The present invention provides a desiccant based honeycomb chemical filter comprising a matrix formed of a substrate having a desiccant generated in situ or deposited thereon, said desiccant being selected from the group consisting of metal silicates, silica gel, molecular sieves, activated alumina, activated carbon or hydrophobic zeolite, and any mixture thereof, said substrate being further impregnated with one or more of an oxidizing agent, or an alkali metal hydroxide, or strong or weak acid(s), or reducing agents.
DESICCANT BASED HONEYCOMB CHEMICAL FILTER AND METHOD OF MANUFACTURE THEREOF

FIELD OF THE INVENTION

The present invention relates to a chemical filter for removal of contaminants from air present in industrial or domestic environments. More particularly, the present invention relates to a desiccant based honeycomb chemical filter. The present invention also relates to a method for the manufacture of a desiccant based honeycomb chemical filter.

BACKGROUND TO THE INVENTION

As stated above, this invention relates to a desiccant based chemical filter, such as a honeycomb chemical filter, for the removal of contaminants from air present in industrial or domestic environments. The present invention also provides to a method for the manufacture of a desiccant based honeycomb chemical filter.

The contaminants to be removed are typically gaseous contaminants but can also comprise contaminants in the form of fine liquid droplets which are not easily removable by conventional means.

The term 'desiccant' as used herein is intended to cover both macroporous and microporous desiccant or desiccants. The desiccant or desiccants based honeycomb matrix used in the invention is impregnated with different impregnating materials. The invention also provides methods for removal of gaseous contaminants, pollutants and odors from industrial processes using the said chemical filter. This invention also relates to a process for manufacturing macroporous /microporous desiccant or desiccants based honeycomb matrix impregnated with different impregnants for removing gaseous contaminants contained in air or odour in the air.

It is well-recognized that it is difficult to manage airborne molecular contaminants due to the difficulty in counting and/or measuring such particles present in a given area. This difficulty is primarily due to the fact that gases are different from particles. The impact of contaminants on productivity as well as on human life, if not attended properly, can be severe. The presence of chemical contaminants even in part per billion (ppb) concentrations can alter the chemical properties of the surrounding environment and adversely affect human life and productivity. Traditional filters have been insufficient in protecting a given environment from gaseous contaminants, particularly inorganic pollutants such as ammonia, amine, ozone, oxides of sulfur, nitrogen oxides, sulphides, and other...
molecular contaminants that remain undetected by particle counters.

The problems in the art will be understood from the following discussion of some solutions available for removal of volatile organic components that form a significant part of airborne molecular contaminants, particularly in an industrial environment.

One problem in the art has been ensuring that while odour is controlled, there is no sacrifice on the humidity level that is desirable for human comfort and health. However, maintaining an appropriate level of humidity as well as odour control presents a particular problem, since existing dehumidifying devices either remove too much moisture from the air or are inefficient in removing odours.

Odours in industrial environments, and also in non-industrial environments such as passenger compartments of transportation vehicles are due to volatile organic compounds present in the circulating air. These organic compounds are formed in high levels but at relatively low concentrations from engine exhaust, solvents, gas turbines, cogeneration plants, petrochemical plants, and in many industrial processes where waste gases contain materials such as solvent vapors, inks, paints, etc. Volatile organic compounds comprise not only hydrocarbons, whether saturated, unsaturated, or aromatic, but also contain oxygenated materials such as alcohols, esters, ethers, and acids, nitrogen containing compounds (principally amines), sulfur containing materials (mercaptans and thioethers) and halogen-containing materials, especially chlorine-substituted hydrocarbons but also organic fluorides and bromides. The presence of such compounds in a gas stream is recognized as a health risk and/or cause the gas stream to have an unpleasant and undesirable odour.

A traditional method for solvent recovery from humid air comprised an adsorption system which typically was operated till the solvent concentration in the outlet gas stream reached a detectable preset breakthrough level. When this level was reached, the flow of gas to the adsorber was stopped. The adsorbent bed then contained solvent, other condensable organic contaminants, and some amount of water dependent on the inlet relative humidity of the solvent laden gas stream.

This method was subsequently modified by the introduction of a hot inert gas or steam, either saturated or superheated, which displaces the solvent from the adsorbent to produce a solvent/water mixture upon condensation. Typically two adsorber beds were used, where one was adsorbing while the other bed underwent regeneration.
Further developments in regenerating and recovering solvents from adsorbent beds involve the use of inert gases or air and low temperature condensation of the solvent from the regenerating gas/air. However, these techniques are not directly applicable in all given situations where required.

It is known that volatile organic compounds can be removed from air by adsorption such as thermal swing adsorption. Air streams needing treatment can be found in most chemical and manufacturing plants, especially those using solvents. At concentration levels from 500 to 15,000 ppm, recovery of the VOC from steam used to thermally regenerate activated carbon adsorbent is economically justified. Concentrations above 15,000 ppm are typically in the explosive range and require the use of a hot inert gas rather than air for regeneration. While, below about 500 ppm, recovery is not economically justifiable, environmental concerns often dictate adsorptive recovery followed by destruction. Activated carbon is the traditional adsorbent for these applications, and represents its second largest use.

U.S. Pat. No. 4,421,532 discloses a process for the recovery of VOCs from industrial waste gases by thermal swing adsorption including the use of hot inert gases circulating in a closed cycle to desorb the VOCs.

One device employed for drying air in confined areas such as a home, a ship or a building down to 0.001 lbs water per pound of air is discussed in U.S. Pat. No. 4,134,743. This document discloses a process and apparatus where the adsorbent body is a wheel of thin sheets or layers of fibrous material containing about 10 to 90% by weight of a freely divided molecular sieve material. The apparatus includes a means for passing air to be processed in one direction through the wheel and a means for passing a regenerative air stream countercurrent to the air to be processed. In addition a cooling stream is provided in a direction co-current with the air stream.

U.S. Pat. No. 4,887,438 discloses a desiccant assisted air conditioning system for delivering dehumidified refrigerated air to a conditioned space based on an adsorbent wheel. Meckler teaches the use of a desiccant wheel coated with silica gel, or a preferred hygroscopic salt, lithium chloride, to remove moisture from air. This citation teaches the use of waste heat from the refrigeration condenser to heat the reactivation air and employs a liquid refrigerant injection into the compressor to increase the pressure ratio in a positive displacement compressor to counter the problem of "thermal dumpback". Thermal dumpback is the
associated heat conducted from the desiccant wheel to the treated air which occurs following the exposure of the wheel to heated regeneration air. This associated heat adds to the overall cooling load on the refrigeration system.

U.S. Pat. No. 5,242,473 discloses a gas dehumidifying apparatus which exhibits improved dehumidification efficiency to provide a treated gas with a high dryness level employing two dehumidifier rotors, wherein the second rotor uses a synthetic zeolite. Gas to be dehumidified is passed first to a silica gel coated rotor and then to the zeolite coated rotor. The rotors are regenerated by supplying a stream of heated gas through the second rotor and then the first rotor so that the adsorbent in the first rotor is regenerated at a lower temperature than the zeolite in the second rotor. A portion of the treated gas is used to countercurrently cool the rotors following regeneration.

Of the techniques for removing volatile organic contaminants in low concentrations from a gas stream by adsorption, the most common is exemplified in U.S. Pat. No. 4,402,717. This document teaches an apparatus for removing moisture and odour from a gas stream comprising a cylindrical honeycomb structure made from corrugated paper, uniformly coated with an adsorbent and formed in the shape of a disk or wheel. The multiplicity of adsorbent-coated parallel flow passages formed by the corrugations in the paper serve as gas passage ways which are separated as a zone for the removal of water and odor causing components in the gas, and as a zone for the regeneration of the adsorbent. The zones for removal and regeneration are continuously shiftable as the wheel is rotated circumferentially about its centerline. Labyrinth seals separate the outer side of the rotary structure from the cylindrical wall of a sealed casing.

One of the problems associated with prior art attempts to use a honeycomb matrix is the adsorbent that is used. Hydrophilic adsorbents such as silica gel are typically chosen for dehumidification applications, but are poor adsorbents for volatile organic contaminant removal. One such process combination is discussed in U.S. Pat. No. 5,181,942. On the other hand, hydrophobic adsorbents, such as high silica zeolites are typically recommended for VOC removal applications, but are poor adsorbents for dehumidification application. Thus, applications for both dehumidification and VOC removal may typically require both types of adsorbents.

Attempts have been made whereby a single adsorbent can be employed for both operations. Furthermore, the adsorbent-coated paper and some adsorbent salts have a limited range of humidity and temperature within which they can
maintain structural integrity. This failure also limits the regeneration medium to dry, moderate temperature gases and air. Contact between the adsorbent and gas stream and hence adsorbent capacity for VOCs is limited to very thin layers of adsorbent on the surface of the fiber. This feature also limits the ultimate life of adsorbent wheel, resulting in frequent wheel replacement.

It is recognized that chemical filtration strategies are ideal for use in control of airborne molecular contamination. However, the use of honeycomb matrix based desiccants has not generally been considered due to the difficulty in ensuring both humidity control and chemisorption.

Filters are generally devices such as a membrane or a layer that is designed to block or contain objects or substances while letting others through. Filtration media are generally of three types - mechanical, biological and chemical. Chemical filters are preferred for removal of gaseous contaminants in a given environment since they provide air purification by both adsorbing and absorbing odours by adsorbents/impregnated adsorbents such as potassium permanganate by controlled oxidizing action.

Gaseous contaminants either could be inorganic or organic. In order to remove gaseous contaminants, selection of adsorbents to adsorb gas or reactants for adsorbing gas/impregnates is very important for the chemical filter media. As discussed above, a single chemical filter media may not adequately control multiple contaminants or classes of AMC.

**OBJECTS OF THE INVENTION**

One object of the present invention is to provide a desiccant based honeycomb chemical filter where the desiccant is generated in situ, and impregnated with oxidizing chemicals, acidic or alkaline chemicals, or reducing chemicals to remove/contain gaseous contaminants, where the loading percentage of the impregnants is significantly higher than thought possible in the art.

The primary object of the present invention is to increase the capability of removing gaseous contaminants whether, acidic, alkaline, or organic and increase chemical filter life by the use of higher amounts of impregnants.

A further object of this invention is to produce a desiccant based honeycomb matrix with or without active carbon or hydrophobic zeolite for impregnation with different impregnants/reactants to remove gaseous contaminants.

Another object of the invention is to provide a desiccant based honeycomb
matrix where pressure loss is minimized. The desiccant may or may not contain active carbon or hydrophobic zeolite and may or may not be impregnated with impregnants for enhanced removal of different contaminants.

A further object of the invention is to provide a desiccant based honeycomb matrix chemical filter that provides an energy saving, and may be with or without active carbon or hydrophobic zeolite, and may be impregnated with different impregnants or un-impregnated, while also enabling enhanced odour control.

SUMMARY OF THE INVENTION

Accordingly, to overcome the problems encountered in the prior art, the present invention provides a honeycomb matrix comprising a desiccant prepared in-situ, for use as a chemical filter, as described herein below.

The present invention provides a desiccant based honeycomb chemical filter comprising a matrix formed of a substrate having a desiccant generated in situ or deposited thereon, said desiccant being selected from the group consisting of metal silicates, silica gel, molecular sieves, activated alumina, activated carbon or hydrophobic zeolite, and any mixture thereof, said substrate being further impregnated with one or more of an oxidizing agent, or an alkali metal hydroxide, or strong or weak acid(s), or reducing agents.

The substrate comprises an inorganic substance or an organic substance or a mixture thereof.

In a further embodiment, the substance is in the form of a fiber or a pulp.

In a further embodiment, the inorganic fiber is selected from the group consisting of glass fiber, Kraft paper, ceramic paper and any combination thereof, and is preferably a glass fiber.

In another embodiment, the desiccant is selected from a metal silicate and silica gel or combination of metal silicate and activated carbon and/or a hydrophobic desiccant such as zeolites selected from HISIV and ZSM-5.

The metal silicate is selected from potassium silicate and sodium silicate, preferably where the silicate is a neutral grade sodium silicate.

In a preferred embodiment, in the sodium silicate, the ratio of SiO$_2$ to Na$_2$O is in the range of 1:2.0 to 1:4.0, preferably in the range of 1:3.

In another embodiment of the invention, the impregnated material is selected from the group consisting of potassium permanganate, sodium permanganate, potassium hydroxide, sodium hydroxide, phosphoric acid and sodium thiosulphate.
In a further embodiment of the invention, the loading of the impregnated material in the matrix is in the range of 4% to 40%, preferably about 20%.

In a further embodiment of the invention, the binder is preferably absent.

In a further embodiment of the invention, the basic weight of the substrate is in the range of 50 to 150 gsm, preferably in the range of 80 to 120 gsm.

The present invention also provides a method for manufacture of a desiccant based honeycomb chemical filter, comprising the steps of

(i) dehydrating of macroporous or microporous desiccant/desiccants based honeycomb matrix at temperature 100°C or more;

(ii) impregnating the dehydrated desiccant based honeycomb matrix with suitable impregnants and then drying the said matrix with retaining the moisture content in the range of 15-30%.

The method when used in a situation comprising in situ generation for desiccant comprises the following steps

(i) treating a substrate material with a desiccant forming material or a desiccant selected from the group consisting of metal silicates, silica gel, molecular sieves (hydrophillic or hydrophobic), activated alumina, activated carbon, and any mixture thereof, wherein the desiccant forming material is in the form of a solution with a concentration in the range of 15 - 40%;

(ii) drying the treated material to reduce moisture content thereof to a range of 15-40%;

(iii) soaking the substrate matrix in water soluble metal salt or salts or strong or weak acids to form a hydrogel and obtain a gel matrix;

(iv) washing the gel matrix obtained in step iii to remove excess reactants and unwanted by-products;

(v) drying the gel matrix under controlled conditions of temperature to convert the hydrogel to an aerogel matrix;

(vi) treated the aerogel matrix with one or more of an oxidizing agent, or an alkali metal hydroxide, or strong or weak acid(s), or reducing agents;

(vii) drying the treated aerogel matrix of step (vi) above at a temperature in the range of 50 - 140°C to obtain said chemical filter.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a solution for the problems in the art of ensuring maximum efficiency in adsorption of chemical contaminants using a honeycomb based chemical filter using a desiccant. Honeycomb matrixes can be
formed from a variety of substrates such as plastic sheet, metal/aluminum foil, organic and/or inorganic fiber substrates which are "paper" like, and which may be quite porous. The deposition or loading of a desiccant material on the substrate is a function, generally, of the substrate of choice, the amount of desiccant that is to be deposited or loaded, and the temperature at which the air/matrix is intended for use. Among known techniques for deposition or loading the desiccant on the substrate to prepare the matrix are coating, impregnation and in situ synthesis.

Coating and impregnation are chosen where the desiccant powder is produced in bulk and are intended for industrial applications other than HVAC air treatment. In situ synthesis is generally chosen in case of desiccant material such as silica gels and metal silicates.

In the desiccant based honeycomb chemical filter of the invention, the desiccant is generated in situ, and impregnated with oxidizing chemicals, acidic or alkaline chemicals to remove/contain gaseous contaminants, and the loading percentage of impregnants is significantly higher than thought possible in the art.

In a surprising finding, this has resulted in an increase in the capability of removing gaseous contaminants whether, acidic, alkaline, or organic and increase chemical filter life by the use of higher amounts of impregnants.

The desiccant based honeycomb matrix can be made with or without active carbon or hydrophobic zeolite, for impregnation with different impregnants/reactants to remove gaseous contaminants. Another significant feature of the invention is that pressure loss is minimized in the desiccant based honeycomb matrix. The desiccant may or may not contain active carbon or hydrophobic zeolite and may or may not be impregnated with impregnants for enhanced removal of different contaminants.

The desiccant based honeycomb matrix chemical filter of the invention also provides an energy saving, and may be with or without active carbon, and may be impregnated with different impregnants or un-impregnated, while also enabling enhanced odour control.

As stated previously, air to be treated, particularly outside air, generally contains several gaseous contaminants, e.g. VOCs, odour, and the like. It is desirable to remove these through the desiccant matrix. In air to be treated, particularly when the air is pre-cooled to near saturation, such gaseous contaminants are sometime water soluble and together are condensed in the macro porous desiccant mainly through capillary adsorption. The prior art contains
some references to the use of microporous desiccants. However, these desiccants generally have limited capillary adsorption and therefore limited adsorption of gaseous contaminants.

The prior art also shows a limited loading capability for both the desiccant and any impregnants due to the fact that generally the desiccant is coated or impregnated on the substrate. Thus, adsorption in these cases is a surface phenomena. This necessarily results in the use of less dense paper, typically in the range of 20 to 80 gsm thickness. The requirement of coating of the substrate is generally a limiting factor in the substrate choice, and it was considered unfeasible to increase the density of the substrate without compromising on the surface area required for adsorption.

The problems encountered in the prior art are solved in the present invention by providing a honeycomb matrix comprising a desiccant prepared in-situ, for use as a chemical filter, as described herein below.

The invention is achieved by a macroporous or microporous desiccant based honeycomb matrix. The substrate is made from various inorganic or organic substances or combinations thereof. The substance can be a fiber or pulp. The inorganic fiber is selected from glass fibre, Kraft paper, ceramic paper, etc. and is preferably glass fiber. The desiccants that can be synthesized in situ preferably comprise metal silicate/ silica gel which provide a high adsorption capacity.

The invention will now be described with reference to an exemplary and non-limiting embodiment where glass fiber is used as the substrate. The process comprises laminating water glass treated/ impregnated glass fiber in the shape of either a honeycomb or as a block. The matrix is treated with either water soluble salts (whether divalent or trivalent) or any acid (whether organic or inorganic) to convert the water glass silicate into the active desiccant material with a pore diameter in the range of 10 - 70 Å, a pore volume of 0.10-0.80gm/cc and surface area of 300-700 m².

The desiccant based honeycomb matrix is generally a flat and/or corrugated sheet of the fiber glass substrate where the active desiccant has been synthesized into the pores. The fiber glass substrate used in the present invention is a highly porous material having the fiber diameter of 4-15 micron, fiber length 6-10mm, thickness in the range of 0.10-0.75 mm. The binder content of the fiber glass substrate used in the process is not greater than 8% and the preferred binder used in making substrate is polyvinyl alcohol or combination with acrylic. It is advisable
to use a low content of organic binder in the substrate so that the percentage of organic content in the desiccant based honeycomb matrix is also reduced.

Generally, the need for adding an external binder is avoided where the water soluble silicate desiccant is generated in situ. The silicate material itself carries out the function of a desiccant once treated with suitable reactant/s. However, when the desiccant is to be coated on the matrix, it is advisable to add an additional binder material. Water soluble silicates can be used for this purpose. In the alternative commercially available binders may also be used where the desiccant is coated on the matrix.

The basic weight of the substrate used in the process is 50-150 gsm, preferably between 80 to 120 gsm. The fiber used in the making of such substrates can be of electrical grade fiber. The flat sheet that is to be corrugated is first passed through the desired concentration of water glass solution (from 15-40%, preferably 30%), with or without active carbon material or hydrophobic zeolite. The sheets are dried in a drying chamber, preferably using infra-red heating to ensure that the moisture content is in the desired range of 15-40%, preferably 20%. While, other types of water soluble silicates such as potassium silicate can also be used, sodium silicate is preferable due to its cost efficiency, high solubility of its by-products, higher bonding strength and easy availability.

It is preferable to use neutral grade water soluble silicate, preferably sodium silicate to impregnate the fiber glass substrate. It is observed that the presence of high alkali content in alkaline grade as compared to neutral grade silicate necessitates use of a large quantity of reactants. Use of alkaline grade sodium silicate also results in a higher rate of gelation than neutral grade sodium silicate.

The gelling rate adversely affects the characteristics of the desiccants.

The process of in situ synthesis of the desiccant material on the substrate can be accomplished simultaneously with the deposition of active carbon and/or hydrophobic zeolite material such as HISIV or ZSM05. The use of active carbon and/or hydrophobic zeolitic material enhances the level of odour removal and also provides a significant enhancement in structural strength of the substrate itself. Active carbon (and/or hydrophobic zeolitic material) in the form of powder is mixed with water soluble silicate or any suitable binder-inorganic or organic prior to the formation of the desiccant in situ. This enables capture/encapsulation of active carbon particles or hydrophobic zeolite within the desiccant, thereby enhancing odour control, VOC adsorption, and the structural strength of the matrix itself.
The corrugated sheet is produced by passing the silicate (or silicate and active carbon or hydrophobic zeolite) impregnated flat sheet through a toothed roller. Moisture content of the silicate or silicate and active carbon or hydrophobic zeolite impregnated sheets, grade of water glass, corrugated rolls, the control of clearances on the adhesive rolls and the curing heat of the adhesive are important in making a single facer unit. The corrugated sheet and the flat sheet are pressed together using additional rolls of the same material that is used for corrugation. It is preferable to avoid the use of adhesive while making single facer, even with the same water glass used for impregnation, in order to avoid powdering or higher pressure drops after synthesis. This is achieved by keeping higher moisture content in the flat sheet.

The single facer that is a combination of flat and corrugated sheets can be converted into either a block or a cylindrical shape. Neutral grade sodium silicate is preferably applied to ensure adhesion of the single facers sequentially prior to stacking or winding. Application of SiO$_2$: Na$_2$O in the range of 1:2.0 To 1:4.0 preferably 1:3.0 before winding or stacking of the single facer gives additional strength to the honeycomb matrix. The honeycomb matrix that is produced is then dried to enhance the adhesion of the sheets. If the ratio of SiO$_2$:Na$_2$O is <2, the bonding strength is weaker and if ratio of SiO$_2$: Na$_2$O is above 4.0 the adhesive strength is less.

Another advantage of the use of neutral grade silicate is the rate of drying. Moisture loss is affected by ratio of SiO$_2$: Na$_2$O because water retention is a direct function of alkalinity. Higher ratio of silicate retains less amount of water. Drying of more alkaline silicates of the ratio of SiO$_2$: Na$_2$O in the range of 2.0-2.6 is slower than silicate having SiO$_2$: Na$_2$O in the range of 3.0-3.3.

The honeycomb matrix produced above either in block or cylindrical form is then soaked in water soluble metal salt/salts or strong or weak acid/acids-inorganic or organic in different proportions and other forms of solution to produce silicate hydrogel. The reaction between water glass silicates with metal salts to form insoluble metal silicate hydrogel is given in the table below:

\[
\begin{align*}
    Na_2SiO_3 + Al_2(SO_4)_3 & \to Al_2(SiO_3)_3 + Na_2SO_4 \\
    Na_2SiO_3 + MgSO_4 & \to MgSiO_3 + Na_2SO_4 \\
    Na_2SiO_3 + MgCl_2 & \to MgSiO_3 + NaCl \\
    Na_2SiO_3 + AlCl_3 & \to Al_2(SiO_3)_3 + NaCl \\
    Na_2SiO_3 + HCl & \to H_2SiO_3 + NaCl
\end{align*}
\]
Washing of the gel matrix is essential to remove by-products and excess reactants formed during the synthesis of active materials. The acidity of gel matrix due to presence of excess reactants deteriorates the construction material used in the system. The hydrogel is washed at controlled pH. The gelling pH of the matrix or the concentration of reactant/reactants, temperature, reaction time used in the process changes the active material characteristics such as pore size, porosity, pore volume and surface area.

The element is further dried under specified conditions to convert the hydrogel into the aerogel. It is observed that the type of silicate, types of salts its pH, concentration, temperature and time during which the gel is aged or otherwise treated, greatly affects the gel characteristics such as pore diameter, pore volume, surface area, adsorption capacity etc. The other important factors which affects gel characteristics are salt contents and surface tension of the liquid medium as it is being evaporated from the pores of the gel.

The manufacture of the desiccant based honeycomb matrix chemical filter of the invention comprises laminating water glass treated/impregnated glass fiber substrate (with or without carbon or hydrophobic zeolite) in the form/shape of cylinder or block, treating this element with one or more water soluble divalent/trivalent salt(s) or organic or inorganic acid(s) - strong or weak, to convert the water glass into active material, and then impregnating the active material with different impregnants.

The produced aerogel is immersed/impregnated with a solution of oxidizing agent(s), alkaline solution and weak acid solution or any reactants suitable for removing gaseous contaminants of different concentration at different temperature for different soak time. When the honeycomb is sufficiently impregnated with solution, the excess impregnated is drained off and kept in another vessel after readjusting the concentration.

Time required for full impregnation varies as a function of the structure of the adsorbent, temperature and other factors. The honeycomb material is then placed in an oven and heated till free moisture and water evaporate or are driven out of the mixture leaving impregnating material within the pores of honeycomb silicate aerogel material and/or silicate hydrogel and activated carbon or hydrophobic zeolite. Preferably, the drying temperature of the impregnated honeycomb material is in the range of 50-140°C. The exposure time in the heating is varied with quality and quantity of materials, heating efficiency and other factors.
After preparation, the honeycomb matrix impregnated with impregnants is preserved until ready for use. The impregnation steps are described below:

a. dehydration of macroporous or microporous desiccant/desiccants based honeycomb matrix

b. impregnation with impregnant/impregnants

c. drying the impregnated macroporous or microporous desiccant/desiccants based honeycomb matrix.

In our experiments the preferred impregnants used are potassium and sodium permanganate, sodium or potassium hydroxide, weak acids such as phosphoric acid, and a reducing agent such as sodium thiosulphate. The loading of impregnants depends on various factors such as types of desiccants, concentration of impregnants, soak time, temperature, number of dips, etc.

**Impregnation with Potassium Permanganate and Sodium Permanganate**

Macroporous or microporous desiccant (with or without active carbon) material supported honeycomb matrix is impregnated with potassium/sodium permanganate solution in a range of 5 to 40%, preferably 15% concentration in case of potassium permanganate and 30% in case of sodium permanganate, at 10°C to 90°C, most preferably at 70°C for 5 to 60 minutes, most preferably for 15 minutes, to get the maximum impregnant loading without affecting the mechanical strength of the matrix and water/CTC adsorption capacity. Table 1 gives details of factors that affect percentage of loading of impregnant (KMnO₄ vs. NaMnO₄) and adsorption capacity of impregnated honeycomb desiccant based matrix.

### TABLE 1

**A. CONCENTRATION OF IMPREGNANTS**

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<thead>
<tr>
<th>Parameters</th>
<th>KMnO₄</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th>NaMnO₄</th>
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<tr>
<td>1.% Loading</td>
<td>1.9</td>
<td>2.7</td>
<td>4.8</td>
<td>4.7</td>
<td>-</td>
<td>-</td>
<td>4.8</td>
<td>9.6</td>
<td>12.7</td>
<td>15.9</td>
<td>24.0</td>
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<td>2. Water adsorption/CTC</td>
<td>35</td>
<td>32</td>
<td>29</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<td>48</td>
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**B. SOAK TIME OF KMnO₄ & NaMnO₄**

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<th></th>
<th></th>
<th>NaMnO₄</th>
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<td>2. Water adsorption/CTC</td>
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C. TEMPERATURE OF KMnO₄ & NaMnO₄

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<th>Parameters</th>
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<th>NaMnO₄</th>
</tr>
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<td>9.5</td>
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<tr>
<td>2. Water adsorption/CTC</td>
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<td>27</td>
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D. KMnO₄ & NaMnO₄ (Macro vs Micro)

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<td>NaMnO₄</td>
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<tr>
<td>1. % Loading</td>
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Concentrations, soak time, solution temperature, successive dipping and surface property of active material play an important role in achieving the twin objective loading and adsorption. The method of Impregnation of alkali preferably sodium or potassium hydroxide, or of acid preferably phosphoric acid and a reducing agent such as sodium thiosulphate with micro or macroporous and or with hybrid desiccant (insoluble metal silicate and activated carbon acid) is described hereinafter. A slurry of activated carbon is prepared in water glass solution and honeycomb matrix is wound in the form of a block or a cylinder. The honeycomb matrix supported with water glass and activated carbon is treated with a salt solution as described above. The desiccant material, whether macroporous or microporous (and with or without active carbon), supported honeycomb matrix is impregnated with different concentrations of impregnants.

**Impregnation with Potassium hydroxide**

Macroporous or microporous desiccant (with or without active carbon and/or hydrophobic zeolitic material) material supported honeycomb matrix is impregnated with potassium hydroxide solution in the range of 2 to 15%, most preferably 6%, at 10°C to 50°C, most preferably at 30°C for 5 to 60 minutes, most preferably for 15 minutes to get the maximum impregnant loading without affecting the mechanical strength of the matrix and water/CTC adsorption capacity. Table 2 gives details of factors affecting the % of loading of impregnant (KOH) and adsorption capacity of impregnated honeycomb desiccant based matrix.

**TABLE 2**

A. CONCENTRATION OF IMPREGNANTS - macroporous vs. macroporous with activated carbon (hybrid desiccants)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Macro</th>
<th>Hybrid desiccants</th>
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</thead>
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<tr>
<td></td>
<td>C1</td>
<td>C2</td>
</tr>
<tr>
<td>1. % Loading</td>
<td>5.8</td>
<td>11.0</td>
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<td>46</td>
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B. SOAK TIME OF KOH

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</thead>
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<tr>
<td>1.% Loading</td>
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<td>10.1</td>
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<td>2. Water adsorption/CTC</td>
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<td>-</td>
</tr>
</tbody>
</table>

C. TEMPERATURE OF KOH

<table>
<thead>
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<th>Parameters</th>
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<th>Hybrid desiccants</th>
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</thead>
<tbody>
<tr>
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<td>T2</td>
</tr>
<tr>
<td>1.% Loading</td>
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<td>8.7</td>
</tr>
</tbody>
</table>

5 Impregnation with Phosphoric Acid

Macroporous or microporous (with or without active carbon) material supported honeycomb matrix is impregnated with phosphoric acid solution in the range of 2 to 15%, most preferably 8%, at 10°C to 50°C, most preferably at 30°C for 5 to 60 minutes, most preferably for 15 minutes to get the maximum impregnant loading without affecting mechanical strength of the matrix and water/CTC adsorption capacity. Table 3 gives details of factors affecting percentage loading of impregnant (Phosphoric acid) and adsorption capacity of impregnated honeycomb desiccant based matrix.

TABLE 3

A. CONCENTRATION OF IMPREGNANTS - macroporous vs. macroporous and activated carbon (Hybrid desiccants)

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<th>Parameters</th>
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</tr>
</thead>
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<td>C2</td>
</tr>
<tr>
<td>1.% Loading</td>
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<tr>
<td>2. Water adsorption/CTC</td>
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<td>-</td>
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</table>

B. SOAK TIME OF PHOSPHORIC ACID

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<th>Hybrid desiccants</th>
</tr>
</thead>
<tbody>
<tr>
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<td>ST2</td>
</tr>
<tr>
<td>1.% Loading</td>
<td>8.3</td>
<td>7.8</td>
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<tr>
<td>2. Water adsorption/CTC</td>
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<td>-</td>
</tr>
</tbody>
</table>

C. TEMPERATURE OF PHOSPHORIC ACID

<table>
<thead>
<tr>
<th>Parameters</th>
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<th>Hybrid desiccants</th>
</tr>
</thead>
<tbody>
<tr>
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<td>T2</td>
</tr>
<tr>
<td>1.% Loading</td>
<td>8.3</td>
<td>6.7</td>
</tr>
<tr>
<td>2. Water adsorption/CTC</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Impregnation with Sodium Thiosulphate

Macroporous or microporous (with or without active carbon or hydrophobic zeolite) material supported honeycomb matrix is impregnated with sodium thiosulphate solution in the range of 5 to 45%, preferably 15%, at 10°C to 50°C, most preferably at 30°C for 5 to 60 minutes, preferably for 15 minutes to get maximum impregnant loading without affecting the mechanical strength of the matrix and water/CTC adsorption capacity. Table 4 gives details of factors affecting%

% of loading of impregnant (Sodium thiosulphate) and adsorption capacity of impregnated honeycomb desiccant based matrix:

TABLE NO.:4

A. CONCENTRATION OF IMPREGNANTS - macroporous vs. macroporous and activated carbon (Hybrid desiccants)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Macro</th>
<th>Hybrid desiccants</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>C1</td>
<td>C2</td>
</tr>
<tr>
<td>1. % Loading</td>
<td>7.4</td>
<td>10.1</td>
</tr>
</tbody>
</table>

B. SOAK TIME OF SODIUM THIOSULPHATE SOLN.

<table>
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<tr>
<th>Parameters</th>
<th>Macro</th>
<th>Hybrid desiccants</th>
</tr>
</thead>
<tbody>
<tr>
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<td>ST1</td>
<td>ST2</td>
</tr>
<tr>
<td>1. % Loading</td>
<td>18.9</td>
<td>16.6</td>
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</tbody>
</table>

C. TEMPERATURE OF SODIUM THIOSULPHATE SOLN.

<table>
<thead>
<tr>
<th>Parameters</th>
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<th>Hybrid desiccants</th>
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</thead>
<tbody>
<tr>
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<td>T1</td>
<td>T2</td>
</tr>
<tr>
<td>1. % Loading</td>
<td>15.4</td>
<td>9.7</td>
</tr>
</tbody>
</table>

The present invention provides several significant advantages over prior art methods. Some of these are summarized below.

1. While macroporous material is generally preferred, potassium permanganate can also be impregnated to a large loading percentage on microporous material.
2. There is an enhancement in the extent of loading due to several factors, some of which are given below:

(a) The GSM of the substrate is significantly higher than that used in the prior art. The GSM generally ranges from 50 to 150 gsm, preferably between 80 to 120 gsm. This enables higher desiccant loading on the substrate leading to a larger impregnate loading of the impregnate;

(b) The formation in situ of the desiccant is done such that it is pH neutral and is generally minimally affected by the chemical nature of the gas, whether acidic or basic-Binders are preferably avoided and not deemed necessary for the formation of the desiccant preparation and the desiccant materials thus obtained are all active materials. This also results in the final increase in surface density of the substrate of from 400 to 750 gsm.

(c) The extent of loading is enhanced also due to a combination of different factors such as soak time, temperature and concentration. For example, permanganates, usually Na permanganate achieves a loading of greater than 30%. Additionally the necessary moisture content is maintained, higher adsorption capacity of the desiccant since it is synthesized in situ, additional advantages include the fact that the desiccant can be bacteriostatic, and non-flammable.

(d) Desiccant can be combined with active carbon or hydrophobic zeolite, preferably by mixing the carbon powder or a hydrophobic desiccant such as zeolitic material selected from HISIV or ZSM-05 with a suitable binder, preferably inorganic prior to formation of the desiccant in situ. This enables capture/encapsulation of the carbon particles within the desiccant. This enables an enhancement in odour control, VOC adsorption, and in the structural strength of the matrix itself.

3. The invention enables if required, a sacrifice of the surface area of the matrix to enable multiple fluid geometries, and allows lower pressure drops without any significant material sacrifice of the performance.

4. Up to 100 % efficiency is achieved even despite reduced contact time when run at higher face velocities of 400 to 600 fpm [2 to 3 m/sec].
We claim:
1. A desiccant based honeycomb chemical filter comprising a matrix formed of a substrate having a desiccant generated in situ or deposited thereon, said desiccant being selected from the group consisting of metal silicates, silica gel, molecular sieves, activated alumina, activated carbon, and any mixture thereof, said substrate being further impregnated with one or more of an oxidizing agent, or an alkali metal hydroxide, or strong or weak acid(s), or reducing agents.
2. A chemical filter as claimed in claim 1 wherein the substrate comprises an inorganic substance or an organic substance or a mixture thereof.
3. A chemical filter as claimed in claim 2 wherein the substance is in the form of a fiber or a pulp.
4. A chemical filter as claimed in claim 3 wherein the inorganic fiber is selected from the group consisting of glass fiber, Kraft paper, ceramic paper and any combination thereof.
5. A chemical filter as claimed in claim 4 wherein the inorganic fiber is glass fiber.
6. A chemical filter as claimed in claim 1 wherein the desiccant is selected from a metal silicate and silica gel or combination of metal silicate and activated carbon and/or a hydrophobic desiccant such as zeolites selected from HISIV and ZSM-5.
7. A chemical filter as claimed in claim 6 wherein the metal silicate is selected from potassium silicate and sodium silicate.
8. A chemical filter as claimed in claim 7 wherein the silicate is a neutral grade sodium silicate.
9. A chemical filter as claimed in claim 8 wherein in the sodium silicate, the ratio of SiO$_2$ to Na$_2$O is in the range of 1:2.0 to 1:4.0, preferably in the range of 1:3.
10. A chemical filter as claimed in claim 1 wherein the impregnated material is selected from the group consisting of potassium permanganate, sodium permanganate, potassium hydroxide, sodium hydroxide, phosphoric acid and sodium thiosulphate.
11. A chemical filter as claimed in claim 10 wherein the loading of the impregnated material in the matrix is in the range of 4% to 40%, preferably about 20%.
12. A chemical filter as claimed in any preceding claim wherein binder is
preferably absent.

13. A chemical filter as claimed in any preceding claim wherein activated carbon
and/or hydrophobic desiccant material such as HIS IV or ZSM-5 is added
along with desiccant being generated in situ.

14. A chemical filter as claimed in any preceding claim wherein the basic weight
of the substrate is in the range of 50 to 150 gsm.

15. A chemical filter as claimed in any preceding claim wherein the basic weight
of the substrate is in the range of 80 to 120 gsm.

16. A method for manufacture of a desiccant based honeycomb chemical filter,
comprising the steps of

(i) dehydrating of macroporous or microporous desiccant/desiccants
based honecomb matrix at temperature 100°C or more;
(ii) impregnating the dehydrated desiccant based honeycomb matrix
with suitable impregnants and then drying the said matrix with retaining the
moisture content in the range of 15-30%.

17. A method as claimed in claim 16 comprising

(i) treating a substrate material with a desiccant forming material or a
desiccant selected from the group consisting of metal silicates, silica gel,
molecular sieves, activated alumina, activated carbon, and any mixture
thereof, wherein the desiccant forming material is in the form of a solution
with a concentration in the range of 15 - 40 %;
(ii) drying the treated material to reduce moisture content thereof to a
range of 15-40%;
(iii) soaking the substrate matrix in water soluble metal salt or salts or
strong or weak acids to form a hydrogel and obtain a gel matrix;
(iv) washing the gel matrix obtained in step iii to remove excess
reactants and unwanted by-products;
(v) drying the gel matrix under controlled conditions of temperature and
pH to convert the hydrogel to an aerogel matrix;
(vi) treated the aerogel matrix with one or more of of an oxidizing agent,
or an alkali metal hydroxide, or strong or weak acid(s), or reducing agents;
(vii) drying the treated aerogel matrix of step vi. above at a temperature
in the range of 50 - 140°C to obtain said chemical filter.

18. A method as claimed in claim 16 wherein the substrate comprises an
inorganic substance or an organic substance or a mixture thereof.
19. A method as claimed in claim 16 wherein the substance is in the form of a fiber or a pulp.

20. A method as claimed in claim 19 wherein the inorganic fiber is selected from the group consisting of glass fiber, Kraft paper, ceramic paper and any combination thereof.

21. A method as claimed in claim 20 wherein the inorganic fiber is glass fiber.

22. A method as claimed in claim 18 wherein the desiccant is selected from a metal silicate and silica gel.

23. A method as claimed in claim 22 wherein the metal silicate is a water soluble silicate selected from potassium silicate and sodium silicate.

24. A method as claimed in claim 23 wherein the silicate is a neutral grade sodium silicate.

25. A method as claimed in claim 24 wherein in the sodium silicate, the ratio of SiO₂ to Na₂O is in the range of 1:2.0 to 1:4.0 preferably 1:3.

26. A method as claimed in claim 16 wherein the impregnated material is selected from the group consisting of potassium permanganate, sodium permanganate, potassium hydroxide, sodium hydroxide, phosphoric acid and sodium thiosulphate.

27. A method as claimed in claim 26 wherein the loading of the impregnated material in the substrate is in the range of 4% to 40%, preferably 30%.

28. A method as claimed in any of claims 16 to 27 wherein binder is preferably absent.

29. A method as claimed in any of claims 16 to 28 wherein activated carbon or hydrophobic zeolite is added along with desiccant being generated in situ.

30. A method as claimed in claim 16 wherein the moisture content of the desiccant containing matrix is preferably 20%.

31. A method as claimed in claim 17 wherein step vii is carried out at a temperature in the range of 10°C to 90°C for a period of 5 to 60 minutes.

32. A method as claimed in claim 31 wherein step vii is carried out at a temperature of 70°C and for a period of preferably 15 minutes.

33. A chemical filter as claimed in any of claims 1 to 15 for use in the removal of contaminants from the air supply stream, said contaminants comprising gases, odour elements or volatile organic compounds.

34. Desiccant wheel exchanger comprising a chemical filter as claimed in any of claims 1 to 15.
**INTERNATIONAL SEARCH REPORT**

**International application No**

PCT/IB2013/000062

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**A. CLASSIFICATION OF SUBJECT MATTER**

INV. B01J20/32 B01J20/28 B01D53/28 B01D53/02 F24F3/14

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**B. FIELDS SEARCHED**

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

**Minimum documentation searched (classification system followed by classification symbols)**

B01D B01J F24F

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

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

EPO-Internal, WPI Data

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**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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<td>WO 2011/045814 A2 (DESICCANT ROTORS INTERNAT PVT LTD [IN]) 21 April 2011 (2011-04-21)</td>
<td>1-8,10, 11, 13-24, 26,27, 29,33,34</td>
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<td>X</td>
<td>JP 2003 103165 A (NICHIAS CORP) 8 April 2003 (2003-04-08) abstract</td>
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**Further documents are listed in the continuation of Box C.**

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* Special categories of cited documents:

- "X" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier application or patent but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which establishes the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
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- "A" document member of the same patent family

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**Date of the actual completion of the international search**

11 April 2013

**Date of mailing of the international search report**

19/04/2013

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**Name and mailing address of the ISA/**

European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016

**Authorized officer**

Hilgenga, Klaas
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<td></td>
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JP 2003103165 A 08-04-2003 NONE

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International application No
PCT/IB2013/00062

Form PCT/ISA/210 (patent family annex) (April 2005)