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**Adamson et al.**

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(54) **REGENERATABLE SYSTEM FOR CONTAMINANT REMOVAL**

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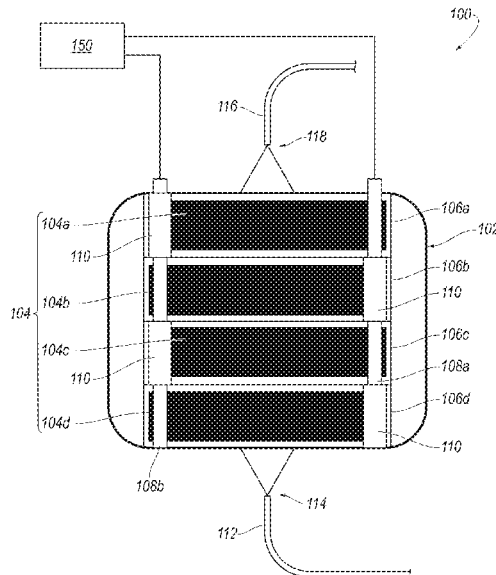
(57) **ABSTRACT**

A system and method for water purification by capture of contaminants in an aqueous mixture is described herein. A system and method for regenerating the capture system is also described. An integrated capture and regeneration system and method is also described including a separation vessel that houses a capture bed and an electrode in electrical contact with the bed and a power source for applying a voltage to the electrode. The applied voltage enhances capture of the contaminant from aqueous liquid on the capture bed and modulation of the applied voltage enhances release of contaminant on the capture bed into aqueous wash liquid to regenerate the bed. The aqueous wash liquid may contain a counter ion that binds to the contaminant forming an aggregate contaminant phase that separates from the aqueous wash liquid.

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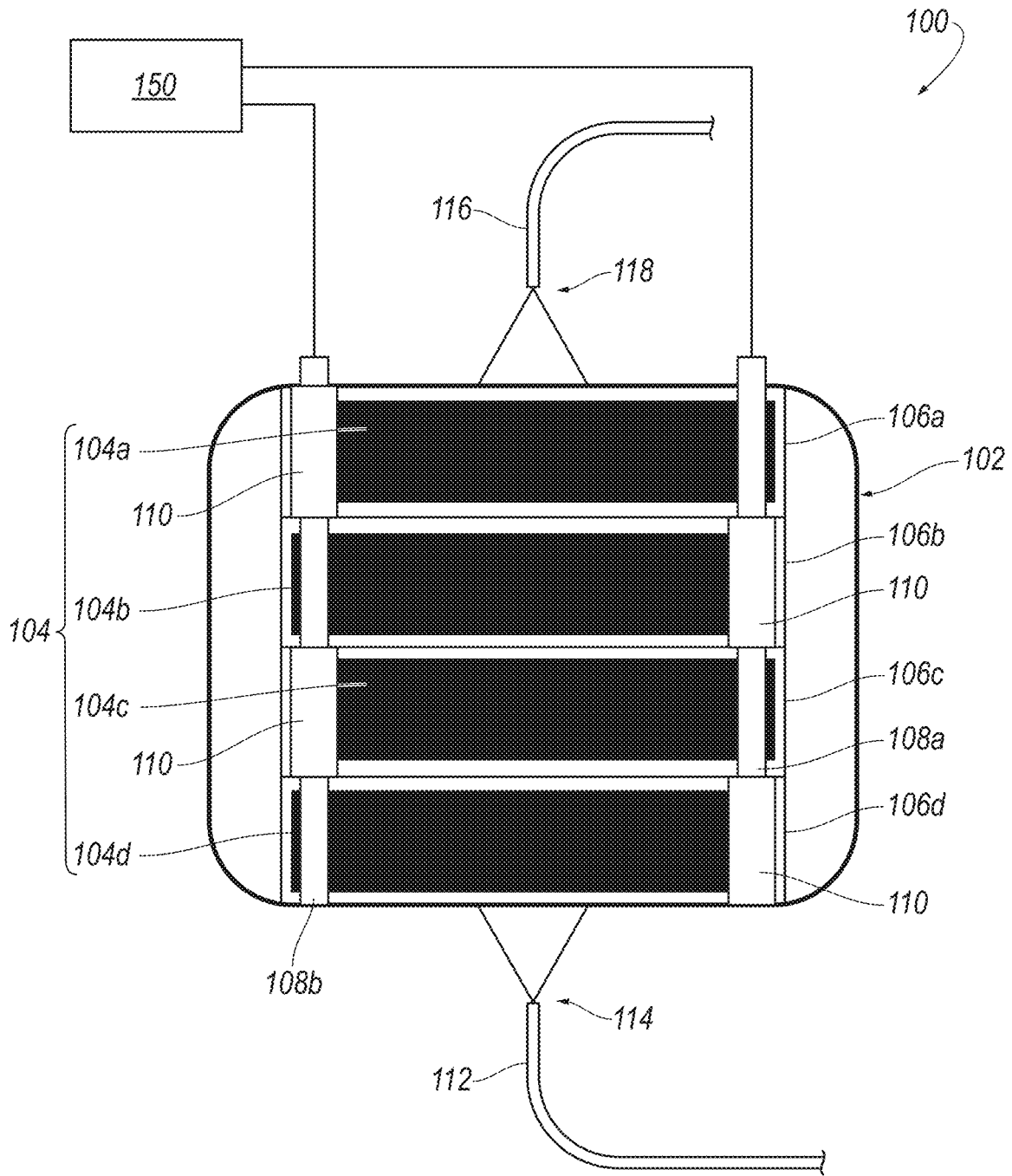


FIG. 1

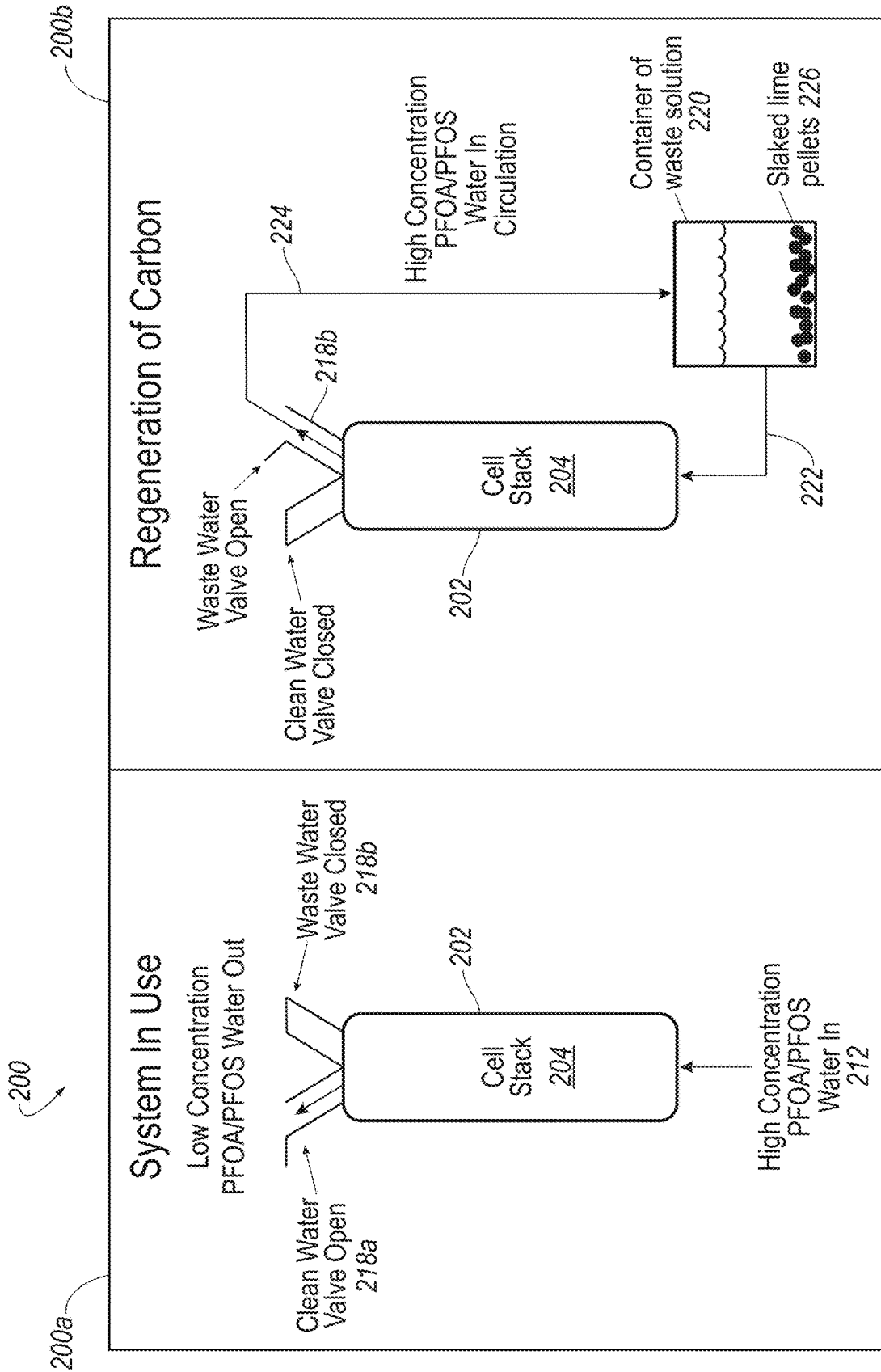


FIG. 2B

FIG. 2A

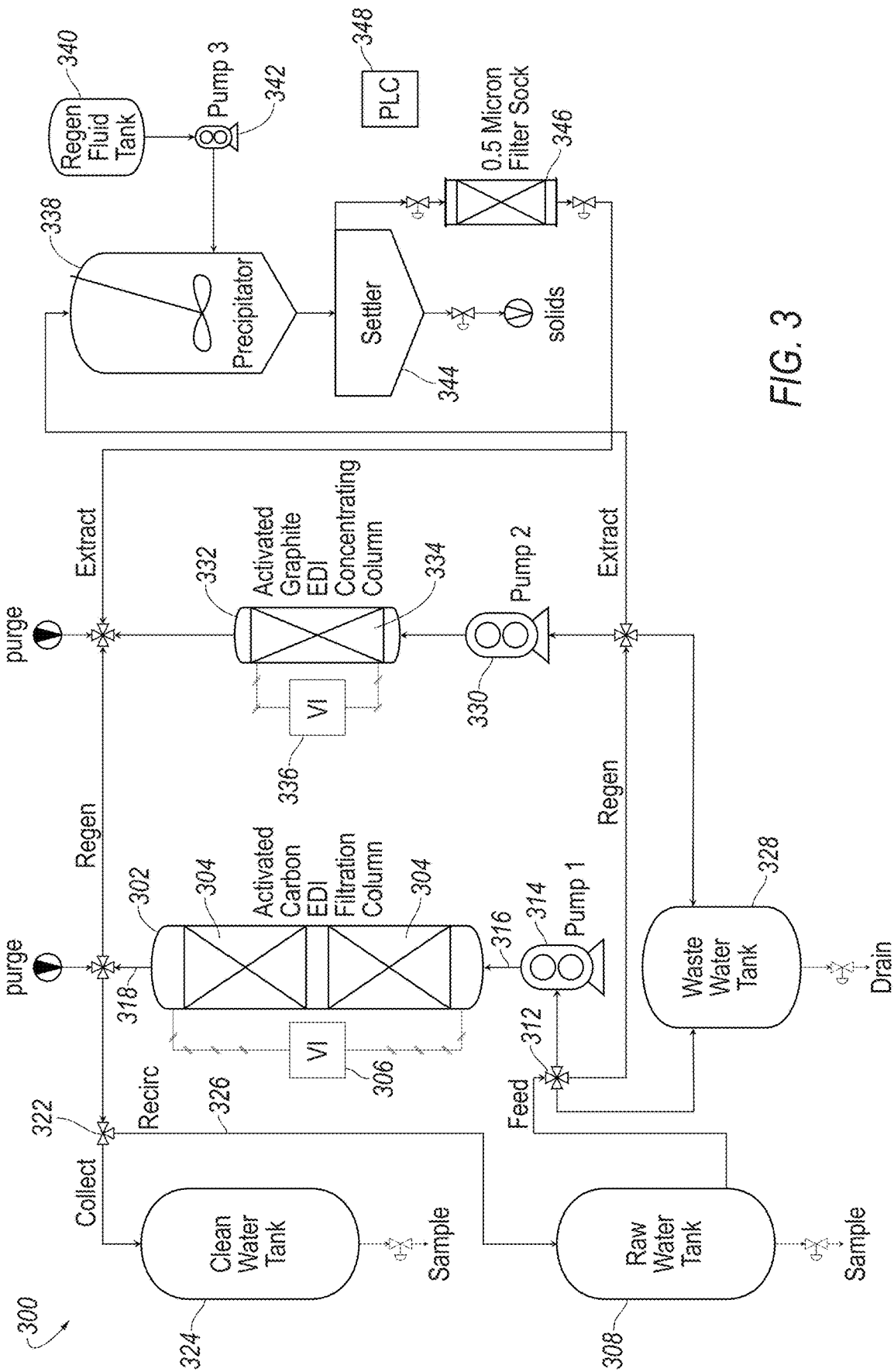


FIG. 3

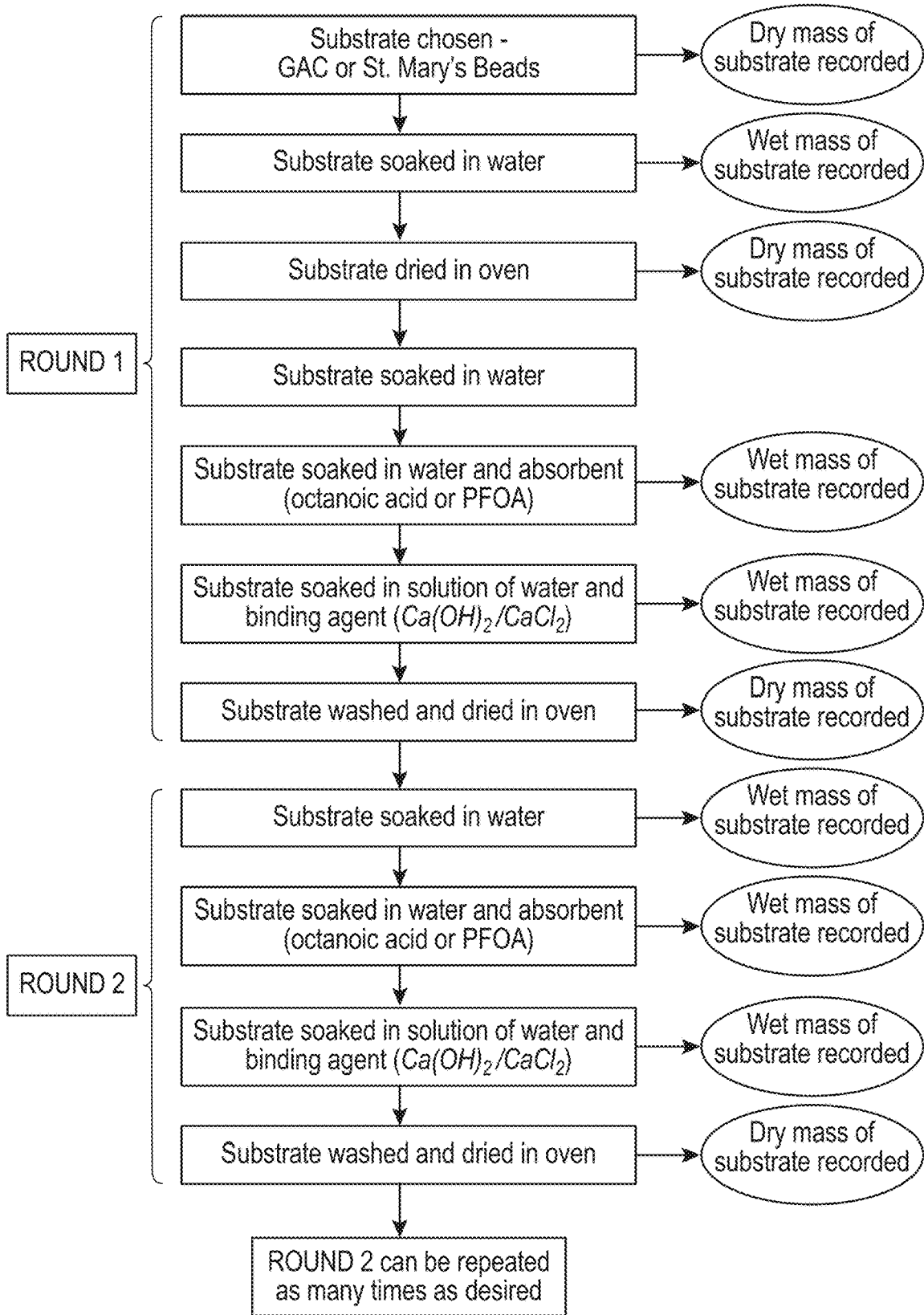


FIG. 4

SM, PFOA, Ca(OH)<sub>2</sub>

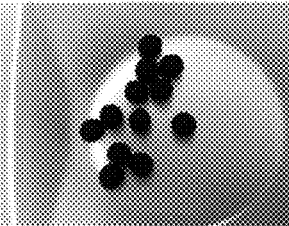
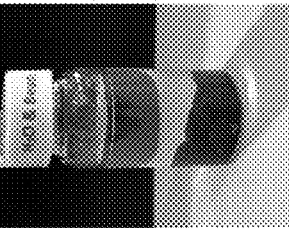
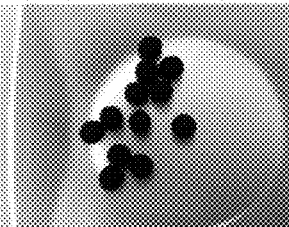
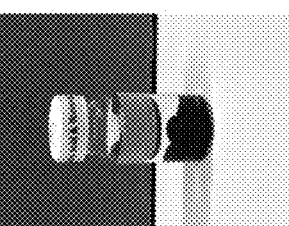
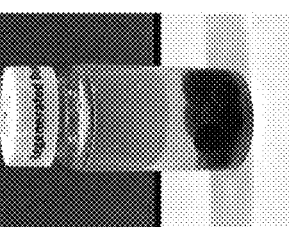
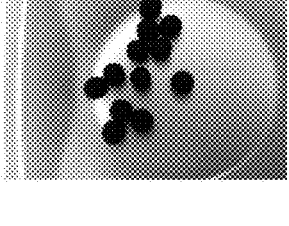
Water Wash		ROUND 1			
St Mary's beads Dry mass recorded		Beads soaked in water Wet mass recorded		Beads dried in oven Dry mass recorded	
		Beads soaked in PFOA & water Wet mass recorded		Beads soaked in Ca(OH) <sub>2</sub> solution Wet mass recorded	
				Beads washed in water and dried in oven Dry mass recorded	

FIG. 5

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## REGENERATABLE SYSTEM FOR CONTAMINANT REMOVAL

### TECHNICAL FIELD

The present invention relates to systems and methods for removing ionic contaminants from an aqueous mixture using a capture bed and for regenerating the capture bed for further use.

### BACKGROUND

This section provides background information related to the present disclosure and is not necessarily prior art.

Water purification technologies are fundamentally important to everyday life. Contaminants must be removed to purify water to an acceptable level in order for the water to be used as drinking water or for other purposes. Per- and polyfluoroalkyl substances (PFAS) are of particular concern and of particular importance to remove from water. Existing technologies for water purification by removal of contaminants, including PFAS, suffer from issues of efficiency and environmental sustainability. For example, technologies that trap contaminants, such as PFAS, in an ion exchange resin or carbon bed currently require replacement of the bed and disposal of the spent bed to a landfill.

Therefore, while there are existing technologies for removing contaminants such as PFAS (and other similar polyfluorinated hydrocarbons) from water, a need continues to exist for improved technologies that provide more effective removal of contaminants and that are regeneratable for continued use without the need for expensive and inefficient replacement of, and environmentally harmful disposal of, system components.

### SUMMARY OF THE INVENTION

In one aspect, disclosed herein is a method of removing a contaminant from an aqueous mixture. The method includes flowing a contaminated aqueous mixture comprising one or more ionic contaminants through a vessel that houses a capture bed and an electrode in electrical contact with the capture bed. The method also includes applying a voltage to the electrode that is in electrical contact with the capture bed, such that the one or more ionic contaminants is bound to the capture bed. The method further includes flowing an aqueous wash liquid through the vessel. The method further includes modulating the voltage applied to the electrode, such that the one or more ionic contaminants bound to the capture bed is released from the capture bed and is washed from the capture bed via the aqueous wash liquid. The aqueous wash liquid may contain a counter ion that binds to the ionic contaminant forming an aggregate contaminant phase that can be removed from the aqueous wash liquid.

In another aspect, disclosed herein is a system for removing a contaminant from water. The system includes a separation vessel and disposed therein a capture bed, an electrode in electrical contact with the capture bed, and a power source electrically coupled to, and configured to apply a voltage to, the electrode that is in electrical contact with the capture bed. The system also includes a controller configured to control and modulate the voltage applied from the power source to the electrode. The system further includes an intake line fluidly coupled to the vessel and configured to introduce a flow of a contaminated aqueous mixture to the vessel such that one or more ionic contaminants in the

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contaminated aqueous mixture binds to the capture bed, and a regeneration line fluidly coupled to the vessel and configured to introduce a flow of aqueous wash liquid to the vessel to wash ionic contaminant from the capture bed.

In another aspect, disclosed herein is a method of regenerating a capture bed. The method includes providing a vessel that houses a capture bed having one or more ionic contaminants bound to the capture bed, and an electrode in electrical contact with the capture bed, and flowing an aqueous wash liquid through the vessel. The method further includes applying a voltage to the electrode, such that the one or more ionic contaminants bound to the capture bed is released from the capture bed and is washed from the capture bed via the aqueous wash liquid. The aqueous wash liquid may contain a counter ion that binds to the ionic contaminant forming an aggregate contaminant phase that can be removed from the aqueous wash liquid.

In another aspect, disclosed herein is a system for regenerating a capture bed. The system includes an electrode in electrical contact with a capture bed housed within a separation vessel, and a power source electrically coupled to, and configured to apply a voltage to the electrode. The system also includes a controller configured to control and modulate the voltage applied from the power source to the electrode. The system further includes a regeneration line fluidly coupled to the separation vessel and configured to introduce a flow of aqueous wash liquid to the separation vessel to wash ionic contaminant from the capture bed.

Other features and advantages of the invention will be apparent from the following detailed description, figures, and from the claims.

### BRIEF DESCRIPTION OF THE DRAWINGS

The following figures are provided by way of example and are not intended to limit the scope of the claimed invention.

FIG. 1 is an illustration of a capture system for removing contaminants from water according to an embodiment of the invention.

FIG. 2A is a schematic of a capture system and FIG. 2B is a schematic of a regeneration system according to another embodiment of the invention.

FIG. 3 is a process diagram of an integrated capture and regeneration system according to another embodiment of the invention.

FIG. 4 is a flow chart showing the steps of Example 1.

FIG. 5 shows the visual appearance of the carbon substrate of Example 1.

### DETAILED DESCRIPTION

#### I. Definitions

The terminology used herein is for the purpose of describing particular exemplary configurations only and is not intended to be limiting. As used herein, the singular articles “a,” “an,” and “the” may be intended to include the plural forms as well, unless the context clearly indicates otherwise. The terms “comprises,” “comprising,” “including,” and “having,” are inclusive and therefore specify the presence of features, steps, operations, elements, and/or components, but do not preclude the presence or addition of one or more other features, steps, operations, elements, components, and/or groups thereof. The method steps, processes, and operations described herein are not to be construed as necessarily requiring their performance in the particular order discussed



or illustrated, unless specifically identified as an order of performance. Additional or alternative steps may be employed.

The terms first, second, third, etc. may be used herein to describe various elements, components, regions, layers and/or sections. These elements, components, regions, layers and/or sections should not be limited by these terms. These terms may be only used to distinguish one element, component, region, layer or section from another region, layer or section. Terms such as "first," "second," and other numerical terms do not imply a sequence or order unless clearly indicated by the context. Thus, a first element, component, region, layer or section discussed below could be termed a second element, component, region, layer or section without departing from the teachings of the example configurations.

The terms, upper, lower, above, beneath, right, left, etc. may be used herein to describe the position of various elements with relation to other elements. These terms represent the position of elements in an example configuration. However, it will be apparent to one skilled in the art that the elements may be rotated in space without departing from the present disclosure and thus, these terms should not be used to limit the scope of the present disclosure.

As used herein, when an element is referred to as being "on," "engaged to," "connected to," "attached to," or "coupled to" another element, it may be directly on, engaged, connected, attached, or coupled to the other element, or intervening elements may be present. In contrast, when an element is referred to as being "directly on," "directly engaged to," "directly connected to," "directly attached to," or "directly coupled to" another element, there may be no intervening elements or layers present. Other words used to describe the relationship between elements should be interpreted in a like fashion (e.g., "between" versus "directly between," "adjacent" versus "directly adjacent," etc.). As used herein, the term "and/or" includes any and all combinations of one or more of the associated listed items.

As used herein, the term "electrode" refers to a solid electric conductor that carries electric current to another element, such as a capture bed.

As used herein, the term "activated carbon" refers to a form of carbon processed to have small pores that increase the available surface area.

As used herein "polyfluoroalkyl ion" refers an ionic compound comprising an alkyl chain with multiple fluoro substitutions, which is optionally further substituted, such as with ether, alcohol, amine (including substituted amine), and carboxylic acid groups.

"Per- and polyfluoroalkyl substance" or "PFAS" includes but is not limited to the following substances: perfluorobutanoic acid, perfluoropentanoic acid, perfluorohexanoic acid, perfluoroheptanoic acid, perfluorooctanoic acid, perfluorononanoic acid, perfluorodecanoic acid, perfluoroundecanoic acid, perfluorododecanoic acid, perfluorotridecanoic acid, perfluorotetradecanoic acid, perfluorohexadecanoic acid, perfluorooctadecanoic acid, perfluorobutanesulfonic acid, perfluoropentanesulfonic acid, perfluorohexanesulfonic acid, perfluorooctanesulfonic acid, perfluorononanesulfonic acid, perfluorodecanesulfonic acid, perfluorododecanesulfonic acid, perfluorooctanesulfonamide, N-methylperfluoro-1-octanesulfonamide, N-ethylperfluoro-1-octanesulfonamide, 1H,1H,2H,2H-perfluorohexanesulfonic acid (4:2), 1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2), 1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2), 1H,1H,2H,2H-perfluorododecanesulfonic acid (10:2), N-methyl perfluorooctanesulfonamidoacetic acid, N-ethyl

perfluorooctanesulfonamidoacetic acid, 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol, 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol, tetrafluoro-2-(heptafluoropropoxy)propanoic acid ("GenX"), 4,8-dioxa-3H-perfluorononanoic acid, 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid, or 9-chlorohexadecafluoro-2-oxanone-1-sulfonic acid. PFAS also includes partial fluorinations. The conjugate bases of these acids are examples of polyfluoroalkyl ions. Capturing PFAS includes capturing a conjugate base of a PFAS.

"PFOS" refers to perfluorooctanesulfonic acid. Capturing/releasing PFOS includes capturing/releasing its conjugate base, perfluorooctanesulfonate.

"PFOA" refers to perfluorooctanoic acid. Capturing/releasing PFOA includes capturing its conjugate base, perfluorooctanoate.

Systems and methods are described herein. It will be understood that embodiments of the invention described with reference to a system may be applicable to the methods described herein, and vice versa. For example, from a description of a particular carbon bed, such as an activated carbon bed, in a system, it will be understood that the activated carbon bed may be used in a method. Likewise, as another example, from a description of application of a particular voltage in a method, it will be understood that the system may be configured to apply the particular voltage.

## II. System for Regenerating a Capture Bed

In one aspect, provided herein is a system for regenerating a capture bed, or a "regeneration system." Regenerating refers to removing ionic contaminant from the capture bed, i.e., contaminant that was bound to the capture bed during a water purification process. Systems and methods for capturing ionic contaminants on a capture bed, and thereby removing them from an aqueous mixture are described herein. As more ionic contaminants are bound to the capture bed the bed becomes less effective at removing the ionic contaminants. Eventually, the contaminants must be released from the capture bed or the capture bed itself must be replaced. Regenerating the capture bed in situ by releasing the bound ionic contaminants allows for continued use of the capture bed without costly replacement and environmentally harmful disposal of the spent capture bed.

The system for regenerating a capture bed includes an electrode in electrical contact with a capture bed that is housed within a separation vessel; a power source electrically coupled to, and configured to apply a voltage to the electrode; and a controller configured to control and modulate the voltage applied from the power source to the electrode. By enabling a voltage to be applied to an electrode in electrical communication with the capture bed, the regeneration system is able to apply voltage to the capture bed that drives the release of ionic contaminant from the capture bed.

In some embodiments, the electrode comprises graphite, titanium, stainless steel, cast iron, a conductive metal oxide, a conductive diamond, a titanium suboxide, titanium nitride, titanium carbide, titanium boride, a doped manganese oxide, or mixtures or composites thereof.

The system for regenerating a capture bed also includes a regeneration line fluidly coupled to the separation vessel and configured to introduce a flow of aqueous wash liquid to the separation vessel to wash ionic contaminant from the capture bed. The application of voltage to the electrode together with flow of wash liquid to the capture bed via the regeneration line drives the release of ionic contaminant from the capture bed, resulting in regeneration of the capture bed for

further use. In some embodiments, the regeneration line is fluidly coupled to a regeneration line pump and/or a regeneration line valve to control the flow of wash liquid supplied to the separation vessel. In some embodiments, the regeneration system includes a flow controller (e.g., a PLC controller) to control the regeneration line pump and/or regeneration line valve.

In some embodiments, the regeneration system is a subsystem of an integrated capture and regeneration system. Such integrated systems are described below. Integrated systems can be installed at a site as a stand-alone system for providing purified water. Alternatively, the regeneration system may be an add-on system to an existing capture system. For example, there are existing systems for water purification with capture beds, e.g., carbon beds or ion exchange resin beds; the regeneration systems described herein may be installed as an add-on system to provide for in situ regeneration of an existing water purification system. In some embodiments, the regeneration system allows for continued use of the capture bed in the existing system by release, sequestration, and removal of the ionic contaminants in the capture bed.

The following embodiments describe an exemplary installation of a regeneration system onto an existing capture system. In order to apply voltage to an existing capture system, the electrodes are installed by insertion into the existing capture bed, and hooked up to the power source controlled by the controller. A regeneration line is fitted onto the existing piping of the capture system (or directly onto the separation vessel) to add separate inlet and outlet flow of wash liquid into the separation vessel. Valves, e.g., control valves, are installed to control and switch the source of flow into the separation vessel between (1) an aqueous mixture to be purified (during a capture cycle) and (2) a wash liquid to regenerate the capture bed.

In some embodiments, the regeneration system includes concentration and removal of the ionic contaminant released from the capture bed. In some embodiments, a contaminant sequestration agent is employed that can more efficiently be removed from the system than removal of the capture bed. In some embodiments, the sequestration agent is more environmentally friendly to dispose of than disposal of a spent carbon bed or spent ion exchange resin bed (i.e., with bound contaminant).

In some embodiments, the sequestration agent is a counter ion in the wash liquid configured to bind to the ionic contaminant to form an aggregate contaminant phase. Suitable sequestration agents and counter ions are described below. In some embodiments, the system further includes a filter configured to remove the aggregate contaminant phase from the wash liquid. Since the aggregate contaminant phase is sparingly soluble to insoluble in the water phase, the precipitate tends to form a distinct solid or liquid phase that is large enough to either float or sink or be captured in a particulate filter. In some embodiments, a skimmer can be used to capture the aggregate contaminant phase.

In some embodiments, the regeneration system further comprises a regeneration vessel that houses a stationary ion source configured to bind the one or more ionic contaminants in the aqueous wash liquid, wherein the regeneration vessel is fluidly coupled to the separation vessel. In some embodiments, the stationary ion source comprises lime, e.g., a plurality of slaked lime pellets. In some embodiments, the stationary ion source is an alkaline metal coated surface where the surface electrostatically or by dispersion forces reversibly holds the alkaline element until a contaminant can

form a precipitate. The contaminant is held at the surface until the surface binding is reversed (e.g., reversing polarity of electrodes).

In some embodiments, the regeneration system further comprises a sequestration agent vessel comprising a sequestration agent in a liquid media. In some embodiments, the regeneration system further comprises a mixing tank for mixing the sequestration agent with the wash liquid and optionally a settler apparatus for collecting solids precipitated from the liquid in the mixing tank. In some embodiments a filter is fluidly coupled to the mixing tank for filtering solids from the mixing tank, for example, solids that were not separated in the settler apparatus.

In some embodiments, the aqueous wash liquid comprises untreated contaminated aqueous mixture. In some embodiments, the aqueous wash liquid comprises a C<sub>1-5</sub> alcohol.

FIG. 2 is a schematic for an exemplary system 200 according to another embodiment of the present invention. FIG. 2A shows a capture system 200a in use for capturing contaminants, specifically PFOA and/or PFOS, from a water source, which is described in detail below. FIG. 2B shows a regeneration system 200b. The regeneration system as schematically shown in FIG. 2B may be part of an integrated capture and regeneration system or may be an add-on regeneration system. A regeneration vessel 220 comprising wash liquid (“container of waste solution”) is fluidly coupled via a regeneration line 222 to the separation vessel 202 that houses the capture bed, specifically a capture bed stack 204 (“cell stack”) and is configured to flow wash liquid through the separation vessel 202. A regeneration outlet line 224 is fluidly coupled to the opposite end of the separation vessel. A valve 218b controls flow out of the separation vessel via the regeneration outlet line 224. The regeneration outlet line 224 is fluidly coupled to the regeneration vessel 220 thus completing the circulation loop. The system 200b is configured to recirculate the wash liquid through the separation vessel 202 multiple times resulting in a wash liquid with high concentration of contaminant (e.g., PFOA/PFOS). The regeneration vessel 220 contains a stationary ion source 226, which are slaked lime pellets as shown in this embodiment. The slaked lime pellets 226 are configured to bind to the PFOA/PFOS in the regeneration vessel 220. Slaked lime pellets 226 can easily be removed from the system for disposal. Disposal of slaked lime pellets is more economical and environmentally friendly than disposal of an activated carbon bed or ion exchange resin bed.

### III. Method for Regenerating a Capture Bed

Another aspect provided herein is a method for regenerating a capture bed, or a “regeneration method.” The method of regenerating a capture bed includes providing a vessel that houses a capture bed having one or more ionic contaminants bound to the capture bed, and an electrode in electrical contact with the capture bed. The vessel may be part of an integrated capture and regeneration system that includes a system for capturing a contaminant, as described below. Alternatively, the vessel may be part of an existing contaminant capture system (water purification system), wherein the regeneration method is performed on the existing vessel/capture system by installing a regeneration system (as described above) onto the existing vessel/capture system.

The method of regenerating a capture bed further includes flowing an aqueous wash liquid through the vessel and applying a voltage to the electrode, such that the one or more

ionic contaminants bound to the capture bed is released from the capture bed and washed from the bed via the aqueous wash liquid.

In some embodiments, the aqueous wash liquid is flowed into the separation vessel at a rate of from about 5 to about 400 liters per minute per square meter of capture bed to release bound ionic contaminant from the capture bed and wash the release ionic contaminant out of the capture bed.

In some embodiments, a voltage having a positive polarity of from about 0.01 V to about 1.5 V (e.g., about 0.01 V to about 1.2 V) is applied to the electrode in order to drive the release of the ionic contaminant from the capture bed to be washed away by the wash liquid. In some embodiments, a voltage having a negative polarity of from about -0.01 V to about -1.6 V is applied to the electrode in order to drive the release of the ionic contaminant. In some embodiments, an AC voltage is applied, optionally with a DC offset, to drive release of the ionic contaminant.

In some embodiments, the wash liquid comprises a sequestration agent. In some embodiments, the sequestration agent is a counter ion. In some embodiments, the counter ion is a cation selected from  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{B}^{3+}$ , or  $\text{Fe}^{3+}$ . Cations are suitable for use in regenerating a capture bed with a bound anionic contaminant, such as perfluoroalkyl anions, or phosphate or borate contaminants. In some embodiments, the counter ion is  $\text{Ca}^{2+}$ . In some embodiments, the counter ion is supplied to the wash liquid by addition of calcium hydroxide, calcium oxide, or calcium chloride to the wash liquid. In some embodiments, the wash liquid is basic and the source of  $\text{Ca}^{2+}$  is calcium hydroxide. In some embodiments, the wash liquid is acidic and the source of  $\text{Ca}^{2+}$  is calcium chloride.

In some embodiments, the pH of the aqueous wash liquid is modulated to cause the aggregate contaminant phase to precipitate from the aqueous wash liquid. For example, lime or other hydroxide can be added to the aqueous wash liquid to change the pH. In some embodiments, the pH is modulated distal to (i.e., downstream of) the capture bed.

In some embodiments, the counter ion is an anion selected from a phosphate, a sulfate, or a borate. Anions are suitable for use in regenerating a capture bed with a bound cationic contaminant, such as perfluoroalkyl cations. In some embodiments the counter ion is supplied to the wash liquid by addition of calcium phosphate, calcium borate, calcium sulphate, magnesium phosphate, magnesium borate, or magnesium sulphate to the wash liquid.

In some cases, perfluoroalkyl compounds may be non-ionic and must first be partially decomposed before they can be released using the counter ion. In such instances, the regeneration method further comprises partially decomposing the nonionic perfluoroalkyl compound(s), such as by chemical, photochemical, electrochemical decomposition or by application of DC or AC electrical discharge.

In some embodiments, upon flowing the wash liquid comprising the sequestration agent through the separation vessel, the sequestration agent and the released ionic contaminant form an aggregate contaminant phase. In some embodiments, the aggregate contaminant phase separates from the aqueous wash liquid by precipitation. In other embodiments, the aggregate contaminant phase forms a foam. In other embodiments, the aggregate contaminant phase forms a dispersed phase within the aqueous wash liquid.

In some embodiments, the aqueous wash liquid is at least substantially saturated with the ionic contaminant upon exiting the capture bed.

In some embodiments, the regeneration method comprises adding a sequestration agent to the wash liquid. In some embodiments, a sequestration agent vessel is provided containing the sequestration agent in liquid media (e.g., aqueous media) and the sequestration agent is flowed from the sequestration agent vessel to be added to the wash liquid. The flow may be controlled by a pump and/or valve. In some embodiments, the sequestration agent is mixed with the wash liquid, for example in a mixing tank. In some embodiments, the wash liquid mixed with the sequestration agent is wash liquid that is substantially saturated with the ionic contaminant.

In some embodiments, the regeneration method further comprises contacting the released ionic contaminant in the aqueous wash liquid with a stationary ion source, such that the ionic contaminant is bound to the stationary ion source forming an aggregate contaminant phase.

In some embodiments, the regeneration method further comprises removal of the aggregate contaminant phase. In some embodiments, removal of the aggregate contaminant phase comprises filtering the aggregate contaminant phase from the wash liquid.

In some embodiments, the regeneration method further comprises disposal of the removed aggregate contaminant phase, e.g., to a landfill. The aggregate contaminant phase may also be destroyed, e.g., by calcination, thermal decomposition, or vitrification.

In some embodiments, the regeneration method further comprises pre-oxidizing the ionic contaminant comprising converting alcohol groups of the ionic contaminant to carboxylic acid groups by chemical or electrochemical means.

In some embodiments, the ionic contaminant comprises an organic end with an ionic moiety. In some embodiments, the ionic contaminant is selected from the group consisting of a polyfluoroalkyl ion, a borate, a phosphate, a polyphosphate, a sulfate, an organic acid, a fatty acid, a humic substance, a shortchain PFAS, a water-soluble medication, a detergent, a water-soluble insecticide, a water-soluble fungicide, a water-soluble germicide, and any combination thereof. In some embodiments, the ionic contaminant is a polyfluoroalkyl ion. In some embodiments, the polyfluoroalkyl ion is perfluorooctanesulfonate or perfluorooctanoate. Perfluorooctanesulfonate is the conjugate base of perfluorooctanesulfonic acid (PFOS). Perfluorooctanoate is the conjugate base of perfluorooctanoic acid (PFOA). In some embodiments, the polyfluoroalkyl ion is perfluorobutanesulfonate or perfluorobutanoate. Perfluorobutanesulfonate is the conjugate base of perfluorobutanesulfonic acid (PFBS). Perfluorobutanoate is the conjugate base of perfluorobutanoic acid (PFBA).

The system of FIG. 2B can also be described in terms of a regeneration method of which it illustrates. Wash liquid ("waste solution") is flowed to a separation vessel that houses a capture bed ("cell stack") and flows through the vessel. Ionic contaminants bound to the cell stack are released as wash liquid flows through the vessel and voltage is applied (not shown) to the cell stack. A valve ("waste water valve") is opened to direct flow of wash liquid with released ionic contaminant (PFOA and/or PFOS) to a regeneration vessel ("container of waste solution") via a regeneration outlet line. A stationary ion source (slaked lime pellets in this embodiment) binds the PFOA/PFOS. The slaked lime pellets with bound PFOA/PFOS can be removed from the system and disposed of.

#### IV. System for Capturing a Contaminant

Another aspect provided herein is a system for capturing an ionic contaminant, or a "capture system." The capture

system includes a separation vessel that houses a capture bed configured to capture ionic contaminants in an aqueous mixture flowed through the separation vessel. In some embodiments, the system also provides for regeneration of the capture bed as an integrated capture and regeneration system, or an “integrated system.” The integrated system may include any of the features of a regeneration system and/or capture system as described herein.

The capture system includes: a separation vessel and disposed therein a capture bed; an electrode in electrical contact with the capture bed; a power source electrically coupled to, and configured to apply a voltage to, the electrode that is in electrical contact with the capture bed; a controller configured to control and modulate the voltage applied from the power source to the electrode; and an intake line fluidly coupled to the vessel and configured to introduce a flow of a contaminated aqueous mixture to the vessel such that one or more ionic contaminants in the contaminated aqueous mixture binds to the capture bed.

The integrated capture and regeneration system includes a capture system and further includes a regeneration line fluidly coupled to the vessel and configured to introduce a flow of aqueous wash liquid to the vessel to wash ionic contaminant from the capture bed.

In some embodiments, the capture system further includes a pump fluidly coupled to the intake line and configured to pump the contaminated aqueous mixture into the separation vessel. In some embodiments, the capture system further includes a valve fluidly coupled to the intake line and configured to control the flow of the contaminated aqueous mixture into the separation vessel.

In some embodiments, the controller is configured to reduce or reverse the current applied from the power source. In some embodiments, the controller is further configured to reduce the voltage applied to the electrode, reverse the polarity of the voltage applied to the electrode, terminate the voltage applied to the electrode, or any combination thereof. As described in the methods below, the power source is configured to apply a first voltage to the electrode during flow of contaminated aqueous mixture to capture bed (during a capture cycle). During flow of wash liquid to the capture bed (during a regeneration cycle), terminating, reducing or reversing the current drives the release of the bound contaminant from the capture bed.

In some embodiments, the capture bed (e.g., activated carbon bed) is surface-modified with functional groups selected from the group consisting of an acid, a hydroxide, a chloride, a bromide, a fluoride, an ether, an epoxide, a quinone, a ketone, an aldehyde, a pyrrole, a thiophene, and any combination thereof.

In some embodiments, the capture bed is at least partially conductive. In some embodiments, the capture bed is porous. In some embodiments, the capture bed is an activated carbon bed. In some embodiments, the capture bed is an ion exchange resin bed. In some embodiments, the capture bed is a composite of activated carbon and ion exchange resin. In some embodiments, the capture bed is an activated carbon metal oxide composite. In some embodiments, the capture bed is a FILTRASORB® activated carbon bed from Calgon Carbon.

In some embodiments, the capture bed comprises powder, granules, beads, pellets, cloths, felts, nonwoven fabrics, or composites comprising a material selected from carbon, nitrogen-doped carbon, boron-doped carbon, charcoal, graphite, biochar, coke, carbon black, or any combination

thereof. In some embodiments, the capture bed comprises activated charcoal powder, granules, pellets, beads, or any combination thereof.

In some embodiments, the capture bed comprises activated carbon having an average surface area of from about 100 m<sup>2</sup>/g to about 2000 m<sup>2</sup>/g. In some embodiments, the capture bed has a conductivity of from about 0.01 S/cm to about 100 S/cm. In some embodiments, the capture bed has a porosity of from about 30% to about 95%.

In some embodiments, the capture bed is surface-modified with functional groups selected from the group consisting of an acid, a hydroxide, a chloride, a bromide, a fluoride, an ether, an epoxide, a quinone, a ketone, an aldehyde, a pyrrole, a thiophene, and any combination thereof. In some embodiments, the capture bed has an ionic complexing species bound to it. In some embodiments, the ionic complexing species is Ca<sup>2+</sup>, Mg<sup>2+</sup>, phosphate, borate, or silicate. In some embodiments, the ionic complexing species is an alkaline ion mixed with fatty acid or wax.

In some embodiments, the capture bed further comprises a binder dispersed in the capture bed. In some embodiments, the binder comprises a wax, a starch, a sugar, a polysaccharide, or any combination thereof. In some embodiments, the wax is a polyethylene wax. In some embodiments, the wax is carnauba wax.

In some embodiments, the capture bed is disposed longitudinally along the flow axis of the separation vessel such that the contaminated aqueous mixture flows by the capture bed. In other embodiments, the capture bed is disposed laterally across the separation vessel such that the water flows through the capture bed.

In some embodiments, the capture bed is adjacent to a separator. In some embodiments, the capture bed is wrapped in a separator, enclosed within a separator, or sandwiched between two separators.

In some embodiments, the capture system further comprises a second separation vessel that houses a second capture bed and a second electrode in electrical contact with the second capture bed. In some embodiments, the power source or a second power source is configured to apply a voltage to the second electrode that is in electrical contact with the second capture bed.

In some embodiments, the separation vessel further houses a second capture bed and a second electrode in electrical contact with the second capture bed. In some embodiments, the second capture bed is adjacent to the first capture bed with a separator disposed between the first and second capture beds. In some embodiments, the separator is disposed around the first and second capture beds in a Z-fold, S-fold, or C-fold arrangement. In some embodiments, the separator is disposed around one or more capture beds in a spiral wound or jelly roll configuration. In some embodiments, the power source is configured to apply a positive voltage to one of the first and second capture beds, and a negative voltage to the other of the first and second capture beds.

In some embodiments, the separation vessel comprises a stack comprising a plurality of capture beds. In some embodiments, the plurality of capture beds in the stack are separated from each other by one or more separators. In some embodiments, the plurality of capture beds are in electrical contact with the first or second electrode.

In some embodiments, the power source is configured to apply a positive voltage to the first electrode, wherein the first electrode is in electrical contact with a first plurality of capture beds, and wherein the power source is configured to

apply a negative voltage to the second electrode, wherein the second electrode is in electrical contact with a second plurality of capture beds.

In some embodiments, the first plurality of capture beds are stacked in an alternating fashion with the second plurality of capture beds.

In some embodiments, the vessel is a pipe, column, or tank.

In some embodiments, the separator comprises a porous plastic. In some embodiments, the porous plastic is a plastic mesh. In some embodiments, the separator comprises an inert material. Suitable materials for the separator include nylon, polyamide, polypropylene, and HDPE.

FIG. 1 illustrates an exemplary capture system according to an embodiment of the present invention. In this embodiment, a separation vessel or column 102 (e.g., PVC pipe) houses a stack 104 of carbon powder capture beds 104a-d. The stack 104 is arranged with each carbon powder capture bed 104a-d wrapped in a non-woven separator 106a-d. Stacks can be added, and the column 102 lengthened, to fit the desired amount of carbon. The stack 104 is configured with the wrapped carbon powder capture beds 104a-d disposed laterally across the vessel 102 such that flow through the vessel 102 will flow through each capture bed 104a-d of the stack 104. Electrodes 108 (or “current collectors”) made of graphite filled polymer are in electrical contact with the carbon powder capture beds 104a-d. The electrodes 108 are electrically coupled to a power source 150. A first electrode 108a is inserted longitudinally through the stack 104 and is in electrical contact with a first 104a and third 104c capture bed of the stack, but is electrically insulated from a second 104b and fourth 104d capture bed of the stack. Non-conductive tape 110 is wrapped around a portion of the first electrode 108a in its electrically insulated areas in the second and fourth capture bed. A second electrode 108b is inserted longitudinally (and separated from the first electrode 108a) through the stack 104 and is in electrical contact with the second 104b and fourth 104d capture bed of the stack, but is electrically insulated from the first 104a and third 104c capture bed. Non-conductive tape 110 is wrapped around a portion of the second electrode 108b in its electrically insulated areas in the first 104a and third 104c capture bed. An intake line 112 is fluidly coupled to a first end of the separation vessel 102 and an outlet line 116 is fluidly coupled to a second end of the separation vessel 102. The inlet line 112 includes an inlet valve 114. The outlet line 116 includes an outlet valve 118.

FIG. 2 is a schematic for exemplary system 200 according to another embodiment of the present invention. FIG. 2A shows a capture system 200a in use for capturing contaminants, specifically PFOA or PFOS, from a water source. An intake line 212 is fluidly coupled to a vessel 202 that houses a cell stack 204 and configured to supply water in need of treatment due to high concentration of PFOA and/or PFOS (e.g., having levels PFOA and/or PFOS above the upper limit as defined by EPA or other regulatory body) into the vessel 202. The cell stack 204 comprises capture beds in a stack, optionally with separator between the beds. An outlet has valves 218a, 218b for a clean water outlet and a waste water (i.e. wash liquid) outlet. FIG. 2B shows a regeneration system 200b as described above. The regeneration system of FIG. 2B may be installed together with the system of FIG. 2A as part of an integrated system.

FIG. 3 is a process diagram for an exemplary integrated system 300 according to another embodiment of the present invention. In this embodiment, a raw water tank 308 contains the contaminated aqueous mixture in need of ionic

contaminant removal. The raw water tank 308 is fluidly coupled to a pump 314 (“pump 1”) with an intervening valve 312 to control flow. Pump 1 314 is fluidly coupled to the inlet 316 of a separation vessel 302 (e.g., an activated carbon EDI filtration column) that houses activated carbon capture beds 304. A voltage/current source 306 is electrically coupled to the capture beds 304 of the separation vessel 302. The outlet 318 of the column 302 is fluidly coupled to a clean water tank 324 with an intervening valve 322. The intervening valve 322 is also fluidly coupled to the raw water tank 308 via recirculation line 326 providing for optional recirculation of the liquid for one or more additional cycles of contaminant removal. The raw water tank 308 is also fluidly coupled to another pump 330 (“pump 2”) via the same intervening valve 312 that controls flow into pump 1 314. Pump 2 330 is fluidly coupled to a second separation vessel 332 (an activated graphite EDI concentrating column) that houses an activated graphite capture bed 334. A voltage/current 336 source is electrically coupled to the activated graphite bed 334. The second separation vessel 332 is fluidly coupled to the clean water tank 308 and to the recirculation valve 322.

Still referring to FIG. 3, a regeneration vessel 328 (waste water tank) containing wash liquid is fluidly coupled to both Pump 1 314 and Pump 2 330 and both separation vessels 302, 332. The wash liquid upon flowing through the capture beds 304 and/or 334 can be referred to as extract and is fluidly coupled to a precipitator 338. The precipitator 338 is also fluidly coupled to a regeneration fluid tank 340 via another pump 342 (“Pump 3”). The regeneration fluid tank 340 contains a counter ion in a liquid media. The precipitator 338 is fluidly coupled to a settler 344 for removing precipitated solids from the extract upon mixing with the counter ion. The settler 344 is also fluidly coupled to a filter 346 for further removal of solids from the liquid phase exiting the settler. The system 300 can be controlled by a PLC controller 348.

## V. Method for Capturing a Contaminant

Another aspect provided herein is a method for capturing an ionic contaminant, or a “capture method.” The capture method includes flowing an aqueous mixture comprising one or more ionic contaminants through a separation vessel that houses a capture bed in order to bind the one or more ionic contaminants to the capture bed, thereby removing the one or more ionic contaminants from the aqueous mixture. In some embodiments, the method includes regeneration of the capture bed as part of an integrated capture and regeneration method, or an “integrated method.”

The capture method further includes applying a voltage to the electrode that is in electrical contact with the capture bed, such that the one or more ionic contaminants is bound to the capture bed. The applied voltage enhances the binding of the one or more ionic contaminants to the capture bed.

The integrated method further includes a regeneration cycle comprising flowing an aqueous wash liquid through the separation vessel and modulating the voltage applied to the electrode, such that the one or more ionic contaminants bound to the capture bed is released from the capture bed and is washed from the capture bed via the aqueous wash liquid. The modulated voltage drives the release of the bound ionic contaminant from the capture bed. The integrated method, specifically the regeneration cycle thereof, may include any of the steps and features of the regeneration method described above.

In some embodiments, applying the voltage to the electrode comprises running an electrical current to the electrode, and modulating the voltage comprises reducing or reversing the electrical current running to the electrode. In some embodiments, the voltage applied to the electrode during capture of contaminants has a positive polarity from about 0.01 V to about 1.6 V. In some embodiments, modulating the voltage to release the ionic contaminant comprises reducing the electric current to generate a modulated voltage having a positive polarity of from about 0.01 V to about 1.5 V (e.g., about 0.01 V to about 1.2 V). In some embodiments, modulating the voltage to release the ionic contaminant comprises reversing the electric current to generate a modulated voltage having a negative polarity of from about -0.01 V to about -1.6 V. In some embodiments, modulating the voltage to release the ionic contaminant comprises applying an AC voltage optionally with a DC offset.

In some embodiments, the contaminated aqueous mixture is flowed into the vessel at a rate from about 5 to about 400 liters per minute per square meter of capture bed. In some embodiments, the contaminated aqueous mixture is flowed into the vessel at a rate from about 80 to about 240 liters per minute per square meter of capture bed. In some embodiments, the contaminated aqueous mixture is flowed into the vessel at a rate from about 0.01 to about 10 liters per minute per kilogram of capture bed. In some embodiments, the capture bed has a mass of from about 4,000 to about 10,000 kilograms. In some embodiments, the pressure drop across the capture bed is from about 1 psi to about 200 psi.

In some embodiments, the aqueous wash liquid is flowed into the vessel at a rate from about 5 to about 400 liters per minute per square meter of capture bed. In some embodiments, the aqueous wash liquid is flowed into the vessel at a rate from about 80 to about 240 liters per minute per square meter of capture bed. In some embodiments, the aqueous wash liquid is flowed into the vessel at a rate from about 0.01 to about 10 liters per minute per kilogram of capture bed.

In some embodiments, the capture method further comprises binding an ionic complexing species to the capture bed prior to flowing the contaminated aqueous mixture through the vessel, such that upon flowing the contaminated aqueous mixture through the vessel, the ionic contaminant binds to the capture bed by forming a complex with the ionic complexing species wherein the complex is bound to the capture bed. In some embodiments, the ionic complexing species is  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , phosphate, borate, or silicate. In some embodiments, the ionic complexing species is an alkaline ion mixed with fatty acid or wax.

In some embodiments, the capture bed is situated in the vessel such that the contaminated aqueous mixture flows by the capture bed. In some embodiments, the capture bed is situated in the vessel such that the contaminated aqueous mixture flows through the capture bed.

In some embodiments, the capture method further includes flowing the contaminated aqueous mixture through a second vessel that houses a second capture bed and a second electrode in electrical contact with the second capture bed and applying a voltage to the second electrode that is in electrical contact with the second capture bed.

In some embodiments, the vessel further houses a second capture bed and a second electrode in electrical contact with the second capture bed and the capture method further includes applying a voltage to the second electrode that is in electrical contact with the second capture bed. In some embodiments, the second capture bed is adjacent to the first capture bed with a separator disposed between the first and second capture beds. In some embodiments, a positive

voltage is applied to one of the first and second capture beds, and a negative voltage is applied to the other of the first and second capture beds.

In some embodiments, the vessel comprises a capture bed stack comprising a plurality of capture beds. In some embodiments, the plurality of capture beds are separated from each other by one or more separators. In some embodiments, the plurality of capture beds are in electrical contact with the first or second electrode. In some embodiments, the capture method further comprises applying a positive voltage to the first electrode, wherein the first electrode is in electrical contact with a first plurality of capture beds; and applying a negative voltage to the second electrode, wherein the second electrode is in electrical contact with a second plurality of capture beds. In some embodiments, the first plurality of capture beds are stacked in an alternating fashion with the second plurality of capture beds.

In some embodiments, the capture method further comprises surface-modifying the capture bed with a functional group selected from the group consisting of an acid, a hydroxide, a chloride, a bromide, a fluoride, an ether, an epoxide, a quinone, a ketone, an aldehyde, a pyrrole, a thiophene, and any combination thereof.

In some embodiments, the ionic contaminant comprises an organic end with an ionic moiety. In some embodiments, the ionic contaminant is selected from the group consisting of a polyfluoroalkyl ion, a borate, a phosphate, a polyphosphate, a sulfate, an organic acid, a fatty acid, a humic substance, a shortchain PFAS, a water-soluble medication, a detergent, a water-soluble insecticide, a water-soluble fungicide, a water-soluble germicide, and any combination thereof. In some embodiments, the ionic contaminant is a polyfluoroalkyl ion. In some embodiments, the polyfluoroalkyl ion is perfluorooctanesulfonate or perfluorooctanoate.

The system of FIG. 2A can also be described in terms of a capture method of which it illustrates. Water comprising a high concentration of PFOA and/or PFOS contaminant is flowed into a separation vessel that houses a plurality of capture beds in a stack ("cell stack"). As the water flows through the cell stack the PFOA and/or PFOS ionic contaminant is bound to the capture bed. With the bound contaminant removed from the flow of water through the separation vessel, water with a low concentration of PFOA and/or PFOS flows out of the separation vessel via an outlet line. Flow through the outlet line is controlled by a valve, which is open during the capture cycle ("system in use"). A second outlet line for use during a regeneration cycle is closed.

The system of FIG. 3 can also be described in terms of an integrated capture and regeneration method. During a capture cycle of the integrated method contaminated water from the raw water tank flows into the two separation vessels (activated carbon EDI filtration column and activated graphite EDI concentrating column). This flow a valve and a pump for each separation vessel as shown. Voltage is applied to the capture beds in each separation vessel. The water with contaminant removed by the capture beds is flowed to a clean water tank, which flow is controlled by further valves. Optionally the outlet flow of the separation vessels may be recirculated, as controlled by a recirculation valve, to the raw water tank for additional cycle(s) of purification.

Still referring to FIG. 3, during a regeneration cycle, wash liquid from a waste water tank is flowed through the separation vessels and also controlled using the same valves and pumps as during the capture cycle. The voltage is modulated during the regeneration cycle to drive release of the bound contaminant from the capture bed as the wash

liquid flows through the separation vessel. The wash liquid with released contaminant (“extract”) is flowed to a precipitator where it is mixed with regeneration fluid that is pumped into the precipitator. The regeneration fluid comprises a sequestration agent (e.g., a counter ion) that forms an aggregate contaminant phase with the released contaminant. The aggregate contaminant phase precipitates from the wash liquid in the precipitator and the solids are collected in a settler and removed. Wash liquid exiting the precipitator/settler is filtered and returned for continued use in washing the capture beds.

VI. Examples

Example 1: Regeneration of Carbon Substrate

An experiment was carried out to test the regeneration of carbon substrate using calcium hydroxide and calcium chloride. The experiment tested the capture, regeneration, and sequestration of (i) octanoic acid and (ii) PFOA on carbon substrate using calcium hydroxide or calcium chloride as sequestration agent. Octanoic acid is a simulant of PFAS, which has similar behavior in water but not the disposal issues of actual PFAS. Visual indications and mass measurements were used to determine the results of the experiment.

FIG. 4 shows a flowchart of the testing method. A first carbon substrate was selected, either granulated activated carbon (GAC) or St Mary’s carbon beads. The dry mass of the substrate was recorded. The substrate was next soaked in water and the wet mass recorded. The substrate was then dried in an oven and the mass after drying recorded. The substrate was next soaked in water followed by soaking overnight in a mixture of water and a specified and recorded mass of contaminant—either octanoic acid (OA) or PFOA. The wet mass of the substrate was then recorded to determine capture of OA/PFOA. The substrate was next soaked overnight in a solution of water and a specified and recorded mass of sequestration agent—either Ca(OH)<sub>2</sub> or CaCl<sub>2</sub>. NaOH was also added in the case of CaCl<sub>2</sub>. After soaking and washing, the wet mass of the substrate was recorded. The substrate was then dried in oven and the dry mass after drying was recorded. The sequestration agent solution was dried and the mass of precipitate from the solution was recorded.

The same basic procedure can then be repeated for a second and subsequent rounds of capture, regeneration and sequestration with mass measurements. Second and third rounds were completed for the present experiment. The dried substrate from the final step of Round 1 was soaked in water and the wet mass of substrate was recorded. Next, the substrate was soaked overnight in water and a contaminant—either octanoic acid (OA) or PFOA. The wet mass of the substrate was then recorded to determine capture of OA/PFOA. The substrate was next soaked in solution of water and a sequestration agent—either Ca(OH)<sub>2</sub> or CaCl<sub>2</sub>. NaOH was also added in the case of CaCl<sub>2</sub>. After soaking and washing, the wet mass of the substrate was recorded. The substrate was then dried in oven and the dry mass after drying was recorded. The sequestration agent solution was dried and the mass of precipitate from the solution was recorded.

Tables 1-4 show the results of the mass measurements for the experiment over the course of the initial wash procedure and three rounds of capture, regeneration and sequestration. Table 5 shows the recovered mass of precipitate recovered for each round. FIG. 5 shows visual observation of St. Mary’s Beads over the course of the first round of testing.

The results demonstrated that the carbon substrates were able to releasably capture OA and PFOA, and that the sequestration agent (calcium hydroxide or calcium chloride) was able to bind the OA or PFOA, be released from the carbon substrate, and be sequestered as a precipitate. The process was repeatable, showing the ability to capture OA and PFOA on the carbon substrate and subsequently regenerate the carbon substrate for multiple uses.

TABLE 1

Water Wash.				
Vial Group	Fresh Beads Dry Mass (g)	Wet with water mass (g)	Post Water Dry mass (g)	Water wash delta mass (g)
SM, PFOA, Ca(OH) <sub>2</sub>	2.5545	3.3140	Not taken	NA
SM, OA, Ca(OH) <sub>2</sub>	2.5425	3.3125	2.5459	0.0034
SM, PFOA, CaCl <sub>2</sub>	2.5320	3.3114	2.5340	0.0020
GAC, PFOA, Ca(OH) <sub>2</sub>	0.5209	1.2054	0.4976	-0.0233
GAC, PFOA, CaCl <sub>2</sub>	0.5295	1.4055	0.5026	-0.0269
GAC, OA, CaCl <sub>2</sub>	0.5220	1.3529	0.4930	-0.0290

TABLE 2

First Round of Capture/Release							
Vial Group	Mass of contaminant (OA/PFOA) (g)	Wet mass of beads after OA/PFOA (g)	Mass of sequestrant Ca(OH) <sub>2</sub> /CaCl <sub>2</sub> (g)	Wet mass after regen 1 (g)	Dry mass after regen 1 (g)	First regen dry delta mass (g)	Bead/GAC mass regeneration (%)
SM, PFOA, Ca(OH) <sub>2</sub>	0.0963	3.3308	0.0075	3.3345	2.5641	0.0096	99.6242
SM, OA, Ca(OH) <sub>2</sub>	0.1203	3.3151	0.0344	3.3209	2.5470	0.0011	99.9568
SM, PFOA, CaCl <sub>2</sub>	0.0581	3.3319	0.0089	3.3315	2.5419	0.0079	99.6882
GAC, PFOA, Ca(OH) <sub>2</sub>	0.0582	1.2376	0.0085	1.2052	0.5272	0.0296	94.0514
GAC, PFOA, CaCl <sub>2</sub>	0.0528	1.4704	0.0120	1.4067	0.5391	0.0365	92.7378
GAC, OA, CaCl <sub>2</sub>	0.1258	1.3942	0.0603	1.3618	0.5756	0.0826	83.2454

TABLE 3

Second Round of Capture/Release								
Vial Group	Wet mass after soaking in water (g)	Mass of contaminant (OA/PFOA) (g)	Wet mass of beads after OA/PFOA (g)	Mass of sequestrant (Ca(OH) <sub>2</sub> /CaCl <sub>2</sub> ) (g)	Wet mass after regen 2 (g)	Dry mass after regen 2 (g)	Second regen dry delta mass (g)	Bead/GAC mass regeneration (%)
SM, PFOA, Ca(OH) <sub>2</sub>	3.3306	0.0596	3.3390	0.0079	3.3360	2.5548	0.0003	99.9883
SM, OA, Ca(OH) <sub>2</sub>	3.2067	0.1204	3.3017	0.0388	3.3209	2.5571	0.0112	99.5601
SM, PFOA, CaCl <sub>2</sub>	3.3194	0.0501	3.3291	0.0231	3.3289	2.5350	0.0010	99.9605
GAC, PFOA, Ca(OH) <sub>2</sub>	1.1961	0.0510	1.2529	0.0133	1.2077	0.5281	0.0305	93.8706
GAC, PFOA, CaCl <sub>2</sub>	1.3947	0.0514	1.4315	0.0360	1.3988	0.5650	0.0624	87.5846
GAC, OA, CaCl <sub>2</sub>	1.3270	0.1202	1.3587	0.0726	1.3762	0.5529	0.0599	87.8499

TABLE 4

Third Round of Capture/Release								
Vial Group	Wet mass after soaking in water (g)	Mass of contaminant (OA/PFOA) (g)	Wet mass of beads after OA/PFOA (g)	Mass of sequestrant (Ca(OH) <sub>2</sub> /CaCl <sub>2</sub> ) (g)	Wet mass after regen 3 (g)	Dry mass after regen 3 (g)	Third regen dry delta mass (g)	Bead/GAC mass regeneration (%)
SM, PFOA, Ca(OH) <sub>2</sub>	3.3190	0.0543	3.3317	0.0102	3.3303	2.5559	0.0014	99.9452
SM, OA, Ca(OH) <sub>2</sub>	3.2392	0.1116	3.3133	0.0403	3.3130	2.5459	0.0034	99.8665
SM, PFOA, CaCl <sub>2</sub>	3.3181	0.0531	3.3310	0.0286	3.3310	2.5376	0.0056	99.7790
GAC, PFOA, Ca(OH) <sub>2</sub>	1.1806	0.0511	1.2250	0.0151	1.2189	0.5461	0.0252	94.9357
GAC, PFOA, CaCl <sub>2</sub>	1.4195	0.0522	1.4574	0.0391	1.4366	0.5961	0.0666	86.7489
GAC, OA, CaCl <sub>2</sub>	1.2767	0.1272	1.3020	0.0811	1.2835	0.5781	0.0561	88.6207

TABLE 5

Recovered Mass of Precipitate										
Vial Group	Regeneration Precipitate	ROUND 1			ROUND 2			ROUND 3		
		Expected mass of precipitate (g)	Actual mass of precipitate (g)	Recovered Yield (%)	Expected mass of precipitate (g)	Actual mass of precipitate (g)	Recovered Yield (%)	Expected mass of precipitate (g)	Actual mass of precipitate (g)	Recovered Yield (%)
SM, PFOA, Ca(OH) <sub>2</sub>	Calcium perfluoro-octanoate	0.088	0.073	83.66	0.064	0.053	83.49	0.057	0.050	87.50
SM, OA, Ca(OH) <sub>2</sub>	Calcium octanoate	0.136	0.106	77.94	0.136	0.130	95.60	0.126	0.122	96.28
SM, PFOA, CaCl <sub>2</sub>	Calcium perfluoro-octanoate	0.061	0.019	30.43	0.054	0.028	53.08	0.056	0.050	88.29
GAC, PFOA, Ca(OH) <sub>2</sub>	Calcium perfluoro-octanoate	0.061	0.052	86.04	0.055	0.035	63.67	0.053	0.038	70.22
GAC, PFOA, CaCl <sub>2</sub>	Calcium perfluoro-octanoate	0.055	0.039	71.38	0.055	0.038	68.31	0.055	0.048	87.36
GAC, OA, CaCl <sub>2</sub>	Calcium octanoate	0.142	0.072	50.21	0.136	0.128	93.83	0.140	0.106	73.89

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## Other Embodiments

It is to be understood that while the invention has been described in conjunction with the detailed description thereof, the foregoing description is intended to illustrate and not limit the scope of the invention, which is defined by the scope of the appended claims. Other aspects, advantages, and modifications are within the scope of the following claims.

What is claimed is:

1. A method of removing a contaminant from an aqueous mixture comprising:

a) flowing a contaminated aqueous mixture comprising one or more ionic contaminants through a vessel that houses a capture bed stack comprising a plurality of capture beds, a first electrode inserted through each one of the capture beds, and a second electrode inserted through each one of the capture beds; wherein the plurality of capture beds are separated from each other by one or more separators, and wherein each one of the plurality of capture beds is in electrical contact with either the first electrode or the second electrode, and wherein the first electrode is in electrical contact with a first plurality of capture beds but is electrically

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- insulated from a second plurality of capture beds, and wherein the second electrode is in electrical contact with the second plurality of capture beds but is electrically insulated from the first plurality of capture beds;
- b) applying a voltage to the first electrode and to the second electrode, such that the one or more ionic contaminants is bound to the plurality of capture beds;
- c) flowing an aqueous wash liquid through the vessel; and
- d) modulating the voltage applied to the first electrode and to the second electrode, such that the one or more ionic contaminants bound to the plurality of capture beds is released from the plurality of capture beds and is washed from the plurality of capture beds via the aqueous wash liquid.
2. The method of claim 1, wherein the aqueous wash liquid comprises a counter ion that binds to the ionic contaminant forming an aggregate contaminant phase, and wherein the method further comprises removing the aggregate contaminant phase from the aqueous wash liquid.
3. The method of claim 2, wherein the counter ion is a cation selected from  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{B}^{3+}$ , or  $\text{Fe}^{3+}$ ; or wherein the counter ion is an anion selected from a phosphate, a sulfate, or a borate.
4. The method of claim 2, wherein the aggregate contaminant phase separates from the aqueous wash liquid by precipitation.
5. The method of claim 1, further comprising contacting the released ionic contaminant in the aqueous wash liquid with a stationary ion source, such that the ionic contaminant is bound to the stationary ion source and is thereby removed from the aqueous wash liquid.
6. The method of claim 1, wherein applying the voltage to the first electrode and to the second electrode comprises running an electrical current to each of the first electrode and the second electrode, and modulating the voltage applied to the first electrode and to the second electrode comprises reducing or reversing the electrical current running to the first electrode and to the second electrode.
7. The method of claim 6, wherein modulating the voltage applied to the first electrode and to the second electrode to release the ionic contaminant comprises reducing the electrical current to generate a modulated voltage having a positive polarity of from about 0.01 V to about 1.5 V.
8. The method of claim 6, wherein modulating the voltage applied to the first electrode and to the second electrode to release the ionic contaminant comprises reversing the electrical current to generate a modulated voltage having a negative polarity of from about -0.01 V to about -1.6 V.
9. The method of claim 6, wherein modulating the voltage applied to the first electrode and to the second electrode to release the ionic contaminant comprises applying an AC voltage optionally with a DC offset.
10. The method of claim 1, wherein the contaminated aqueous mixture is flowed into the vessel at a rate from about 5 to about 400 liters per minute per square meter of capture bed in the plurality of capture beds.
11. The method of claim 1, wherein the voltage applied to the first electrode and to the second electrode has a positive polarity from about 0.01 V to about 1.6 V.
12. The method of claim 1, wherein the pressure drop across the first plurality of capture beds and the second plurality of capture beds is from about 1 psi to about 200 psi.
13. The method of claim 1, wherein the aqueous wash liquid is flowed into the vessel at a rate of from about 5 to about 400 liters per minute per square meter of capture bed in the plurality of capture beds.

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14. The method of claim 1, wherein the aqueous wash liquid is at least substantially saturated with the ionic contaminant upon exiting the plurality of capture beds.
15. The method of claim 1, further comprising binding an ionic complexing species to each capture bed of the plurality of capture beds prior to flowing the contaminated aqueous mixture through the vessel, such that upon flowing the contaminated aqueous mixture through the vessel, the ionic contaminant binds to each capture bed of the plurality of capture beds by forming a complex with the ionic complexing species wherein the complex is bound to each capture bed of the plurality of capture beds.
16. The method of claim 15, wherein the ionic complexing species is  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , phosphate, or borate.
17. The method of claim 1, wherein each capture bed of the plurality of capture beds is situated in the vessel such that the contaminated aqueous mixture flows by or through each capture bed of the plurality of capture beds.
18. The method of claim 1, wherein each capture bed of the plurality of capture beds is adjacent to a separator.
19. The method of claim 18, wherein each capture bed of the plurality of capture beds is wrapped in a separator, enclosed within a separator, or sandwiched between two separators.
20. The method of claim 18, wherein the separator comprises a porous plastic.
21. The method of claim 1, further comprising flowing the contaminated aqueous mixture through a second vessel that houses a third capture bed and a third electrode in electrical contact with the third capture bed and applying a voltage to the third electrode that is in electrical contact with the third capture bed.
22. The method of claim 1, comprising applying a positive voltage to the first electrode; and applying a negative voltage to the second electrode.
23. The method of claim 22, wherein the first plurality of capture beds are stacked in an alternating fashion with the second plurality of capture beds.
24. The method of claim 1, wherein the ionic contaminant comprises an organic end with an ionic moiety.
25. The method of claim 1, wherein the ionic contaminant is selected from the group consisting of a polyfluoroalkyl ion, a borate, a phosphate, a polyphosphate, a sulfate, an organic acid, a fatty acid, a humic substance, a shortchain PFAS, a water-soluble medication, a detergent, a water-soluble insecticide, a water-soluble fungicide, a water-soluble germicide, and any combination thereof.
26. The method of claim 25, wherein the ionic contaminant is a polyfluoroalkyl ion.
27. The method of claim 26, wherein the polyfluoroalkyl ion is perfluorooctanesulfonate or perfluorooctanoate.
28. The method of claim 1, wherein each capture bed of the plurality of capture beds is at least partially conductive.
29. The method of claim 28, wherein each capture bed of the plurality of capture beds is an activated carbon bed.
30. The method of claim 28, wherein each capture bed of the plurality of capture beds is an ion exchange resin bed.
31. The method of claim 1, wherein each capture bed of the plurality of capture beds comprises powder, granules, beads, pellets, cloths, felts, nonwoven fabrics, or composites comprising a material selected from carbon, nitrogen-doped carbon, boron-doped carbon, charcoal, graphite, biochar, coke, carbon black, or any combination thereof.
32. The method of claim 31, wherein each capture bed of the plurality of capture beds comprises activated charcoal powder, granules, pellets, beads, or any combination thereof.

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33. The method of claim 1, wherein each capture bed of the plurality of capture beds comprises activated carbon having an average surface area of from about 100 m<sup>2</sup>/g to about 2000 m<sup>2</sup>/g.

34. The method of claim 1, wherein each capture bed of the plurality of capture beds has a conductivity of from about 0.01 S/cm to about 100 S/cm.

35. The method of claim 1, wherein each capture bed of the plurality of capture beds is surface-modified with functional groups selected from the group consisting of an acid, a hydroxide, a chloride, a bromide, a fluoride, an ether, an epoxide, a quinone, a ketone, an aldehyde, a pyrrole, a thiophene, and any combination thereof.

36. The method of claim 1, wherein each capture bed of the plurality of capture beds has a porosity of from about 30% to about 95%.

37. The method of claim 1, wherein each capture bed of the plurality of capture beds further comprises a binder dispersed in each capture bed of the plurality of capture beds.

38. The method of claim 37, wherein the binder comprises a wax, a starch, a sugar, a polysaccharide, or any combination thereof.

39. The method of claim 1, wherein the vessel is a pipe, column, or tank.

40. The method of claim 1, wherein each of the first electrode and the second electrode comprises graphite, titanium, stainless steel, cast iron, a conductive metal oxide, a conductive diamond, a titanium suboxide, titanium nitride, titanium carbide, titanium boride, a doped manganese oxide, or mixtures or composites thereof.

41. The method of claim 1, wherein the aqueous wash liquid comprises untreated contaminated aqueous mixture.

42. The method of claim 1, wherein the aqueous wash liquid comprises a C<sub>1-5</sub> alcohol.

43. A method of regenerating a capture bed stack comprising:

- a) providing a vessel that houses a capture bed stack comprising a plurality of capture beds having one or more ionic contaminants bound to the capture beds, a first electrode inserted through each one of the capture

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beds, and a second electrode inserted through each one of the capture beds; wherein the plurality of capture beds are separated from each other by one or more separators, and wherein each one of the plurality of capture beds is in electrical contact with either the first electrode or the second electrode, and wherein the first electrode is in electrical contact with a first plurality of capture beds of the plurality of capture beds but is electrically insulated from a second plurality of capture beds of the plurality of capture beds, and wherein the second electrode is in electrical contact with the second plurality of capture beds but is electrically insulated from the first plurality of capture beds;

- b) flowing an aqueous wash liquid through the vessel; and
- c) applying a voltage to the first electrode and to the second electrode, such that the one or more ionic contaminants bound to the plurality of capture beds is released from the plurality of capture beds and is washed from the plurality of capture beds via the aqueous wash liquid.

44. The method of claim 43, wherein the aqueous wash liquid comprises a counter ion that binds to the ionic contaminant forming an aggregate contaminant phase that separates from the aqueous wash liquid.

45. The method of claim 44, further comprising removing the aggregate contaminant phase from the aqueous wash liquid.

46. The method of claim 44, wherein the aggregate contaminant phase separates from the aqueous wash liquid by precipitation.

47. The method of claim 46, further comprising modulating the pH of the aqueous wash liquid to cause the aggregate contaminant phase to precipitate from the aqueous wash liquid.

48. The method of claim 43, further comprising contacting the released ionic contaminant in the aqueous wash liquid with a stationary ion source, such that the ionic contaminant is bound to the stationary ion source and is thereby removed from the aqueous wash liquid.

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