

**We claim:**

- 1) A biodegradable filter material useful for air and smoke filtration, said filter material comprises a macroporous polymeric material wherein said polymeric material comprises a first polysaccharide present in a range varying from 2% to 6% (w/v) per 10.35 ml, a second polysaccharide present in an approximate amount of 0.5% (w/v) per 10.35 ml, a first cross linking agent present in a range varying from 1% to 5% (w/v) per 10.35 ml and a second cross linking agent present in a range varying from 0.055% to 0.55% per 10.35ml of said filter material.
- 2) The biodegradable filter material as claimed in claim 1, wherein the particle filtration efficiency of said filter material is increased by about 28.5% as compared to cellulose acetate filter.
- 3) The biodegradable filter material as claimed in claim 1, wherein said filter material degrades to half its dimension material after 24 hours.
- 4) The biodegradable filter material as claimed in claim 1, wherein said first polysaccharide is selected from a group comprising of naturally occurring starch, hydroxyalkylated starch, starch esters, oxidized starch, plasticized starch, hydrolyzed starch, gelatinized starch, grafted starch, crosslinked starch, transglycosylated starch or like.
- 5) The biodegradable filter material as claimed in claim 1, wherein said second polysaccharide component is selected from a group comprising of guar gum, xanthan gum, locust bean gum, carrageenan, gelatin, chitosan, agarose.
- 6) The biodegradable filter material as claimed in claim 1, wherein said first cross linking agent is glyoxal.
- 7) The biodegradable filter material as claimed in claim 6, wherein said glyoxal has concentration of about 25%.
- 8) The biodegradable filter material as claimed in claim 1, wherein said second cross linking agent is CaO.

- 9) The biodegradable filter material as claimed in claim 8, wherein said CaO has about 0.02 M concentration.
- 10) The biodegradable filter material as claimed in claim 1, wherein the size of said first polysaccharide and second polysaccharide ranges from 500nm to 20 micron.
- 11) A process for the production of a biodegradable filter material useful for air and smoke filtration, said process comprising steps:
- a) Dissolving about 350 to 450 mg of a first polysaccharide in 4ml de-ionized water followed by heating at a temperature range of 80<sup>0</sup>C to 100<sup>0</sup>C for about 30 to 45 minutes to yield solution (A);
  - b) Dissolving about 35 to 75 mg of a second polysaccharide in 5ml de-ionized water followed by heating at temperature range of 50<sup>0</sup>C to 60<sup>0</sup>C for about 30 minutes to an hour to yield solution (B);
  - c) Mixing solution (A) with solution (B) followed by homogenization at a temperature range of 50<sup>0</sup>C to 60<sup>0</sup>C for about 30 minutes to an hour;
  - d) Maintaining the temperature of the solution resulting from step (c) to about 45 to 50<sup>0</sup>C and adding about 250 to 500  $\mu$ l of first cross linking agent followed by the vigorous stirring of the solution for about 10 to 30 seconds.
  - e) Adding about 1ml of second cross linking agent to the solution resulting from step (d) followed by the vigorous stirring of the solution for about 10 to 30 seconds to result into said biodegradable filtration material.
- 12) The process as claimed in claim 11, wherein said first polysaccharide is selected from a group comprising of naturally occurring starch, hydroxyalkylated starch, starch esters, oxidized starch, plasticized starch, hydrolyzed starch, gelatinized starch, grafted starch, crosslinked starch, transglycosylated starch or like.
- 13) The process as claimed in claim 11, wherein said second polysaccharide component is selected from a group comprising of guar gum, xanthan gum, locust bean gum, carrageenan, gelatin, chitosan, agarose.
- 14) The process as claimed in claim 11, wherein said first cross linking agent is glyoxal.

- 15) The process as claimed in claim 14, wherein said glyoxal has concentration of about 25%.
- 16) The process as claimed in claim 11, wherein said second cross linking agent is CaO.
- 17) The process as claimed in claim 11, wherein said CaO has about 0.02 M concentration.
- 18) The process as claimed in claim 17, wherein the concentration of said CaO ranges from 0.055% to 0.55%.
- 19) The process as claimed in claim 11, wherein the size of the first polysaccharide and second polysaccharide ranges from 500nm to 20micron.
- 20) The process as claimed in claim 11, wherein the particle filtration efficiency of said biodegradable filter material is increased by about 28.5% as compared to cellulose acetate filter.
- 21) The process as claimed in claim 11, wherein said biodegradable filter material is degraded to half its dimension material after 24 hours.
- 22) A process of fabricating an object comprising biodegradable filter material claimed in claim 1, wherein said process comprising steps of:
  - a) Pouring said filter material into a moulding object followed by the treatment in a liquid cryostat at a temperature ranging from -15 to -20°C for a period ranging from 5 to 10 hours;
  - b) Freeze drying the moulds resulting from step (a) for a period of about 16 hours.

Dated this 22<sup>nd</sup> day of March 2013



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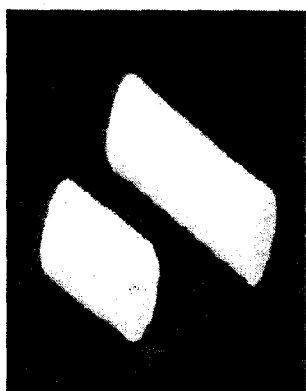


Fig. 1(a)

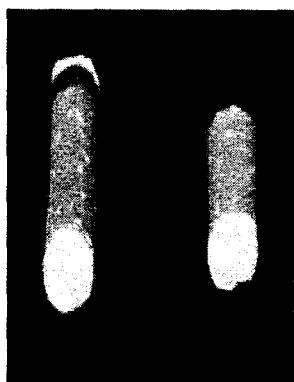


Fig. 1(b)

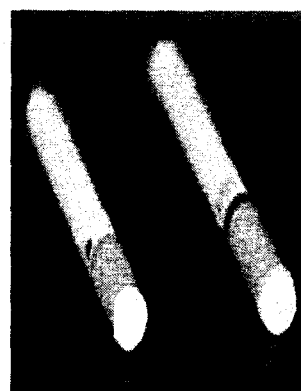


Fig. 1(c)

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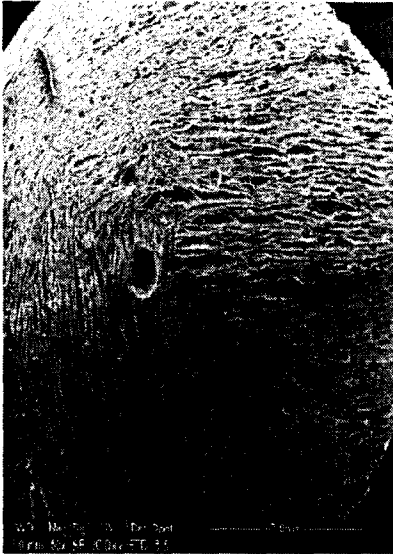


Fig. 2(a)



Fig. 2(c)



Fig. 2(b)



Fig. 2(d)

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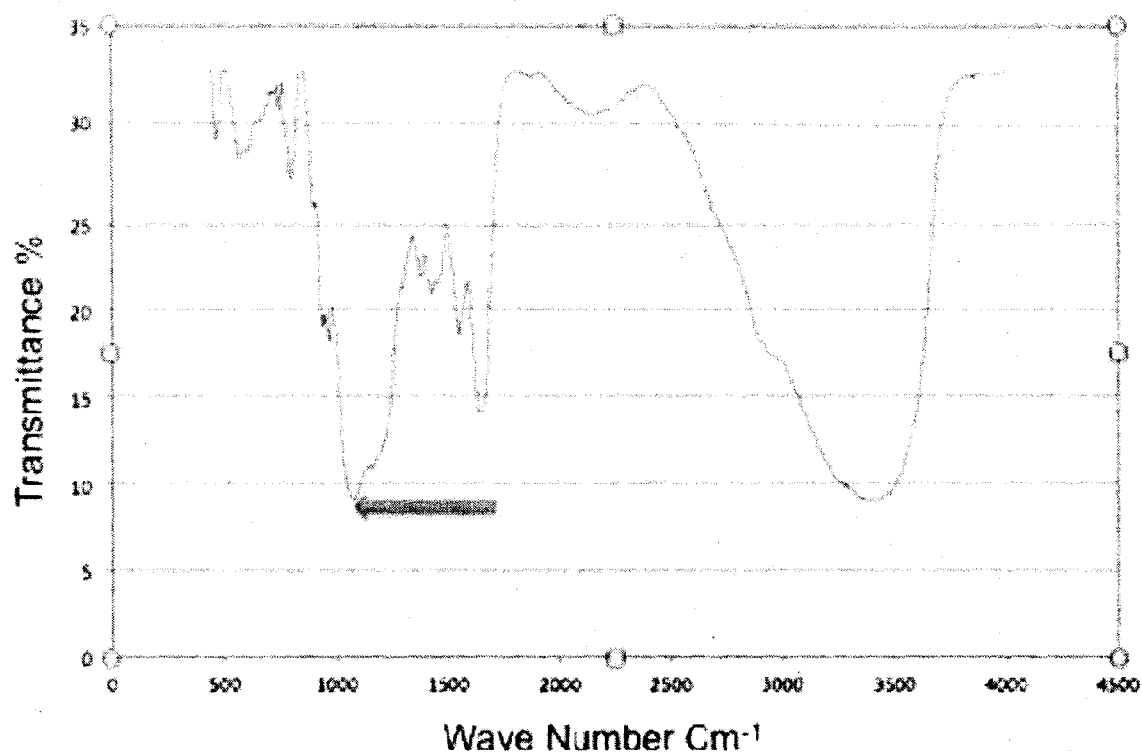
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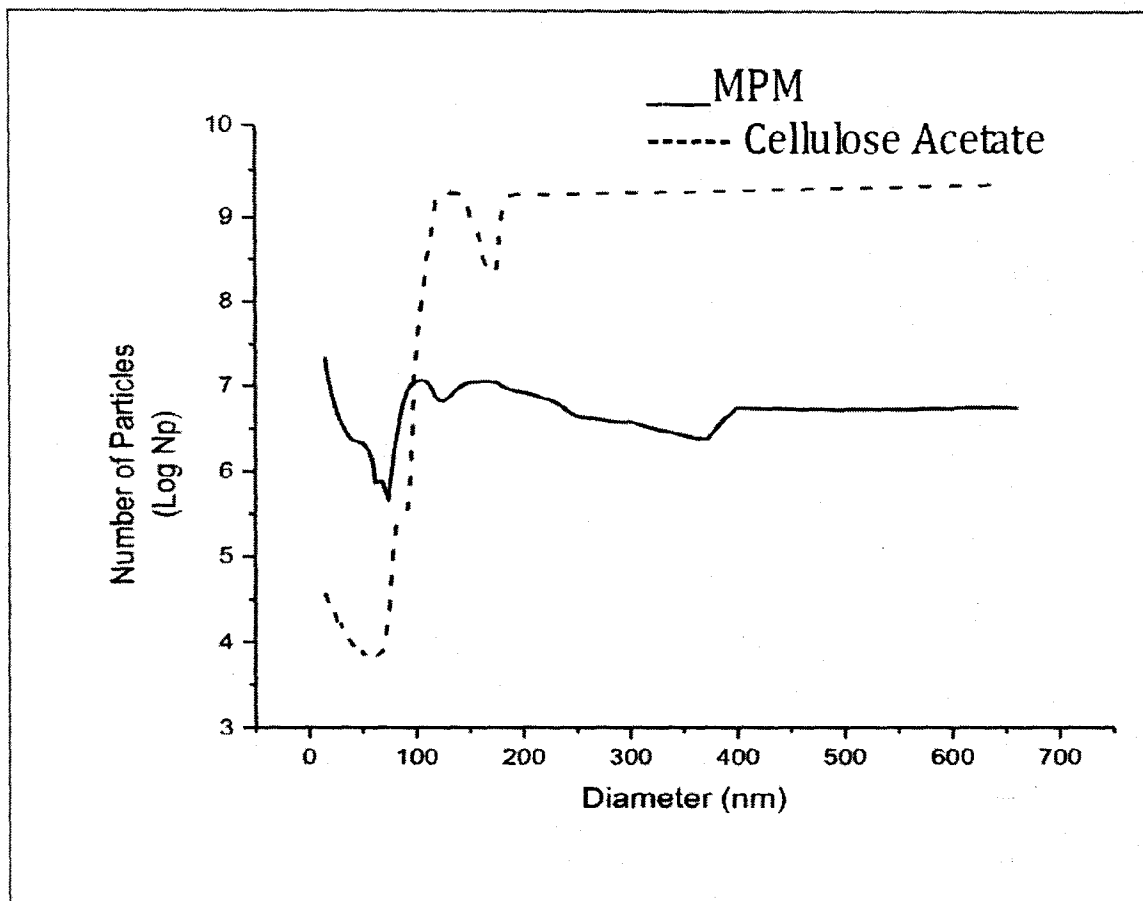
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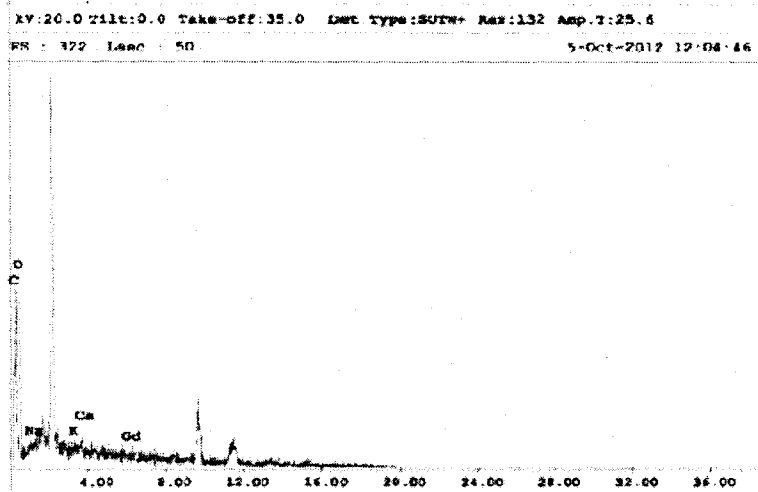


Fig. 5(a)

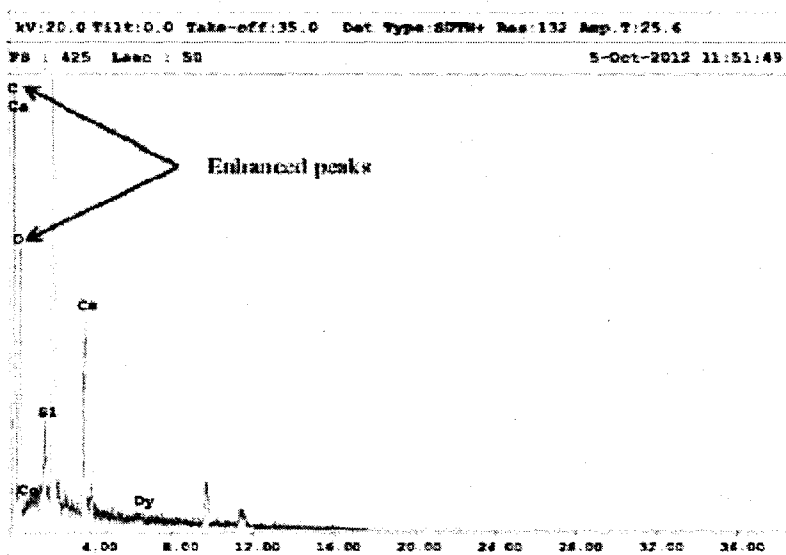


Fig 5(b)

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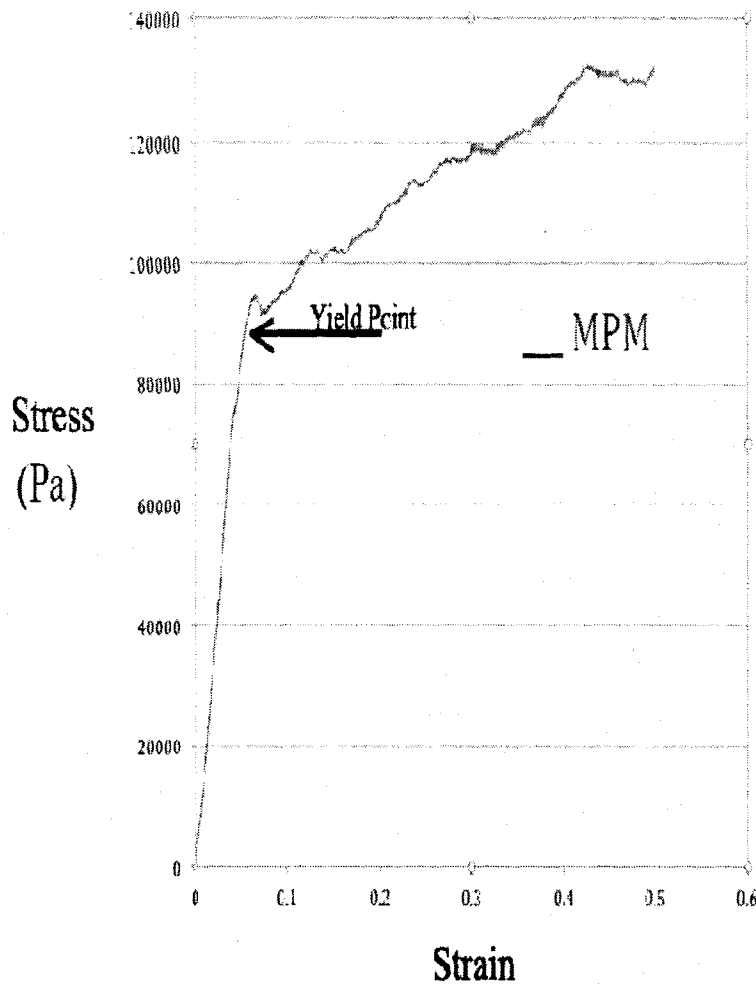
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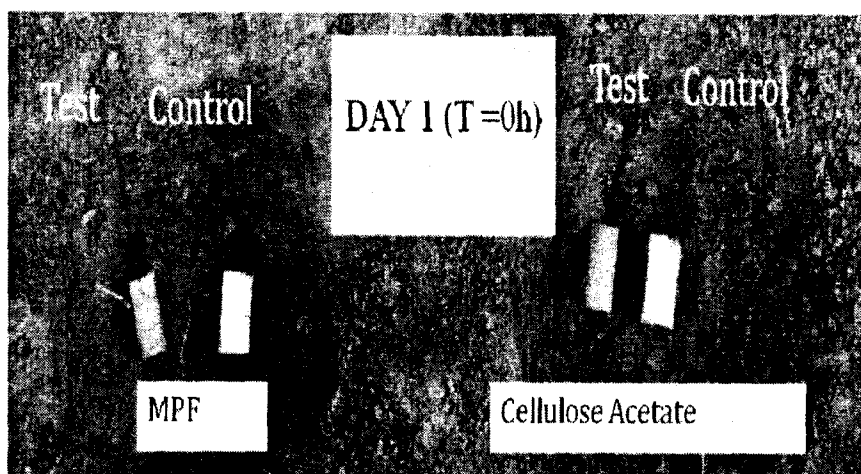


Fig. 7(a)

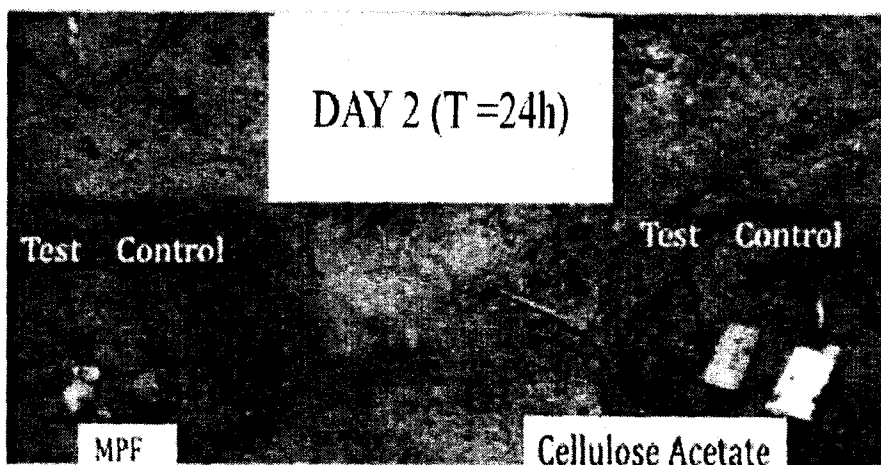


Fig. 7(b)

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**Field of the Invention:**

The present invention relates generally to a biodegradable filter material/composition useful for the purpose of air and smoke filtration. Particularly, there is provided a natural polymer(s) based cigarette filter material/composition.

The filter material/composition advantageously possesses higher filtration efficiency and self degradation efficiency.

**Background and the prior art:**

Cigarette smoking has been known to be practiced for thousands of years and there has been a lot of concern about the hazardous effects of smoking that arises due to several types of particles that are present in the smoke generated during smoking. These particles are proven to be a potential cause for numerous diseases including fatal cancers, respiratory malfunctions, immune system disorders and a lot more. In an attempt to trap these harmful particles "filters" were introduced that would improve the quality of smoke produced due to smoking. With the advent of science and modern technologies a galore of new techniques has been implemented for the development of such filters.

A cigarette filter has the purpose of reducing the amount of smoke, tar and fine particles inhaled during the combustion of a cigarette. Filters also reduce the harshness of the smoke and keep tobacco flakes out of the smoker's mouth.

Cigarette filters are specifically designed to absorb vapors and to accumulate particulate smoke components. Filters also prevent tobacco from entering a smoker's mouth and provide a mouthpiece that will not collapse as the cigarette is smoked. Filters vary in filtration efficiency, depending on whether the cigarette is to be "light" or regular.

One of the conventional filters used now a day is the cellulose acetate filter. Irrespective of its popularity, it has been found that these filters are not very efficient in trapping all harmful particles. The raw material for the manufacture of these cigarette filters is cellulose. The cellulose is acetylated, dissolved and spun as continuous synthetic fibers arranged into a bundle called tow, which is further plasticized, shaped, and cut to length

to act as a filter. Cellulose acetate is biodegradable only in certain chemical forms, such as in the acetic acid ester form of cellulose. In most forms, the biodegradability of cellulose acetate remains relatively low. Further, the biodegradation character of cellulose acetate is most often dependent on the degree of substitution, or the number of acetyl groups per glucose unit of the cellulose acetate molecular structure. For example, if the degree of substitution of cellulose acetate is decreased, the biodegradation rate of cellulose acetate is increased. These filters possess potential environmental threat as they consume a lot of time to degrade (time ranges between 1-10 years) under natural conditions. Also, cellulose acetate is quite expensive which poses a major constraint in the practical use of same in the filter material.

The non-degradable litter generated by these filters is a worrisome issue, as they also possess toxic effects (Register KM. 2000, *Cigarette Butts as Litter, Underwater Naturalist, Bulletin of the American Littoral Society Volume 25*). These filters even can prove more hazardous as they can deliver added toxic components in the mainstream smoke and can have adverse effects rather than proving beneficial (Bradford H. 2011, *The intractable cigarette filter problem, Tob. Control, doi: 10.1136/tc.2010.040113*).

Different techniques have been used that involve coating of different materials over the cellulose fibers, while some make use of material property or affinity in order to trap these harmful properties (Taniguchi et al. 2011, *Cigarette filter material and cigarette material, US Patent No. 7,942,154 B2*).

In other attempts, holes have been made through the filters in order to reduce the tar delivered through the smoke as it leads to increased ventilation (L T Kozlowski, R J O'Connor, G A Giovino, C A Whetzel, J Pauly, and K M Cummings. 2006, *Maximum yields might improve public health- if filter vents were banned: a lesson from the history of vented filters, Tob Control, doi: 10.1136/tc.2006.016501*), but even this technique has also not shown adequate efficiency. Even though a number of companies did adopt the ventilated filters, but research revealed that these filters do not provide any better results with respect to filter properties (L T Kozlowski, R J O'Connor. 2002, *Cigarette filter*

*ventilation is a defective design because of misleading taste, bigger puffs and blocked vents, Tob Control, doi: 10.1136/tc.11.suppl\_1.i40).*

Numerous smoke filters have been developed for reducing the amount of nicotine, and especially tar, present in cigarette smoke introduced into human organism. They may be filters containing additives (e.g. active carbon, charcoal), promoting ventilation or heating, or filters suitable for dispersing smoke. Such are described e.g. in US patents Nos. 3,958,579; 3,882,877; 3,762,422. Their common drawback is that the air resistance is significantly increased by the filter and thus their smoking is rather difficult.

WO2000025611 discloses a cigarette filter which comprises a reagent essentially containing at least one reactive functional group covalently bonded to a non-volatile inorganic substrate. Said reagent contains functional groups like 3-aminopropylsilyl groups which can chemically remove the gaseous component of a smoke stream such as aldehyde component. The filtering agent used in this Invention is synthetic and so does not degrade easily.

On the similar lines, US 3882878 discloses a cigarette filter which comprises cellulose acetate fibers along with a plasticizer agent in an amount of 3 to 10% by weight based on the cellulose acetate fibers. The plasticizer could be a diacetate, dipropionate or dibutyrate ester of 1,3-butylene glycol.

US 5622190 discloses a smoking filter which comprises a first filter plug having a central core of a gathered corrugated web material and a peripheral layer of a cellulose acetate tow filter material surrounding the central core.

US 20120103352 relates to a cigarette filter for selectively and efficiently removing a phenol compound. The filter comprises a cellulose ester tow and a cellulose acetate particle dispersed in the cellulose ester tow and the cellulose acetate particle has the particle size not less than 90% by weight of the cellulose acetate particle passing through

a sieve having an aperture size of 1.7 mm but fail to pass through a sieve having an aperture size of 0.10 mm.

Thus, in the current state of the art, cellular acetate based filter material is being dominantly used for the preparation of filter material for the purpose of air and smoke filtration. However, cellular acetate fibers suffer from the shortcomings of having poor degradability characteristic and therefore it causes harm to the environment. The high cost of cellular acetate poses a great concern to search for an economical alternative suitable to be used in the smoke filters.

Therefore, there is a need of a natural polymer based filtering material which possess high filtration efficiency, quick self degradation capacity and is environment friendly & cost effective.

**Objects of the Invention:**

An object of the present invention is to provide a biodegradable polymer(s) based filtering composition useful for filtering smoke or other chemical particles.

An object of the present invention is to provide a biodegradable polymer(s) based filtering composition having an enhanced particle filtration efficiency as compared to the cellulose acetate based filter material.

An object of the present invention is to provide a biodegradable polymer(s) based filtering composition having about 100% degradability.

An object of the present invention is to provide a biodegradable polymer(s) based filtering composition which has fast self disintegration capacity.

An object of the present invention is to provide a biodegradable polymer(s) based filtering composition which helps in achieving the desired pressure drop while smoking.

An object of the present invention is to provide an economical biodegradable polymer(s) based filtering composition.

These and other advantages of the present invention will become readily apparent from the following detailed description taken in conjunction with the accompanying drawings.

### **Summary of the Invention:**

The following presents a simplified summary of the invention in order to provide a basic understanding of some aspects of the invention. This summary is not an extensive overview of the present invention. It is not intended to identify the key/critical elements of the invention or to delineate the scope of the invention. Its sole purpose is to present some concept of the invention in a simplified form as a prelude to a more detailed description of the invention presented later.

In order to achieve said object, the present invention provides a biodegradable filter material/composition useful for air and smoke filtration. The filter material/composition comprises a macroporous polymeric material wherein said polymeric material comprises a first polysaccharide present in a range varying from 2% to 6%(w/v) per 10.35 ml, a second polysaccharide present in an approximate amount of 0.5% (w/v) per 10.35 ml, a first cross linking agent present in a range varying from 1% to 5% (w/v) per 10.35 ml and a second cross linking agent present in a range varying from 0.055% to 0.55% per 10.35ml of said filter material.

In another aspect of the Invention, there is provided a process for the production of a biodegradable filter material useful for air and smoke filtration, said process comprising steps:

- a) Dissolving about 350 to 450 mg of a first polysaccharide in 4ml de-ionized water followed by heating at a temperature range of 80<sup>0</sup>C to 100<sup>0</sup>C for about 30 to 45 minutes to yield solution (A);

- b) Dissolving about 35 to 75 mg of a second polysaccharide in 5ml de-ionized water followed by heating at temperature range of 50<sup>0</sup>C to 60<sup>0</sup>C for about 30 minutes to an hour to yield solution (B);
- c) Mixing solution (A) with solution (B) followed by homogenization at a temperature range of 50<sup>0</sup>C to 60<sup>0</sup>C for about 30 minutes to an hour;
- d) Maintaining the temperature of the solution resulting from step (c) to about 45 to 50<sup>0</sup>C and adding about 250 to 500 µl of first cross linking agent followed by the vigorous stirring of the solution for about 10 to 30 seconds.
- e) Adding about 1ml of second cross linking agent to the solution resulting from step (d) followed by the vigorous stirring of the solution for about 10 to 30 seconds to result into said biodegradable filtration material.

In another aspect of the Invention, there is provided a process of fabricating an object comprising above said biodegradable filter material. The process of fabricating an object comprises following steps:

- a) Pouring the filter material/composition into a moulding object followed by the treatment in a liquid cryostat at a temperature ranging from -15 to -20<sup>0</sup>C for a period ranging from 5 to 10 hours;
- b) Freeze drying the moulds resulting from step (a) for a period of about 16 hours.

Other aspects, advantages, and salient features of the invention will become apparent to those skilled in the art from the following detailed description, which, taken in conjunction with the annexed drawings, discloses exemplary embodiments of the invention.

**Brief description of the accompanying drawings:**

The above and other aspects, features, and advantages of certain exemplary embodiments of the present invention will be more apparent from the following description taken in conjunction with the accompanying drawings in which:



Figure 1: Representation of Un-encapsulated 1(a), Encapsulated 1(b) MPM and MPM fitted within a cigarette 1(c), respectively.

Figure 2: SEM images of MPM filters.

Figure 3: FTIR representation of MPM

Figure 4. Graphical representation of particle analysis of MPM vs. cellulose acetate.

Figure 5: Elemental analysis of MPM

Figure 6: Stress vs. Strain curve of MPM.

Persons skilled in the art will appreciate that elements in the figures are illustrated for simplicity and clarity and may have not been drawn to scale. For example, the dimensions of some of the elements in the figure may be exaggerated relative to other elements to help to improve understanding of various exemplary embodiments of the present disclosure.

#### **Detailed description of the invention:**

The following description is of exemplary embodiments only and is not intended to limit the scope, applicability or configuration of the invention in any way. Rather, the following description provides a convenient illustration for implementing exemplary embodiments of the invention. Various changes to the described embodiments may be made in the function and arrangement of the elements described without departing from the scope of the invention.

Accordingly, the invention provides a biodegradable filter material appropriate to be used as air and smoke filter. The filter material comprises advantageous characteristic of having complete and quick degradability. The filter material also possess a surprising feature of having high filtration efficiency as compared to a cellulose acetate based filter material.

Particularly, the Invention provides a highly efficient cigarette smoke filter material/composition by making use of specific Macroporous Polymeric Materials (MPM). Generally, MPM are highly porous and are interconnected structures, which are formed by crosslinking of different polymer(s) by using a crosslinker agent. The pore

morphology of these polymeric material is beneficial in providing the desired pressure drop and also facilitates the efficient trapping of harmful tar particles that originate from cigarette smoking.

Particularly, the selection of natural polymers is responsible for achieving the higher capture efficiency of toxic components from smoke. The filter material comprises optimized concentration of these macroporous polymer which were further evaluated by a series of tests in order to investigate their potential as cigarette smoke filters.

The term "starch" as used herein refers to a naturally abundant nutrient carbohydrate,  $(C_6H_{10}O_5)_n$ , found chiefly in the seeds, fruits, tubers, roots, and stem pith of plants, notably in corn, potatoes, wheat, and rice, and varying widely in appearance according to source but commonly prepared as a white amorphous tasteless powder.

As used herein, the term "smoking article" includes smokeable products such as cigarettes, cigars and cigarillos whether based on tobacco, tobacco derivatives, expanded tobacco, reconstituted tobacco or tobacco substitutes and also heat-not-burn products.

#### **Detailed Process:**

##### **Synthesis of macroporous polymeric materials (MPM) as smoke filters:**

The biodegradable filter material/composition comprises macroporous polymeric material. The macroporous polymeric material essentially comprises combination of polysaccharide(s) and cross linking agent(s) in a synergistic manner which results into increased filtration efficiency and high self degradation capacity of the resulting filter material/composition.

The macroporous polymeric material comprises following components:

- a) A first polysaccharide(s) present in a range varying from 2% to 6% (w/v) per 10.35 ml,
- b) A second polysaccharide(s) present in an approximate amount of 0.5% (w/v) per 10.35 ml,

- c) A first cross linking agent(s) present in a range varying from 1% to 5% (w/v) per 10.35 ml
- d) A second cross linking agent(s) present in a range varying from 0.055% to 0.55% per 10.35ml of said filter material/composition.

The first polysaccharide is selected from a group comprising of naturally occurring starch, hydroxyalkylated starch, starch esters, oxidized starch, plasticized starch, hydrolyzed starch, gelatinized starch, grafted starch, crosslinked starch, transglycosylated starch or like. However, in a preferable embodiment, the first polysaccharide comprises a naturally occurring starch.

While, the second polysaccharide component is selected from a group comprising of guar gum, xanthan gum, locust bean gum, carrageenan, gelatin, chitosan, agarose. In another preferred embodiment, guar gum is utilized as the second polysaccharide component.

The first cross linking agent is Glyoxal with about 25% concentration while the second cross linking agent is preferably CaO with about 0.02 M concentration. However, any other chemical agent with similar characteristics as of Glyoxal might also be utilized for this purpose and the Invention is further not restricted to use CaO as the source of calcium and the second cross linking agent in the proposed filter material, instead any other calcium source can also be used for the same purpose. CaO is mainly used as a crosslinking agent, but it may be possible that it can act as an absorbent too.

The size of starch and guar gum ranges between 500nm-20micron. Further, the Starch has been derived from potato and majorly comprises of amylopectin only.

The current polymeric concentration is the optimized concentration. However, it is always possible to change the %composition but it will have a direct effect on the bulk properties of the material like average pore size, flow property, mechanical property.

Said filter has the surprising effect of achieving about 28.5% higher particle filtration efficiency as compared to the conventional cellulose acetate. The data is well substantiated by the henceforth defined experimental results. Figure 4 shows the

graphical representation of the smoke particle capturing efficiency. The filter material also possess 100% degradability. The degradation study shows that the filter material is reduced to half its original dimension after 24 hours and there is almost complete degradability of the filters in less than a week. The details of the study is discussed in the henceforth mentioned experimental results.

Another aspect of the Invention provides a process for the production of above said biodegradable filter material. The process essentially comprises following steps:

- a) Dissolving about 350 to 450 mg of a first polysaccharide in 4ml de-ionized water followed by heating at a temperature range of 80<sup>0</sup>C to 100<sup>0</sup>C for about 30 to 45 minutes to yield solution (A);
- b) Dissolving about 35 to 75 mg of a second polysaccharide in 5ml de-ionized water followed by heating at temperature range of 50<sup>0</sup>C to 60<sup>0</sup>C for about 30 minutes to an hour to yield solution (B);
- c) Mixing solution (A) with solution (B) followed by homogenization at a temperature range of 50<sup>0</sup>C to 60<sup>0</sup>C for about 30 minutes to an hour;
- d) Maintaining the temperature of the solution resulting from step (c) to about 45 to 50<sup>0</sup>C and adding about 250 to 500 µl of first cross linking agent followed by the vigorous stirring of the solution for about 10 to 30 seconds.
- e) Adding about 1ml of second cross linking agent to the solution resulting from step (d) followed by the vigorous stirring of the solution for about 10 to 30 seconds to result into said biodegradable filtration material.

#### **Detailed process for the production of filter material:**

#### **Experimental Details:**

For achieving the above said biodegradable filter material, firstly a starch solution is made by dissolving approximately 350 to 450 mg of the starch in 4 ml de-ionized water to make the solution (A). The solution (A) is heated at about 80 to 100<sup>0</sup>C for about 30 to

45 minutes in a circulated water bath with frequent stirring until solution becomes clear. The clear solution of the mixture shows complete dissolution of starch molecules.

Further, solution (B) is prepared by mixing about 35 to 75 mg guar gum in 5 ml de-ionized water and then the solution is heated at 50 to 60°C for about 30 minutes to an hour to solubilize the guar gum and for ensuring that the viscosity of the resulting solution is substantially reduced and remains constant.

The prepared starch solution i.e. solution (A) and the guar gum solution i.e. solution (B) is mixed and placed on a magnetic stirrer with mild stirring in order to homogenize the resulting solution.

The temperature of the magnetic stirring unit is maintained at 50 to 60°C and the process is continued for about 30 minutes to an hour. After the polymer solution is homogenized, the temperature of the stirring unit is reduced to about 45 to 50°C. As the resulting solution temperature reaches to about 45 to 50°C, thereafter about 250 to 500 µl of first cross linking agent like glyoxal (25%) is further added to the final solution of solution (A) and solution (B).

The resulting mixture is then vigorously stirred for about 10 to 30 second and then about 1 ml of 0.02 M of second cross linking agent like CaO is immediately added to the resulting solution followed by fast stirring for another 10 to 30 seconds. The CaO solution was prepared by weighing 11.2 mg CaO and mixing it in 10 ml of water. From this stock, 1 ml CaO has been used for the experiments. The stirring is done mechanically using a vortexer in a speed of about 100-500 rpm. A magnetic stirrer or an agitator can also be used at similar speeds.

Resultantly, a total volume of 10.35 ml is produced by mixing all the ingredients which gives starch concentration of about 4% (w/v) and the guar-gum concentration of about 0.5% (w/v).

After the cross linkers are mixed thoroughly, the reaction mixture is immediately poured into plastic moulds in the form of four syringes of 9 mm internal diameter and 2.5 ml volume. The syringes are then placed into a liquid cryostat maintained at -20 °C and allowed to freeze completely for a period of 5 to 6 hour. Here, the freezing temperature may range between -15 to -20 °C while freezing time can also range between 5-10 hours. The moulds are then placed in a freeze-dryer for a period of 16 hour until they are completely dry and when there is no ice crystals present. The samples are further kept at room temperature for sometime before they can be used.

## **Experimental Results:**

### **1. Scanning Electron Microscopy (SEM):**

SEM analysis was performed by cutting the samples into circular discs of 8 mm diameter and 2 mm height. It was ensured that the samples were completely dry. The samples were gold coated using the Cressington sputter coater at a current of 40 mA for a period of 90 seconds. After coating the samples, the samples were placed in a desiccator until they were examined in order to keep them moisture free.

Figure 2 (a, b) represents the control with no smoke. Figure 2 (c, d) represents trapped particles when smoke was passed through MPM at 25°C and 60% relative humidity. It is evident from the images, that the harmful tar particles and other hydrocarbons have been trapped within the pores of the MPM with high efficiency. On comparing the surfaces of the control and test samples it can be noticed that the control samples have a smoother surface as compared with the rough surface of the test samples. This is because a lot of harmful tar particles have been trapped incase of the test samples that makes their surface look rough.

### **2. FT-IR Spectroscopy:**

Figure 3 shows Fourier transform infrared spectroscopy (FTIR) representation of MPM. The peak indicated by the arrow at 1108 reveals the dissolution of the aldehyde of

Glyoxal in Hydroxyl group of Starch to form a hemiacetal by nucleophilic substitution. This peak ensures cross-linking of starch with glyoxal.

### **3. Particle Analysis: Comparison between MPM and Cellulose Acetate:**

Particle analysis was performed on the scanning mobility particle size machine at 25°C and 60% relative humidity. The filters i.e. the MPM and cellulose acetate filter were fitted within 69mm cigarettes and the cigarettes were placed in the test end of the equipment in order to quantify the number of particles that emanated from the cigarette smoke through both the filters. It was observed that the MPM performed at least  $10^2$  times better than the conventional cellulose acetate filters when average number of particles was calculated as shown in Figure 4 which shows the graphical representation of the particle analysis of MPM vs. cellulose acetate. The X-axis represents the diameter of the particles in the cigarette smoke and Y-axis represents the number of particles on the logarithmic scale. The higher filtration efficiency of the filter material is the advantageous feature of the present Invention.

### **4. Pressure Drop:**

#### **Comparison between MPM and Cellulose Acetate:**

To analyze the back pressure of the filters, filters of different lengths were placed in the test column of a pressure drop (Cerulean PD Express) machine in such a way that the samples were completely sealed and it was ensured that no air could pass through the sides. The flow rate from the air compressor was kept constant at 15 l/min and the pressure maintained during the test was 60 psi. Different pressure drop readings were taken as shown in table 1 above. Back pressure analysis revealed no significant difference in pressure drop of cigarettes fitted with MPM vs. the conventional filter. In real use also when cigarettes fitted with MPM were given to people for smoking, no difference in pressure drop was observed by any person as compared to normal cellulose acetate filter fitted cigarettes. This was observed randomly for all age group and gender for more than

100 numbers. The below table demonstrates the comparison of pressure drop values in cellulose acetate filter vs. MPM filters.

Filter Type		Filter Length (In mm)	Pressure Drop (In mmWg)	
A	Cellulose Acetate (Un-Encapsulated)	15	37.8	
		27	51.3	
B	Cellulose Acetate (Encapsulated)	15	Only Filter	Filter With Cigarette
			53.1	84.7*
		27	63.7	94.1**
C	MPM (Un-Encapsulated)	15	65.2	
		27	91.6	
D	MPM (Encapsulated)	15	Only Filter	Filter With Cigarette
			69.7	94.2*
		27	94.5	110.3**

\*The length of the cigarette is 69 mm.      \*\*The length of the cigarette is 88 mm.

### **5. Energy Dispersive X-Ray Analysis (EDAX)- Elemental Analysis**

The elemental analysis was performed on equal cross section area for both control and test filter samples. Results indicated about enhanced Carbon and Oxygen peaks incase of the MPM samples with smoke confirming the presence of harmful tar and other hydrocarbon particles. Figure 5 shows the elemental analysis of MPM. The EDAX analysis was performed on both control MPM samples with no smoke and test MPM



samples that were subjected to cigarette smoke as shown in Fig. 5(a) and 5(b), respectively.

## **6. Compressive Strength:**

The compressive strength of the filter material was calculated on the Bose Electroforce mechanical testing machine. The ratio of height and the diameter were kept same (8mm) for all MPM samples. The compressive strength calculated from the graph was approximately  $84 \pm 15$  kPa. After the yield point, the filter material exhibited brittle characteristics and fractures were observed after 50% deformation. Figure 6 shows Stress vs. Strain curve of MPM. The X-axis represents Strain and Y-axis represents Stress experienced by the MPM filters during the mechanical testing. From these results, it can be concluded that the MPM possess enough strength in order to be used as cigarette filters and shows desired stability as filter.

## **7. Degradation Study**

It was observed that MPM degraded at a remarkable rate and after 24hour it had reduced to half its original dimension while no significant change was observed in the cellulose acetate filters with and without smoke. Almost complete degradation of MPM filters has been observed within less than one week in natural and wet environment. In natural and dry environment, the filter material of MPM filters was quite stable. However, the same was not observed in the conventional filters of cellulose acetate, which may take years for complete degradation. This quick and 100% degradability of the filter material is the advantageous feature of the present Invention.

Figure 7 shows the degradation study of MPM and Cellulose acetate Filter. Figure 7 (a, b) represent the degradation rate of the two materials at two time intervals,  $T=0$  hour and  $T=24$  hour. The filter length and diameter was kept the same in both MPM and Cellulose acetated filter (15mm). From these results, it can be concluded that the MPM possess enough strength in order to be used as cigarette filters and shows desired stability as filter.

The samples were placed in soil in order to see the degradation behavior under natural conditions. The test samples represent smoke subjected MPM and Cellulose Acetate filters, while the control included no smoke.

In another aspect of the Invention, there is provided a process of fabricating an object comprising above said biodegradable filter material.

The process comprises following steps:

- a) pouring said filter material into a moulding object followed by the treatment in a liquid cryostat at about -15 to -20 °C for a period ranging from 5 to 10 hours;
- b) Freeze drying the moulds resulting from step (a) for a period of 16 hours.

The aforesaid object is preferably a smoking article but not restricted to the same. The smoking article includes any smokeable products such as cigarettes, cigars and cigarillos.

The freeze drying method used for the production of smokeable article helps in achieving the channeled pores and high pore volume (as supported by SEM images) which resultantly enables minimal resistance to the flow of smoke. Figure 1 shows representation of Un-encapsulated 1(a), Encapsulated 1(b) MPM and MPM fitted within a cigarette 1(c) respectively.

The present invention will be explained further with reference to non-limiting embodiments of the invention.

In an embodiment of the present invention, there is provided a filter material which is more readily degradable than filter elements comprising a conventional cellulose acetate filter material.

In another embodiment of the present invention, there is provided a filter material which exhibits good filtration efficiency.

In another embodiment of the present invention, there is provided a filter material which comprises macroporous polymeric material along with additives like mint, clove oil, apple water etc. for facilitating better taste and better smoking experience.

In another embodiment of the present invention, there is provided a smoking article comprising aforesaid filter material attached to the rod of smokeable material. The smoking article may be a cigarette. The smokeable filler material may be tobacco material or a tobacco substitute material. Preferably the smokeable material is a tobacco material. Suitably the tobacco material comprises one or more of stem, lamina, and tobacco dust.

The invention will now be explained with the help of following examples. However, the scope of the invention should not be limited to these examples as the person skilled in the art can easily vary the proportion of the ingredients and combinations.

**Example 1:**

About 370mg of the starch was dissolved in 4ml de-ionized water followed by heating at 90°C for about 40 minutes. Further 50 mg of the guar gum was dissolved in 5ml de-ionized water and heated at 55°C for about 50 minutes and finally the starch solution and guar gum solution was mixed at the temperature of 60°C. Thereafter, 290 µl of glyoxal was added to the resulting solution followed by vigorous stirring of the solution for about 20 seconds. Further after, 1ml of calcium oxide is added followed by the vigorous stirring of the solution for about 30 seconds to result into said biodegradable filtration material.

**Example 2:**

The smoke filter was fitted at the back of a number of cigarettes manually and many regular smokers (around 100 nos.) were made to smoke and they hardly could notice any difference in back pressure or taste of the cigarette smoke. Some smokers reported lightness in the smoke taste.

**Example 3:**

After the filters were synthesized, the filters were cut into desired length. The filters were manually fitted at the back of a number of cigarettes. The test samples were then placed into the test inlet of a particle analyzer. After the machine was set to “test” mode, the cigarette was lit using a lighter and the machine simulated real smoking conditions. All the particles emanating from the smoke and passing the filter barrier were counted electronically by the machine until whole cigarette was used up. The result was compared with that of the conventional cellulose filter by plotting a graph. The result revealed better performance of the newly designed filter material.

**Example 4:**

In order to measure the back pressure of the synthesized filters, filters of different lengths were cut. Some of the filters were even covered with paper in order to ensure that no air could pass through the sides while some were fitted in the cigarettes. Every type of sample was then placed in the test area of a cerulean PD express machine. The machine was connected with a compressor as a source of air. The flow rate and pressure were strictly controlled and monitored constantly. Incase of each sample, the machine was turned on and the respective reading was carefully noted down. The procedure was followed until all samples were tested.

**ADVANTAGES OF THE INVENTION:**

1. The biodegradable filter material has better particle(s) trapping efficiency (as substantiated by the particle and elemental analysis)
2. The biodegradable filter material has enhanced particle filtration efficiency as compared to the cellulose acetate based filter material.
3. The biodegradable filter material has about 100% degradability in a short time.
4. The biodegradable filter material has fast self disintegration capacity.
5. The biodegradable filter material has accurate mechanical strength to be used as a cigarette filter.
6. The filtering material/ composition is economical.