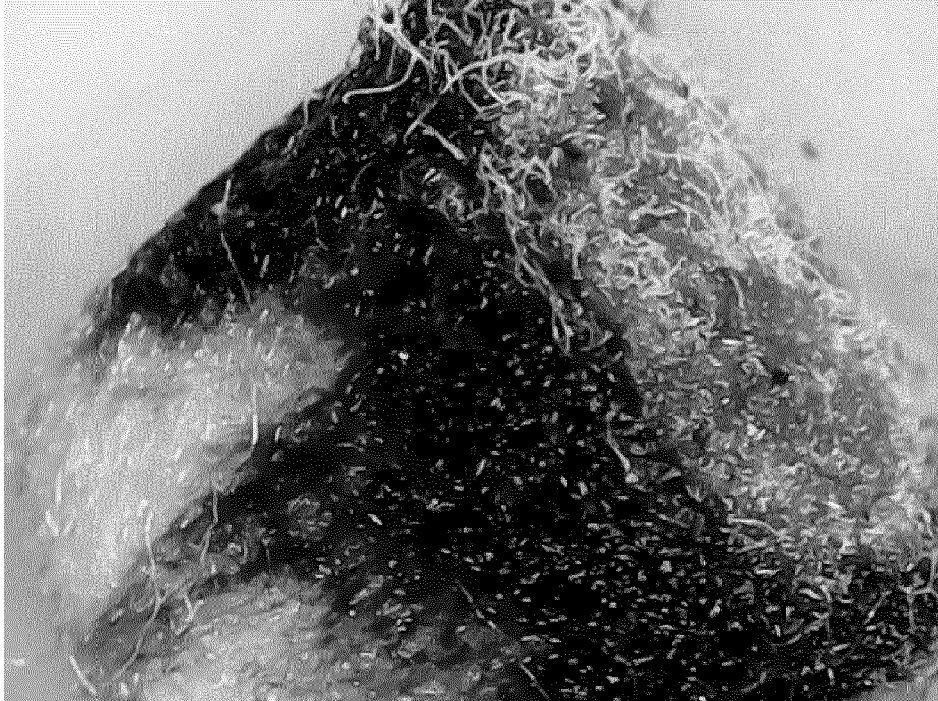


Fig. 4



Fig. 5

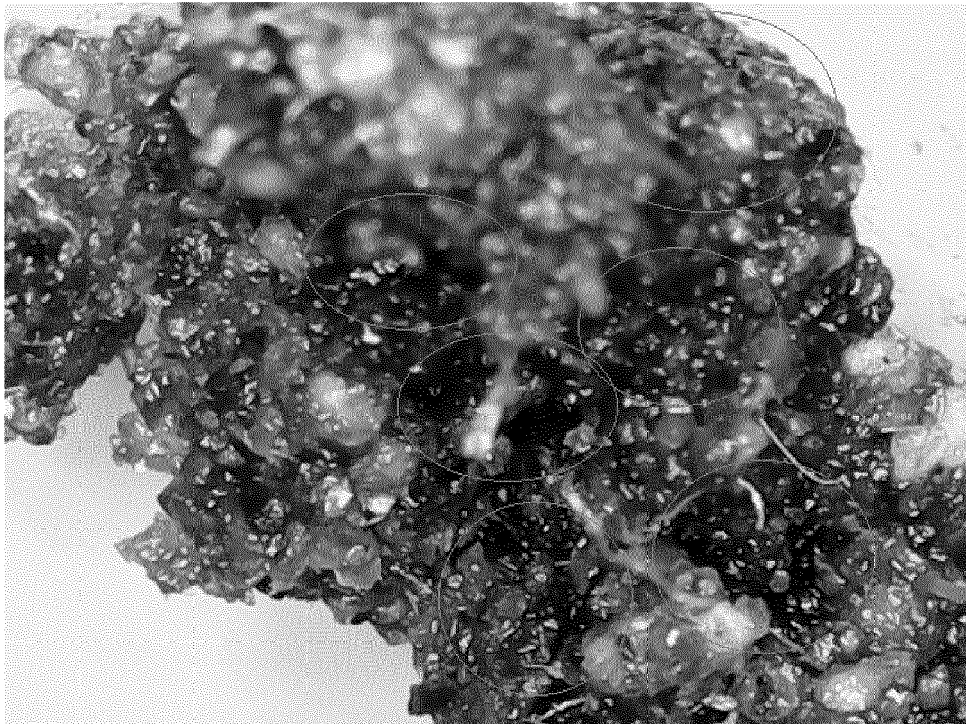


Fig. 6

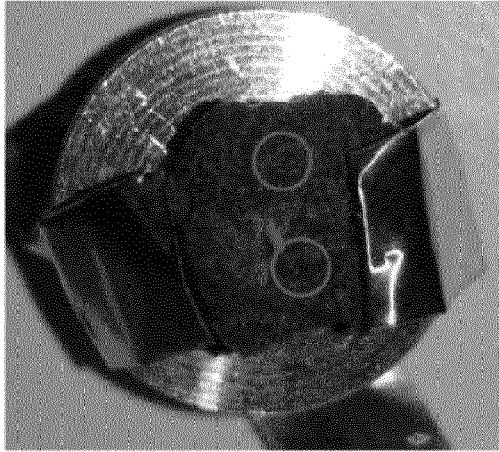


Fig. 7a

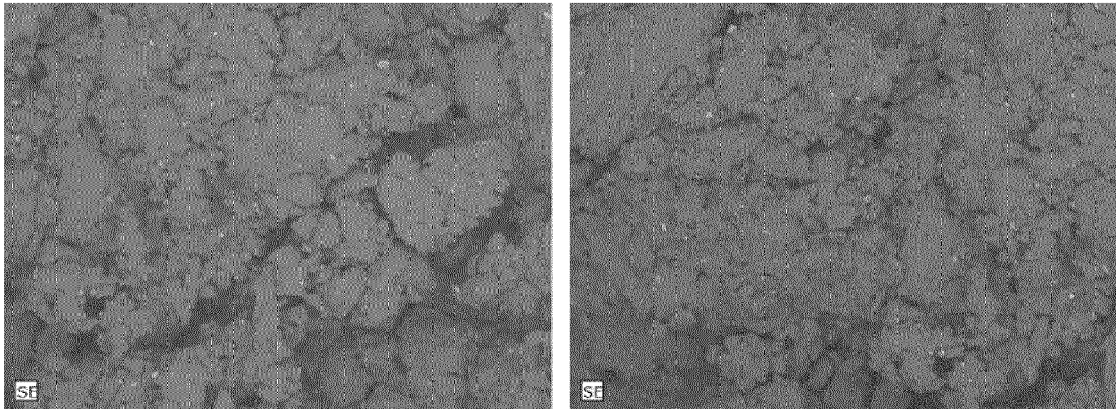


Fig. 7b

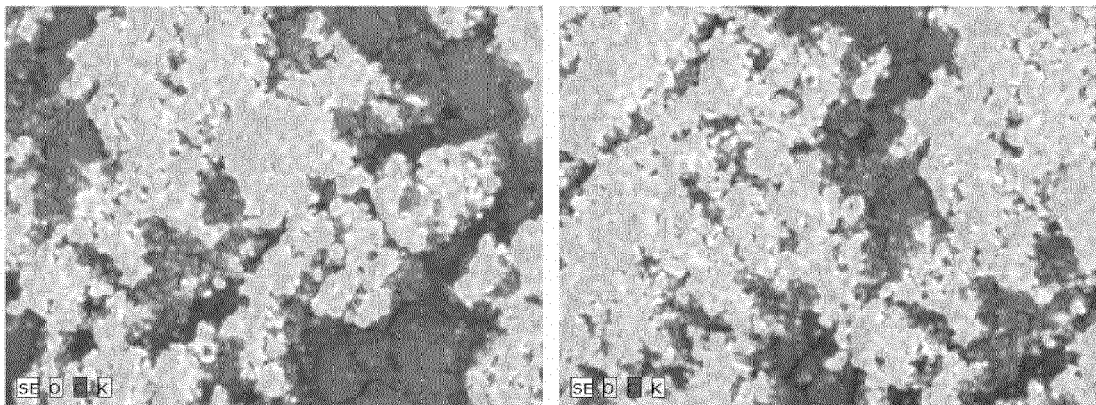


Fig. 7c

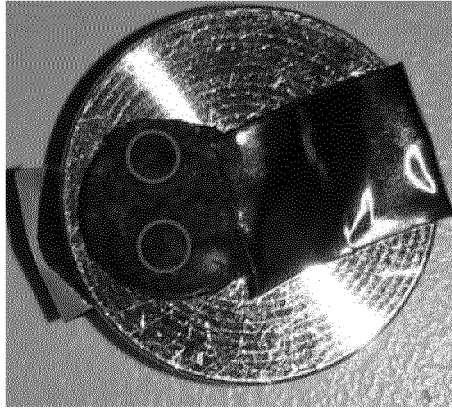


Fig. 8a

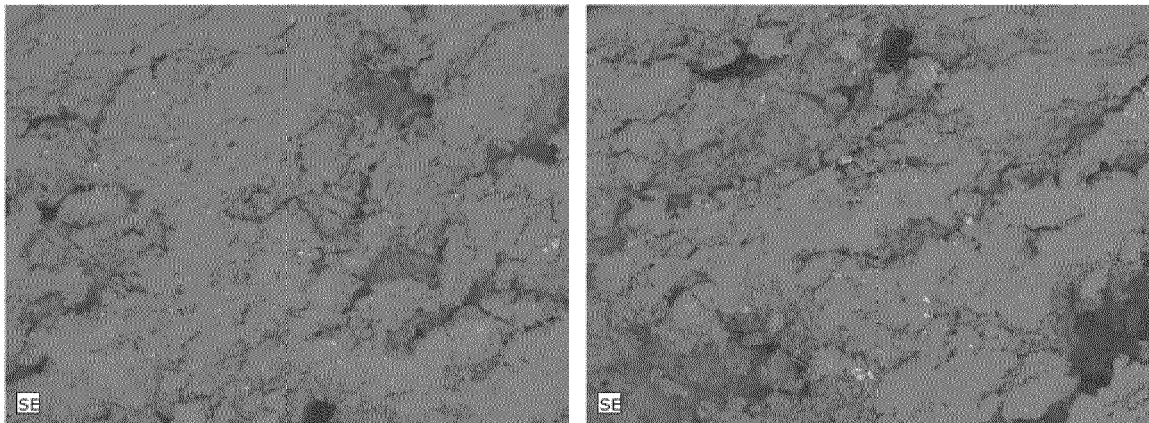


Fig. 8b

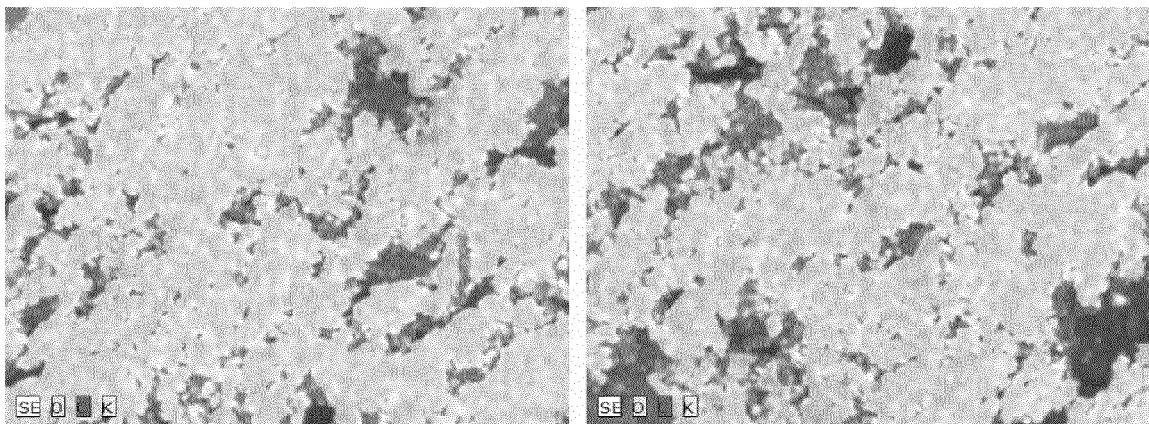


Fig. 8c



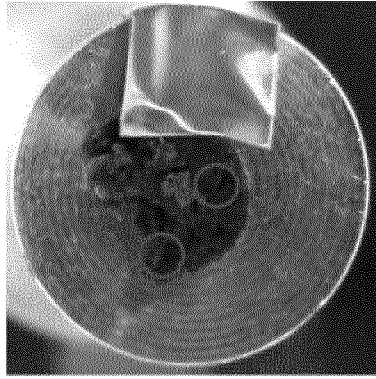


Fig. 9a

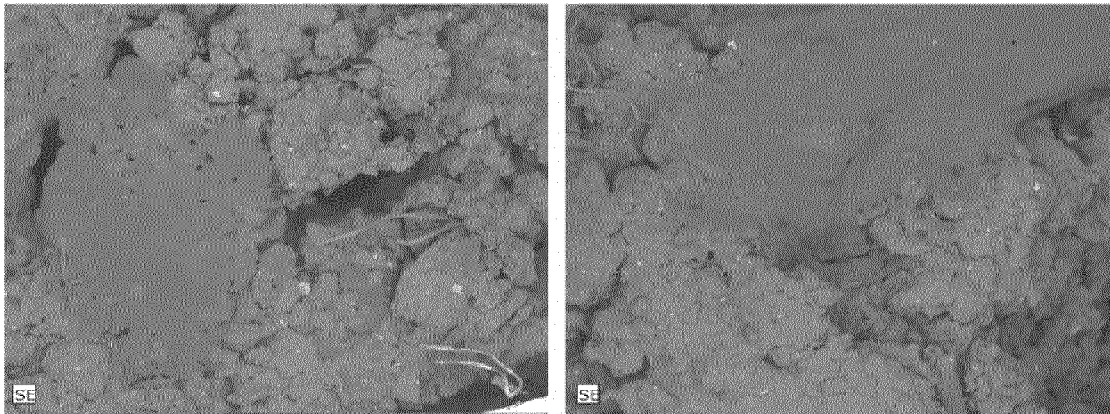


Fig. 9b

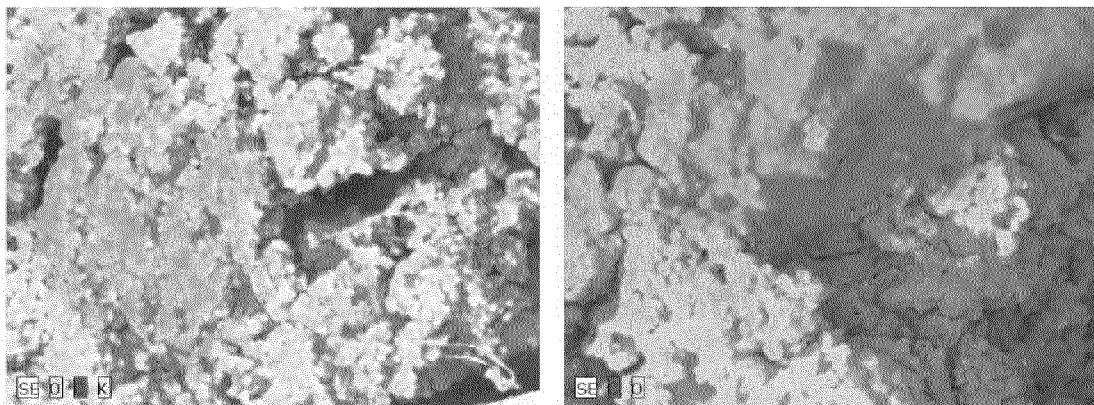


Fig. 9c



EUROPEAN SEARCH REPORT

Application Number  
EP 16 15 0700

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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			A24B A24D
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 25 May 2016	Examiner Leprêtre, François
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