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(54) Title: LIQUID FILTRATION SYSTEMS

(57) Abstract: Provided are plasma-treated binders for use in filtration media comprising sorbent media. Plasma treatment can use silane, oxygen, or both. Plasma-treated binders can be charge-modified with the addition of an anti-microbial agent. The filtration media can be used to make matrixes and systems. Methods of making and using the same are also provided.



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LIQUID FILTRATION SYSTEMS

TECHNICAL FIELD

The present disclosure relates to liquid filtration systems and filter matrixes and media, wherein the filter media contains, for example, plasma-treated polymeric binders.

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BACKGROUND

Numerous types of home water filtration systems are commercially available.

Traditionally, beds of loose carbon particles were used for removing metals and/or organic materials from water. Composite blocks can be made from combinations of sorptive materials, such as adsorbent activated carbon, and polymeric binders, such as ultra high molecular weight polyethylene (UHMW PE), that have been sintered together under conditions of heat and pressure and are useful in water filter technology. Carbon block technology, for example, provides comparable functionality to loose bed carbon particles without the particle shedding or taking up too much space. With carbon block technology, pressure drop across the block can increase as a result of increasing quantities of adsorptive materials. Moreover, exposure of carbon blocks to heat and pressure can limit the types of adsorptive materials available for use in the blocks. For example, carbon block technology is generally precluded from using adsorptive media that is sensitive to thermal degradation, such as ion exchange resins.

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A limitation of current technology is that very small particles are required to make a filter that will remove cryptosporidium cysts and other small particulates. Use of very small activated carbon particles requires finer binder particles and more binder. Finer carbon is more difficult to handle and it is more difficult to produce to a controlled particle size.

Further, the use of very fine adsorbent and binder particles yields a filter with high pressure drop and lower water flow.

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A related problem is that blocks with very fine media particles require relatively high amounts of UHMW PE binder. High binder contents are needed to give a block with good mechanical strength and to minimize particle shedding. Typical binder levels for

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commercial cyst-rated water filter blocks range 25% to 55% by mass. Since polyethylene does not have any adsorptive function, use of higher binder levels limits the capacity of blocks for removal of water contaminants such as lead, VOCs, and chlorine.

- 5 Another difficulty in the current processes for making composite blocks is that carbon media- polymeric binder blends exhibit poor flow properties. Because of this, molds for making carbon blocks are generally not optimally filled. If molds are not filled to maximum density, more binder is required to hold media particles together. Also, differences in filling and fill density cause variations in filter flow properties and
- 10 variations in performance. A common problem is that carbon-polymer blends tend to de-aerate and agglomerate between the mixing step and mold filling step. If the carbon-polymer blend is agglomerated when molds are filled, then the mold does not fill to an optimum density and the filling is variable.
- 15 Silica as a fine particulate has been used previously as a powder flow aid. For example, small amounts of fumed silica are added to powder blends in the pharmaceutical industry in tablet forming processes. The use of fumed silica flow aid in making water filter blocks made from diatomaceous earth can result in blocks that are reduced in strength.
- 20 Still another limitation of current technology is the mechanical strength of blocks. Since the demand for water filters is trending toward smaller size and higher flow, there is a need for thinner walls and smaller outer diameters. Such blocks have low beam strength and this can lead to defects during production process or during handling and shipping.
- 25 Yet another limitation of current technology is the ability to make carbon blocks using granular media (larger than 100 mesh). Current technology uses blends of 325 mesh carbon particles (<43 micron) and 80x325 mesh particles (43-175 micron). Typical mean particle sizes are less than 90 micron. There is a need for low pressure drop blocks made from larger particles. It can be difficult, however, to make blocks with larger sizes of
- 30 granular carbon, e.g., 50x200 mesh (75-295 micron).

A potential disadvantage of current technology is that the UHMW PE used as a binder is hydrophobic. This makes a filter difficult to wet. A filter with a low wettability may be a disadvantage for low pressure application like gravity-flow filtration.

- 5 There is an ongoing need to provide water filtration systems that have high loadings of active material without increasing pressure drop across the system. It would be further desirable to use larger granular carbon and less binder material. In addition, there is need to provide filters that can be easily wetted.

10 SUMMARY

Provided are filtration media, matrixes, and systems for liquid purification that utilize plasma-treated binder particles in conjunction with sorptive media. In an aspect, provided are filtration media comprising an adsorptive media and plasma-treated polymeric binder particles. In an embodiment, the surfaces of the plasma-treated polymeric binder particles
15 comprise an oxide, silicon, or both. In another embodiment, the adsorptive media comprises activated carbon. In a detailed embodiment, the activated carbon comprises particles having an average particle size of 45 micron or greater. Another embodiment provides that the polymeric binder particles further comprise an anti-microbial agent grafted thereto. One or more embodiments provide that the polymeric binder particles are
20 present in an amount in the range of 5 to 30 % by weight of the media. Some embodiments may further include binder particles that were not subjected to plasma-treatment.

A detailed embodiment provides that the filtration media is effective to provide an
25 increased cyst capacity as compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles. Another detailed embodiment provides that the filtration media is effective to provide an increased volatile organic component capacity as compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles. A further embodiment provides that the filtration
30 media has increased beam strength, compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles. Another embodiment provides

that the filtration media has increased hydrophilicity as compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles.

In one or more embodiments, the polymeric binder particles comprise polyethylene. Other
5 embodiments provide that the polyethylene comprises ultra high molecular weight polyethylene. Further embodiments provide that the polymeric binder particles comprise particles having an irregular, convoluted surface. Certain embodiments provide that the particles having an irregular, convoluted surface are formed from ultra high molecular weight polyethylene. Some embodiments provide that the polymeric binder particles
10 further comprise particles of substantially spherical shape.

Another aspect provides a filtration matrix comprising activated carbon and a plasma-treated ultra high molecular weight polyethylene binder adherent to the activated carbon. A detailed embodiment provides that the activated carbon is present in an amount in the
15 range of 50 to 85% by weight and the polyethylene binder is present in an amount in the range of 10 to 30 % by weight. In an embodiment, the polyethylene binder comprises particles having an irregular, convoluted surface. Some embodiments provide that the polyethylene binder further comprises an anti-microbial agent grafted thereto.

20 A detailed embodiment provides that the filtration media has increased hydrophilicity as compared to a comparative filtration matrix that does not contain the plasma-treated polymeric binder particles.

In a further aspect, provided is a filtration system comprising a filter matrix formed from
25 an adsorptive media and a plasma-treated polymeric binder, a housing surrounding the filter media, a fluid inlet, and a fluid outlet. In an embodiment, the adsorptive media comprises activated carbon, the polymeric binder comprises ultra high molecular weight polyethylene particles having an irregular, convoluted surface, and the surface of the polymeric binder comprises an oxide, silicon, or both.

30 Another aspect provides methods of filtering comprising contacting a fluid with a filtration media comprising an adsorptive media and a plasma-treated polymeric binder. In a

detailed embodiment, the filtration media has an increased cyst capacity as compared to a comparative filtration media that does not contain the plasma-treated binder. In one or more embodiments, the method further comprises locating the filtration media in a gravity flow filter device.

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In another aspect, provided are methods of making a filtration system, the methods comprising: treating polymeric binder particles with a plasma to form a plasma-treated polymeric binder; contacting an adsorptive media with the treated particles to form a media mixture; heating the media mixture form a filtration matrix; and inserting the
10 filtration block in a housing to form the filtration system. In one embodiment, the method further comprises grafting an anti-microbial agent to the plasma-treated polymeric binder. In a detailed embodiment, the treating step comprises: providing the polymeric binder particles in a chamber; pulling vacuum on the chamber; subjecting the particles to a gas; and applying RF pulses to the particles. In one or more embodiments, the gas comprises
15 silane, oxygen, or both. In another embodiment, the grafting step comprises: mixing the plasma-treated binder with the anti-microbial agent in an aqueous solution to form a mixture and drying the mixture.

DETAILED DESCRIPTION

20 Provided are filtration media, matrixes, and systems for liquid purification that utilize plasma-treated binder particles in conjunction with sorptive media. The plasma-treated binder particles are treated with a plasma gas, such as silane or oxygen or both to modify the binder particles to make them, for example, hydrophilic and more adhesive. The particles are mixed with sorptive media, such as activated carbon and/or diatomaceous
25 earth, where the mixture is then sintered and formed into a block or element. High loadings of sorptive media (up to, for example, about 90% by weight) are possible. Further, the addition of silica to the surface of binder particles appears to improve flow properties without interfering with the binding process.

30 Reference to “fluid treatment unit” or “fluid filtration system” includes a system containing a filtration media and a method of separating raw fluid, such as untreated

water, from treated fluid. This typically includes a filter housing for a filter element and an outlet to pass treated fluid away from the filter housing in an appropriate manner.

“Bulk wetting property” means the propensity of an article to absorb and soak up water.

5 By improving hydrophilicity, wetting time of the article can be reduced.

“Porous article” means an article having open tortuous pathways from its surface to its interior.

10 “Plasma treatment” refers to a process where high frequency electric or magnetic fields are used to create free radicals of a particular gas in an atmosphere where a binder is present. The free radicals modify the surface of the binder and possibly improve its performance. Plasma treatment “functionalizes” the binder surface. This term can include any other plasma-induced chemical or physical reaction that can change the binder surface property,
15 such as hydrophilicity.

Reference to “TMS” means tetramethylsilane gas, a gas used in plasma treatment.

20 “National Sanitation Foundation” or “NSF” refers to a private agency that certifies water filtration systems and materials used in filtration systems. NSF Standard 42 and 53, as used herein, are published standards that set forth test methods and acceptance criteria for water filtration systems for improving both aesthetic aspects and health effects of drinking water.

25 The term “impulse filling” or “applying impulses” means that a force is applied to the mold, causing a discrete, substantially vertical displacement that induces movement of at least a portion of the particles in the mold, causing the particles to assume a compact orientation in the mold. This includes indirect methods such as hammer blows to a table to which the molds are clamped and impacts to the table from a pneumatic cylinder, and
30 any suitable direct methods that displace the molds with a series of jarring motions. In some embodiments, the impulse filling comprises a series of discrete displacements (i.e., impulses) applied to the mold. Impulse filling differs from vibration in that there is a

period of non-movement or of little movement between the displacements. The period between displacements is typically at least 0.5 (in some embodiments, at least 1, 2, 3, 5, or even at least 10) seconds. The displacement applied to the mold has a vertical component. In some preferred embodiments, the vertical component (as opposed to the horizontal
5 component) accounts for a majority (in some embodiments, a substantial majority (>75%), or even nearly all (>90%)) of the molds movement.

The term “UHMW PE” refers to ultra-high molecular weight polyethylene having molecular weight of, for example, at least 750,000 and is described in commonly-owned
10 U.S. Patent No. 7,112,280, to Hughes *et al.*, incorporated herein by reference in its entirety.

The term “particles having an irregular, convoluted surface” refers to particles of unique morphology as set forth in U.S. Pat. No. 7,112,272 (Hughes et al.), hereby incorporated by
15 reference in its entirety, which, when compared to particles of substantially spherical shape, show higher surface areas and lower bulk density.

Detailed embodiments provide that the polymeric binder comprises ultra high molecular weight polyethylene. Other embodiments provide that the polymeric binder further
20 comprises particles having a generally spherical, non-porous structure. In specific embodiments, the particles having the irregular, convoluted surface have an average particle size in the range of 10 to 120 (or 20-50, or even 30-40) microns. Other specific embodiments provide that the particles having the generally spherical, non-porous structure have an average particle size in the range of 10 to 100 (or 20-80, or even 30-65)
25 microns. Reference to “small” convoluted particles includes particles generally having 30 micron mean and 0.25 g/cc density. Reference to “large” convoluted particles includes particles generally having 120 micron mean and 0.23 g/cc. Reference to “small” spherical particles includes particles generally having 60 micron mean and 0.45 g/cc.

30 The term “electrokinetic adsorption” includes processes that occur when particulates (called adsorbates) accumulate on the surface of a solid or very rarely a liquid (called

adsorbent), through Coulombic force, or other electrostatic interaction thereby forming a molecular or atomic film.

5 The term “adsorptive media” includes materials (called adsorbents) having an ability to adsorb particles via different adsorptive mechanisms. These media can be in the form of, for example, spherical pellets, rods, fibers, molded particles, or monoliths with hydrodynamic diameter between about 0.01 to 10 mm. If such media is porous, this attribute results in a higher exposed surface area and higher adsorptive capacity. The adsorbents may have combination of micropore and macropore structure enabling rapid
10 transport of the particles and low flow resistance. Reference to a “comparative filtration media” means a media that is formed from materials that have not been plasma-treated. That is, no portion of the binder used in the comparative filtration media has been processed with plasma gas such as TMS, O₂, or both. Reference to “comparative filtration matrix” means that the comparative filtration matrix contains a binder that has not been
15 plasma-treated.

The use of plasma-treated binders in standard carbon block formulations provided some improvements over the current state of the art. For example, improved performance of blocks for removal in the NSF 53 test for reduction of cryptosporidium cysts was
20 achieved. In addition, the use of plasma-treated binders permits lower binder contents and thus more active media in blocks, while maintaining strength and integrity. This enables higher capacity for removal of volatile organic compounds (VOC) on a gallons per block volume basis. Using plasma-treated binders also provides an ability to mold blocks with granular carbon (>100 micron mean particle size) with good mechanical strength. Break
25 strengths of blocks are generally improved when plasma-treated binders are used. Further, blocks having more a hydrophilic property are obtained with plasma-treated binders, which, in turn, improves initial wetting.

Methods of plasma treatment of porous materials are provided in U.S. Patent Nos.
30 6,878,419 and 7,125,603, also, method and apparatus for plasma treatment of particles are provided in US Patent Nos. 6,015,597 and 6,197,120 the disclosures of which are incorporated by reference herein. In general terms, plasma treatment is achieved by

treating the binder under vacuum with a gas, such as silane in the form of, for example, tetramethylsilane (TMS) or oxygen (O₂). For TMS treatment, TMS was introduced into a glass chamber at a flow rate of approximately 100 cc/min for 30 minutes. For oxygen treatment, oxygen gas was introduced into the chamber at a flow rate of approximately 180 cc/min for 30 minutes. For combined treatment, the TMS and O₂ treatments were performed sequentially for 30 minutes each. Generally, the pressure when TMS or oxygen gas was present in the chamber did not exceed about 1 torr. The materials in the chamber were then subjected to RF plasma in a pulsed mode. Pulsing was used to minimize the formation of powder dust resulting from gas phase polymerization. In treatments where binders were treated first in a TMS atmosphere, and then subsequently in an O₂ atmosphere, without intending to be bound by theory, it is believed that silicon oxides are deposited on the surface of the polymer binder. For treatment in O₂ gas only, without intending to be bound by theory, it is believed that the surface of the polymer is oxidized. Advantages similar to TMS followed by O₂ treatment were seen with O₂ treatment only. With regard to treatment in TMS gas only, advantages similar to the other treatments were seen with the exception of the hydrophilic effect.

In the case where the plasma treatment is done in two steps, the first plasma is derived from components comprising at least one organosilane having at least one C-H bond, which may be an sp³, sp² or sp C-H bond. Typically, the organosilane has a plurality of C-H bonds, for example, at least 2, at least 3, at least 5, at least 9, and/or even at least 12 C-H bonds, or more. Typically the organosilane(s) are selected such that they have sufficient vapor pressure under plasma treatment conditions that a plasma is formed.

Exemplary organosilanes having at least one C-H bond include: alkylsilanes such as, for example, tetramethylsilane, methylsilane, dimethylsilane, diethylsilane, diethylmethylsilane, propylsilane, trimethylsilane, and ethylsilane; alkoxysilanes and siloxanes such as, for example, tetraethylorthosilicate (TEOS), and tetramethylcyclotetrasiloxane (TMCTS); alkylenepolysilanes such as, for example, disilanomethane, bis(methylsilano)methane, 1,2-disilanoethane, 1,2-bis(methylsilano)ethane, 2,2-disilanopropane, dimethyldisilanoethane,

dimethyldisilanopropane, tetramethyldisilanoethane, and tetramethyldisilanopropane; alkenylsilanes such as, for example, vinylmethylsilane, and divinyl dimethylsilane; aryl silanes such as, for example, phenylsilane, phenyldimethylsilane, and phenyltrimethylsilane; alkylpolysilanes such as, for example, 1,1,2,2-tetramethyldisilane, 5 hexamethyldisilane, 1,1,2,2,3,3-hexamethyltrisilane, and 1,1,2,3,3-pentamethyltrisilane. Combinations of organosilanes may also be used. The organosilane may have substituents such as, for example, amino groups, hydroxyl groups, and/or halo (e.g., fluoro, bromo, chloro) groups, although these may tend to diminish effectiveness of the first plasma treatment. In addition, the first plasma may additionally include gaseous component(s), 10 for example, selected from the group consisting of: oxygen, nitrogen, nitrogen dioxide, nitrous oxide, ammonia, and sulfur dioxide, although these may tend to diminish effectiveness of the first plasma treatment.

Plasma-treated binders can be further subject to grafting with an anti-microbial agent. In 15 one or more embodiments, carbon blocks were made with binders that were first plasma-treated with TMS followed by O₂, and then grafted with an anti-microbial agent. The antimicrobial agent as an organosilicon quaternary ammonium compound in the form of 3-trimethoxysilylpropyl dimethyloctadecyl ammonium chloride, available under the tradename AEM 5700 from Aegis of Midland, MI. In order to graft the anti-microbial, an 20 aqueous solution containing the anti-microbial was prepared and then sprayed onto the plasma-treated binder with mixing. The material was then dried overnight in a convection oven at 85°C with a nitrogen gas flow. It was found that binders that were first plasma-treated with TMS followed by O₂, and then grafted with an organosilicon quaternary ammonium compound gave carbon blocks with dramatically improved performance for 25 turbidity reduction. While not intending to be bound to a particular theory, it is postulated that the positive charge imparted to the binder in this process improves collection of charged particulates.

It was found that the use of plasma-modified binders in a carbon block formulation 30 dramatically improved the efficiency of removal of cryptosporidium cysts. In our testing, a carbon block formulation that exhibited poor cyst reduction (failing the ANSI/NSF 53 standard) was modified to use plasma-treated binders. Commercial binders were replaced

by the same binders functionalized by a plasma process, using TMS, oxygen (O₂), or both. Except for this, the formulation and process were identical. With binders treated with TMS followed by O₂, the blocks exhibited surprisingly robust cyst reduction, to the point that the effluent water had no detectable particles.

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The improvement in cyst performance can allow manufacture of blocks with larger carbon particles. In current commercial carbon block technology, cyst retentive blocks contain activated carbon particles primarily of sizes less than 325 mesh (43 micron). With the functionalized binder, blocks have been made cyst retentive with carbon particles

10 primarily between 80 and 325mesh (45 to 175 micron). This advantage enables the production of lower pressure drop, cyst-retentive blocks. It also allows use of less binder and more active media. Further, it can improve manufacturing, because ultra fine carbon particles are difficult to process and difficult to produce to a tight particle size distribution.

15 Use of plasma-treated binders can significantly increase the mechanical strength of blocks. For blocks with the same formulation, substituting plasma-treated (TMS, O₂ or both) binders for the same commercial binders without modification has increased beam deflection force nearly 2X. Beam deflection force (BDF) or break strength is commonly used as a specification to assure that blocks are strong enough so as not to fail during

20 shipping and handling. Production of blocks with higher beam strength enables manufacture of blocks with smaller diameters and with smaller wall thicknesses. The current trend in demand for water filter blocks is toward smaller blocks to fit in tight spaces (e.g., refrigerators and faucets). Another trend is for higher water flow rates at lower pressure drops. This can be enabled by using smaller block wall thickness.

25

Use of plasma-modified binders reduces the required binder content and thereby allows use of more active media. In current carbon block technology, an exemplary formulation contains a binder content of around 50% and an active media content of around 50% by mass. Using plasma functionalized binders, blocks have been made with binder contents

30 as low as 10% by mass. These low binder contents have been applied to blocks with a difficult-to-mold geometry (e.g., 1.1" ID X 9/16" ID X 6" long).

Blocks made with low binder contents have shown dramatically improved performance for VOC removal in terms of gallons capacity per unit block volume. VOC capacities 2 to 4 times higher than that of current commercial carbon blocks have been achieved. For example, blocks 1.1" diameter and 5" long have achieved VOC service life (per NSF Standard 53) of well over 160 gallons water ($>45 \text{ gal/in}^3$). Most commercial water filter blocks have VOC capacities less than 20 gal/in^3 .

The use of plasma-treated binders allows production of blocks from granular carbons (>100 micron mean). Using blocks containing silica-enhanced binders have been made with 80x200 and 50x200 mesh carbons with no cracking and with minimal particle shedding. This is in sharp contrast to blocks made with the same binders without silica modification. With standard binders, blocks made with these particle sizes have shown very high frequency of cracking and substantial particle shedding.

Another utility of the current invention is to improve the wetting of the binder in a carbon block. Other components in a carbon block (activated carbon, lead reduction media, etc.) are more hydrophilic than polyethylene. A block with improved wettability would have an advantage for gravity flow filtration applications like pitcher applications, where rapid wetting of media is important.

Unless otherwise indicated, all numbers expressing quantities of ingredients, properties such as molecular weight, reaction conditions, and so forth used in the specification and claims are to be understood as being modified in all instances by the term "about."

Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and attached claims are approximations that may vary depending upon the desired properties sought to be obtained by the present disclosure. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claims, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques.

Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the disclosure are approximations, the numerical values set forth in the specific examples

are reported as precisely as possible. Any numerical value, however, inherently contain certain errors necessarily resulting from the standard deviation found in their respective testing measurements.

5 EXAMPLES

EXAMPLE 1

UHMW PE binders in the form of small convoluted particles (Ticona GUR 2126) and small spheres (Ticona GUR 4150-3) were surface modified in one of three ways using: tetramethylsilane (TMS) only, oxygen only, and TMS followed by oxygen. Air flow, 10 BDF, and cyst tests were performed on the blocks.

Generally, a binder was treated in batches of approximately 1500 g in a pilot reactor. The pilot reactor contained a rotating glass tube in an electromagnetic field. About 500 mL of inert beads was added to the binder in order to help it stir in the pilot reactor. Once the 15 binder was placed into the glass tube, the gas inside was evacuated to a pressure of about 0.3 torr. The rotor on the glass chamber was set to rotate at about 8 rpm. For tetramethylsilane treatment, TMS was introduced into the chamber at a flow rate of approximately 100 cc/min for 30 minutes. For oxygen treatment, oxygen gas was introduced into the chamber at a flow rate of approximately 180 cc/min for 30 minutes. 20 For combined treatment, TMS gas and O₂ treatments above were performed sequentially for 30 minutes each. The pressure when TMS or oxygen gas was present in the treatment chamber did not exceed about 1 torr.

Once the desired atmosphere was created, RF pulsing was initiated with a forward of 25 approximately 200W and reflection of approximately 40W. When treatment was complete, the binder was removed from the reactor and the inert beads were removed using a sifter.

For each of the 3 different treatments and 1 control with untreated binder, a batch of 24 30 blocks (1.1" OD X 4.5" L) was made. The formulation of the blocks was as follows.

Material	Wt %
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Coconut activated carbon, nominal 100 micron mean	60
Coconut activated carbon, nominal 20 micron	17
Titanium silicate lead removal media	10
Treated UHMWPE (small convoluted) binder	6.5
Treated UHMWPE (small sphere) binder	6.5

Blocks were made in molds 6" long X 1.1" OD. Molds were filled by impulse filling to achieve maximum density. Molds were baked in a convection oven for 1 hour at 177°C, eight at a time. After removal from the convection oven, blocks were compressed in their mold with a force of approximately 50 lbf. Blocks had an OD of 1.1", ID of .375", and were cut to a length of 4.5". Blocks had a weight of approximately 37-39 grams. Blocks were end-capped and randomly allocated to their respective tests. The BDF sample size was 16, the results for which are provided in Table 1a, and the cyst tests sample size was 8, the results for which are provided in Table 1b.

TABLE 1a:

		TMS + O ₂	TMS	O ₂	None
AF (25 L/min in H ₂ O)	Mean	17.7	18.4	18.4	17.7
	Sdev	1.3	0.7	1.5	1.0
	Max	21.6	20.5	23.1	20.8
	Min	13.7	16.4	13.8	14.6
BDF (lbf)	Mean	14.0	13.2	14.7	11.4
	Sdev	1.5	1.7	1.7	1.6
	Max	18.4	18.4	19.8	16.1
	Min	9.6	8.0	9.6	6.7

TABLE 1b:

	Pass	Fail
TMS + O ₂	100%	0%
TMS	100%	0%
O ₂	100%	0%
None	88%	12%

The BDF data suggests that there is an improvement in strength between treated blocks by either of the 3 treatment methods, compared to untreated blocks. Blocks treated with oxygen plasma were the strongest.

- 5 The cyst test data show that for blocks made with plasma-treated binders (oxygen, TMS or TMS followed by O₂), all blocks passed with at least 99.95% efficiency. For blocks made with untreated polymers, 12% of the blocks tested failed the cyst test.

EXAMPLE 2

- 10 Carbon blocks were made with binders treated with TMS followed by O₂ according to the formula of Table 2a:

TABLE 2a:

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	60
Coconut activated carbon, nominal 20 micron	20
Titanium silicate lead removal media	7
Treated UHMWPE (small convoluted) binder (Ticona GUR 2126)	6.5
Treated UHMWPE (small sphere) binder (Ticona GUR 4150-3)	6.5

- 15 Materials were mixed for 2 minutes in a drill press mixer. Molds (1.1"OD X 3/8" ID X 12" long) were filled by impulse filling to achieve maximum density. Molds were baked at 180°C for 45 minutes in a convection oven. After baking, the blocks were compressed with a piston with a constant force of 100 lbf for 30 sec. Blocks were cooled and removed from the molds. Blocks were cut to lengths of 4" and then end capped.

- 20 Table 2b shows data for 8 blocks made by this method:

TABLE 2b

Block # TMS & O ₂	Mass, g	Air Flow Resistance at 25	Pressure drop at 0.5 gpm, psi
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Plasma		L/min, in H ₂ O	
2-A	33.8	54.9	-
2-B	33.5	52.8	-
2-C	34.4	57.8	-
2-D	33.7	51.8	12.2
2-E	33.5	51.7	-
2-F	34.6	58.7	-
2-G	33.4	50.9	11.7
2-H	33.8	54.2	-
Mean	33.8	54.1	-
Std. Deviation	0.44	2.9	-

Tests were conducted on blocks 2-D and 2-G to measure performance of these blocks for cyst reduction from water. These tests followed NSF Standard 53 protocol using a surrogate test dust. Both blocks 2-D and 2-G showed robust performance for cyst reduction. That is, the blocks showed >99.95 efficiency. The effluent particle count for 3-4 micron particles was non-detect throughout the test.

EXAMPLE 3

COMPARISON

Carbon blocks were made with polyethylene binders (with no plasma treatment) according to the formula of Table 3a:

TABLE 3a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	60
Coconut activated carbon, nominal 20 micron	20
Titanium silicate lead removal media	7
Treated UHMWPE (small convoluted)binder (Ticona GUR 2126)	6.5
Treated UHMWPE (small sphere) binder (Ticona GUR 4150-3)	6.5

Blocks made in accordance with the method of Example 2 were tested and had the properties according to Table 3b:

TABLE 3b

Block # TMS & O ₂ Plasma	Mass, g	Air Flow Resistance at 25 L/min, in H ₂ O	Pressure drop at 0.5 gpm, psi
3-A	31.8	42.8	8.5
3-B	33.2	50.1	-
3-C	33.3	54.1	-
3-D	31.1	38.8	8.0
3-E	33.3	48.8	-
3-F	32.0	42.0	-
3-G	33.7	54.6	-
3-H	31.6	38.8	-
Mean	32.5	46.3	-
Std. Deviation	0.98	6.5	-

As shown, the standard deviations in the block mass and in the air flow resistance were more than double those for blocks made with the plasma-treated binder in Example 3.

- 5 Also, the mean air flow resistance and mean block mass for the block of Example 2 were higher than those of Example 3. These are indications that the modified binder improved mold filling, giving a higher fill density and a more reproducible fill density.

- 10 Tests were conducted on blocks 3-A and 3-D to measure performance of these blocks for cyst reduction from water, following an NSF Standard 53 protocol using a surrogate test dust. Both blocks made with binder that was not modified failed the test for cyst reduction.

EXAMPLE 4

15 COMPARISON

Carbon blocks were made with polyethylene binders (with no plasma treatment) according to the formula of Table 4a. The formulation, compared with Example 3, was adjusted by adding more fine carbon to improve cyst performance.

TABLE 4a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	43

Coconut activated carbon, nominal 20 micron	35
Titanium silicate lead removal media	7
UHMWPE (small convoluted) binder (Ticona GUR 2126)	7.5
UHMWPE (small sphere) binder (Ticona GUR 4150-3)	7.5

Blocks made in accordance with the method of Example 2 were tested and had the properties according to Table 4b.

TABLE 4b:

Block # (No Plasma)	Compression force, lbf	Pressure drop at 0.5 gpm, psi	Cyst performance
4-A	50	10.1	Fail
4-B	100	12.2	Marginal pass
4-C	100	13.4	Marginal pass
4-D	200	17.2	Marginal pass
4-E	200	14.3	Marginal pass
4-F	300	15.8	Fail

5

In these blocks made from binders without plasma treatment, tests characterized as “Fail” had efficiencies less than 99.95% and failed the NSF 53 criterion. Tests characterized as “Marginal Pass” had efficiencies above 99.95% at the prescribed NSF sample points, but at other points during the test, efficiency dropped below 99.95%. Also, the tests labeled

“Marginal Pass” had detectable particles measured in the effluent.

10

EXAMPLE 5

Carbon blocks were made with binders treated with TMS followed by O₂ according to the formula of Table 5a:

15

TABLE 5a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	20
Coconut activated carbon, nominal 20 micron	45

Titanium silicate lead removal media	7
Treated UHMWPE (small convoluted) binder (Ticona GUR 2126)	10
Treated UHMWPE (small sphere) binder (Ticona GUR 4150-3)	18

Blocks made in accordance with the method of Example 2 were tested and had the properties according to Table 5b:

TABLE 5b

Block # (TMS & O ₂ Plasma)	Mass, g	Air Flow Resistance at 25 L/min, in H ₂ O	Beam Deflection Force, lbf
5-A	69.3	92.2	63 lbf
5-B	68.0	86.7	55 lbf
5-C	70.6	100	74 lbf
5-D	68.7	91.1	55 lbf

5

The beam deflection force is defined as the force required to break the block when it is supported on both ends and subjected to a force perpendicular to the block applied at a point equidistant from the ends of the block. It is routinely used as a measure of block strength and is used as a specification to ensure that blocks do not break during shipping and handling.

10

EXAMPLE 6

COMPARISON

Carbon blocks were made with polyethylene binders (with no plasma treatment) according to the formula of Table 6a:

15

TABLE 6a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	20
Coconut activated carbon, nominal 20 micron	45
Titanium silicate lead removal media	7
Treated UHMWPE (small convoluted) binder	10

(Ticona GUR 2126)	
Treated UHMWPE (small sphere) binder (Ticona GUR 4150-3)	18

Blocks made in accordance with the method of Example 2 were tested and had the properties according to Table 6b:

TABLE 6b

Block # (No Plasma)	Mass, g	Air Flow Resistance at 25 L/min, in H ₂ O	Beam Deflection Force, lbf
6-A	66.7	76.6	48 lbf
6-B	67.0	72.2	49 lbf
6-C	64.8	60.6	42 lbf
6-D	68.6	78.4	53 lbf

5

As shown, the beam deflection force was generally lower for blocks made without the plasma-treated binder in Example 6 as compared to the blocks of Example 5. This indicates that the modified binder improved beam deflection force.

EXAMPLE 7

10 Carbon blocks were made with binders treated with TMS followed by O₂ according to the formula of Table 7a:

TABLE 7a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	63
Coconut activated carbon, nominal 20 micron	20
Titanium silicate lead removal media	7
Treated UHMWPE (small convoluted) binder (Ticona GUR 2126)	5
Treated UHMWPE (small sphere) binder (Ticona GUR 4150-3)	5

15 Blocks made in accordance with the method of Example 2 were tested. These blocks were intact with no cracking. Mechanical strength and the amount of particle shedding were

deemed acceptable for water filtration application. The total binder content of the blocks was 10%. The total active media content was 90%.

EXAMPLE 8

- 5 Blocks made with binders that were plasma-treated first with TMS and followed by O₂ were produced in accordance with the formula in Example 2, Table 2a, having 1.1" OD. Table 8 summarizes initial VOC test results for these blocks. Measurements of concentrations of chloroform (CHCl₃) in the effluent water when challenged with water containing 300 ppb chloroform are provided as a function of gallons water treated. In the
- 10 NSF 53 protocol, the filter must maintain an effluent CHCl₃ level below 15 ppb (95% reduction) throughout its rated service life.

TABLE 8

Block # TMS & O ₂ Plasma	Block Length, in.	ID, in.	Vol, in ³	Breakthrough CHCl ₃ concentration (ppb) at given volume (gal)					
				40 gal	80 gal	100 gal	120 gal	140 gal	160 gal
8-A	3.5	9/16	2.46	1.5	2.3	15	50	95	139
8-B	5.0	9/16	3.51	<0.5	0.88	1.2	1.4	1.9	4.1
8-C	4.0	3/8	3.36	<0.5	<0.5	1.1	2.6	15	39
8-D	4.5	3/8	3.78	<0.5	<0.5	0.5	0.6	0.7	2.5
8-E	4.0	3/8	3.36	ND	<0.5	<0.5	0.7	2.3	7.6
8-F	4.5	3/8	3.78	0.7	1.3	2.7	7.0	26	32
8-G	4.5	3/8	3.78	<0.5	<0.5	<0.5	<0.5	0.6	0.7

- 15 The results show >160 gal VOC capacity was attained with a block 4.5" long. For the 5" block, the volume water treated for VOC per unit block volume was >45 gallons/in³.

EXAMPLE 9

- 20 Carbon blocks were made with binders treated with TMS followed by O₂ according to the formula of Table 9. The carbon used was granular in contrast to the fine carbon of the previous examples.

Table 9

Material	Wt %
Mead Westvaco Aquaguard 80X200	86
Treated UHMWPE (large convoluted) binder Ticona GUR 2122	14

Blocks of size 3.5" OD X 0.75" ID X 20" L were made according to the method of Example 2, except that bake time was longer (about 2 hours) to allow heat transfer in the larger mold.

All blocks were intact (no cracks) and showed minimal particle shedding. All previous attempts to make blocks with this carbon particle size and with this block geometry yielded defective blocks (cracking).

EXAMPLE 10

Carbon blocks, formed with a coarser carbon PSD were made with binders treated with TMS followed by O₂ according to the formula of Table 10a:

TABLE 10a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	70
Coconut activated carbon, nominal 20 micron	10
Titanium silicate lead removal media	7
Treated UHMWPE (small convoluted) binder (Ticona GUR 2126)	6.5
Treated UHMWPE (small sphere) binder (Ticona GUR 4150-3)	6.5

Table 10b shows data for 4 blocks made according to the method of Example 2:

TABLE 10b

Block # TMS & O ₂ Plasma	Air Flow Resistance at 25 L/min, in H ₂ O	Pressure drop at 0.5 gpm, psi
10-A	76.3	14.8

10-B	66.0	13.0
10-C	56.7	12.9
10-D	64.4	12.2

Tests were conducted on these blocks to measure performance of these blocks for cyst reduction from water. These tests followed NSF Standard 53 protocol using a surrogate test dust. All of the blocks 10-A to 10-D showed robust performance for cyst reduction.

5

EXAMPLE 11

Disks were made with binders treated with TMS followed by O₂ according to the formula of Table 11a:

TABLE 11a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	80
Treated UHMWPE (small convoluted) binder (Ticona GUR 2126)	20

10

The above components were mixed and then molded into disks (4.5" diameter X 3/8" thick). A cap was placed on mold but the mold was not compressed.

15

The above disks were tested for wettability with water. 10 mL of tap water was applied to the surface of the disk using a syringe. Water was applied over a period of 5 sec. The total time for water on the disk to be completely absorbed into the disk was then recorded. Data from this test are shown in Table 11b:

TABLE 11b

Block # TMS & O ₂ Plasma	Time for complete water absorption, sec
11-A	31
11-B	32
11-C	38
11-D	45
11-E	32
11-F	24
11-G	19

Mean	32
------	----

EXAMPLE 12

COMPARISON

Disks were made with polyethylene binders (with no plasma treatment) according to the formula of Table 12a:

TABLE 12a

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	80
UHMWPE (small convoluted) binder (Ticona GUR 2126)	20

Disks were made by the method of Example 11 and tested for wettability according to Example 11. Data from the testing are shown in Table 12b:

TABLE 12b

Block # No Plasma	Time for complete water absorption, sec
12-A	40
12-B	64
12-C	59
12-D	51
12-E	97
12-F	150
Mean	87

As shown, the wettability time was generally higher for disks made without the plasma-treated binder in Example 12 as compared to the disks of Example 11. This indicates that the modified binder improved wettability.

EXAMPLE 13

Carbon blocks were made with binders that were first treated with TMS followed by O₂, and then grafted with an anti-microbial agent. The antimicrobial agent as an organosilicon quaternary ammonium compound in the form of 3-trimethoxysilylpropyl

dimethyloctadecyl ammonium chloride, available under the tradename AEM 5700 from

Aegis of Midland, MI. In order to graft the anti-microbial, an aqueous 1% solution containing the anti-microbial was sprayed onto the plasma-treated binder and mixed to the consistency of a paste. The material was then dried overnight in a convection oven at a temperature of 85 C with nitrogen gas flowing through the oven

5

Carbon blocks were prepared according to the formula of Table 13:

TABLE 13

Material	Wt %
Coconut Shell activated carbon (-325 mesh)	19
Coconut Shell activated carbon (80X 325 mesh)	21
Titanium silicate lead removal media	10
Untreated UHMWPE (large convoluted) binder (Ticona GUR 2122)	20
Treated & AM grafted UHMWPE (small convoluted) binder (Ticona GUR 2126)	30

10

Blocks having 2.1" OD x 9.3" long were prepared. These blocks were tested for turbidity according NSF 53 standards and achieved a reduction of turbidity in the effluent to less than 0.25 NTU for over 50 cycles. The NSF standard requires a reduction of 0.5 NTU.

EXAMPLE 14

15

Carbon blocks were made with binders that were first treated with TMS followed by O₂, and then grafted with an anti-microbial agent, as discussed by Example 13.

Carbon blocks were prepared according to the formula of Table 14:

TABLE 14

Material	Wt %
Coconut activated carbon, nominal 100 micron mean	60
Coconut activated carbon, nominal 20 micron	17

Titanium silicate lead removal media	10
Treated & AM grafted UHMWPE (small sphere) binder (Ticona GUR 4150-3)	6.5
Treated & AM grafted UHMWPE (small convoluted) binder (Ticona GUR 2126)	6.5

Blocks having 1.1" OD x 4" long were prepared using impulse filling and curing with compression. These blocks were tested for turbidity according NSF 53 standards and achieved a reduction of turbidity in the effluent to less than 0.5 NTU for 19 cycles.

For a comparative block having a binder that was not plasma treated, the block failed the turbidity test. That is, for 19 cycles with the comparative block, the turbidity results were greater than 0.5 NTU.

Reference throughout this specification to "one embodiment," "certain embodiments," "one or more embodiments" or "an embodiment" means that a particular feature, structure, material, or characteristic described in connection with the embodiment is included in at least one embodiment of the invention. Thus, the appearances of the phrases such as "in one or more embodiments," "in certain embodiments," "in one embodiment" or "in an embodiment" in various places throughout this specification are not necessarily referring to the same embodiment of the invention. Furthermore, the particular features, structures, materials, or characteristics may be combined in any suitable manner in one or more embodiments.

Although the invention herein has been described with reference to particular embodiments, it is to be understood that these embodiments are merely illustrative of the principles and applications of the present invention. It will be apparent to those skilled in the art that various modifications and variations can be made to the method and apparatus of the present invention without departing from the spirit and scope of the invention.

Thus, it is intended that the present invention include modifications and variations that are within the scope of the appended claims and their equivalents.

What is claimed is:

1. A filtration media comprising an adsorptive media and plasma-treated polymeric binder particles.

5 2. The filtration media of claim 1, wherein the surfaces of the plasma-treated polymeric binder particles comprise an oxide, silicon, or both.

3. The filtration media of claim 1, wherein the adsorptive media comprises activated carbon.

10

4. The filtration media of claim 3, wherein the activated carbon comprises particles having an average particle size of 45 micron or greater.

15

5. The filtration media of claim 1, wherein the polymeric binder particles further comprise an anti-microbial agent grafted thereto.

6. The filtration media of claim 1, wherein the polymeric binder particles are present in an amount in the range of 5 to 30 % by weight of the media.

20

7. The filtration media of claim 1, wherein the filtration media is effective to provide an increased cyst capacity as compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles.

25

8. The filtration media of claim 1, wherein the filtration media is effective to provide an increased volatile organic component capacity as compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles.

30

9. The filtration media of claim 1, wherein the filtration media has increased beam strength, compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles.

10. The filtration media of claim 1, wherein the filtration media has increased hydrophilicity as compared to a comparative filtration media that does not contain the plasma-treated polymeric binder particles.

5 11. The filtration media of claim 1, wherein the polymeric binder particles comprise polyethylene.

12. The filtration media of claim 11, wherein the polyethylene comprises ultra high molecular weight polyethylene.

10

13. The filtration media of claim 1, wherein the polymeric binder particles comprise particles having an irregular, convoluted surface.

15

14. The filtration media of claim 13, wherein the particles having an irregular, convoluted surface are formed from ultra high molecular weight polyethylene.

15. The filtration media of claim 11, wherein the polymeric binder particles further comprise particles of substantially spherical shape.

20

16. A filtration matrix comprising activated carbon and a plasma-treated ultra high molecular weight polyethylene binder adherent to the activated carbon.

25

17. The filtration matrix of claim 16, wherein the activated carbon is present in an amount in the range of 50 to 85% by weight and the polyethylene binder is present in an amount in the range of 10 to 30 % by weight.

18. The filtration matrix of claim 16, wherein the polyethylene binder comprises particles having an irregular, convoluted surface.

30

19. The filtration matrix of claim 16, wherein the polyethylene binder further comprises an anti-microbial agent grafted thereto.

20. The filtration matrix of claim 1, wherein the filtration media has increased hydrophilicity as compared to a comparative filtration matrix that does not contain the plasma-treated polymeric binder particles.

21. A filtration system comprising a filter matrix formed from an adsorptive media and a plasma-treated polymeric binder, a housing surrounding the filter media, a fluid inlet, and a fluid outlet.

22. The filtration system of claim 21, wherein the adsorptive media comprises activated carbon, the polymeric binder comprises ultra high molecular weight polyethylene particles having an irregular, convoluted surface, and the surface of the polymeric binder comprises an oxide, silicon, or both.

23. A method of filtering comprising contacting a fluid with a filtration media comprising an adsorptive media and a plasma-treated polymeric binder.

24. The method of claim 23, wherein the filtration media has an increased cyst capacity as compared to a comparative filtration media that does not contain the plasma-treated binder.

25. The method of claim 23 further comprising locating the filtration media in a gravity flow filter device.

26. A method of making a filtration system, the method comprising:
treating polymeric binder particles with a plasma to form a plasma-treated polymeric binder;
contacting an adsorptive media with the treated particles to form a media mixture;
heating the media mixture form a filtration matrix; and
inserting the filtration block in a housing to form the filtration system.

27. The method of claim 26, further comprising grafting an anti-microbial agent to the plasma-treated polymeric binder.

28. The method of claim 26, wherein the treating step comprises:

providing the polymeric binder particles in a chamber;

pulling vacuum on the chamber;

subjecting the particles to a gas; and

5 applying RF pulses to the particles.

29. The method of claim 28, wherein the gas comprises silane, oxygen, or both.

30. The method of claim 27, wherein the grafting step comprises: mixing the plasma-

10 treated binder with the anti-microbial agent in an aqueous solution to form a mixture and drying the mixture.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2008/085491**A. CLASSIFICATION OF SUBJECT MATTER*****C02F 3/10(2006.01)i, B01D 39/00(2006.01)i***

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 8: C02F, B01D

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

Korean Utility Models and applications for Utility Models since 1975

Japanese Utility Models and applications for Utility Models since 1975

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

eKIPASS(KIPO internal) "filtration media", "adsorptive media", "plasma-treated polymeric binder particles", "activated carbon", "silicon", "filter matrix", and similar terms

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6833075 B2 (HUGHES, K. D.) See abstract, claims (1-125), and figures (1-2b) 21 Dec. 2004	1-30
A	US 6764601 B1 (LEVY, E. et al.) See abstract, and claims (1-15) 20 Jul. 2004	1-30
A	KR 1020050093851 A (MATTSUSHITA ENG. CO., LTD.; UNITIKA CO., LTD.) See abstract, claims (1-18), and figures (1-19) 23 Sep. 2005	1-30

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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