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(54) **SYSTEM AND METHOD FOR SILVER-NANOPARTICLE-COATED MEMBRANES**

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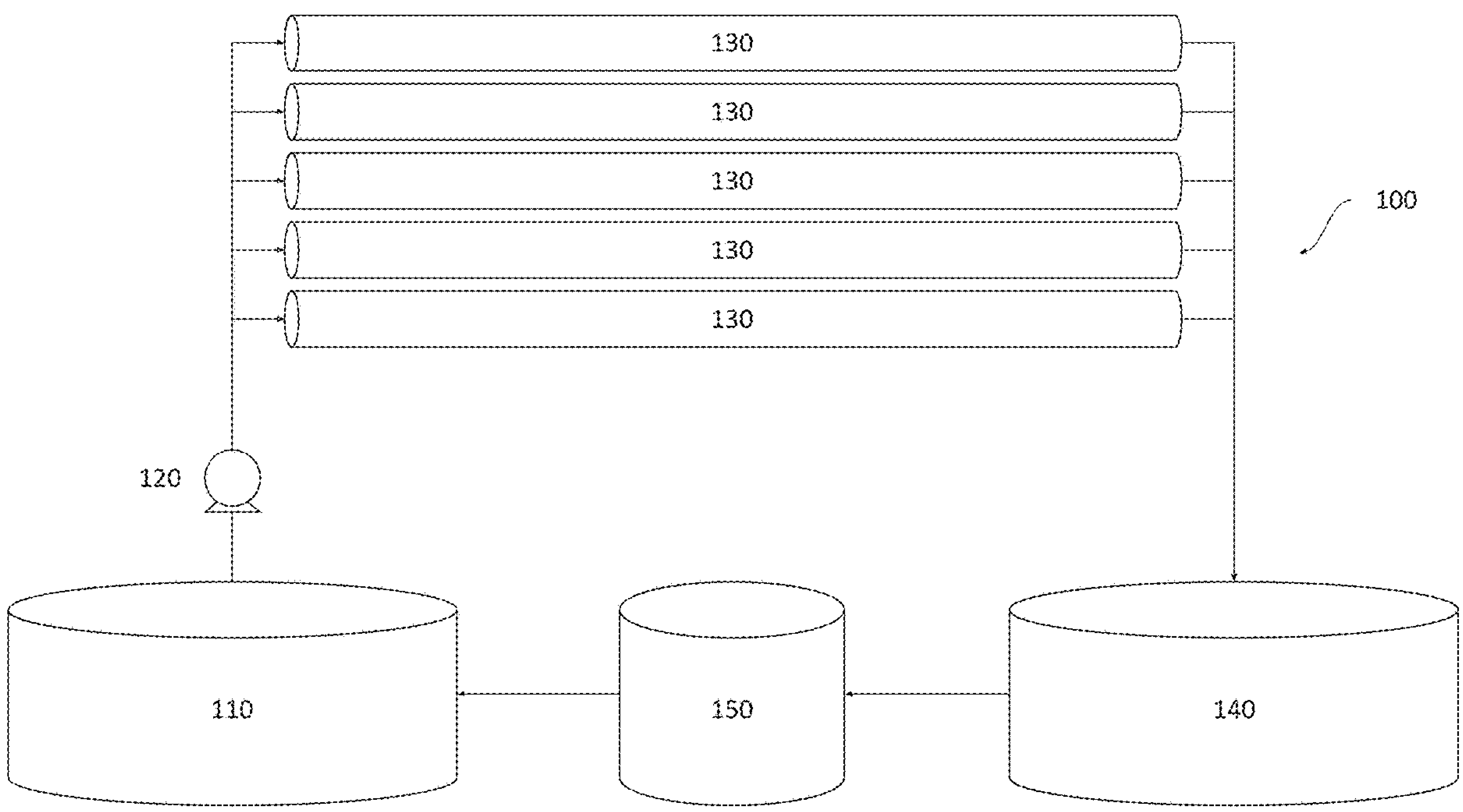
(57) **ABSTRACT**

The present invention provides among other things a silver nanoparticle-coated membrane that reduces biofouling. The membranes exhibit extended antimicrobial properties that prevents the buildup that causes biofouling, which in turn prolongs the life of the membranes. The membranes may be reverse osmosis membranes, microfiltration membranes, ultrafiltration membranes, nanofiltration membranes, or any other membrane suitable for liquid processes.



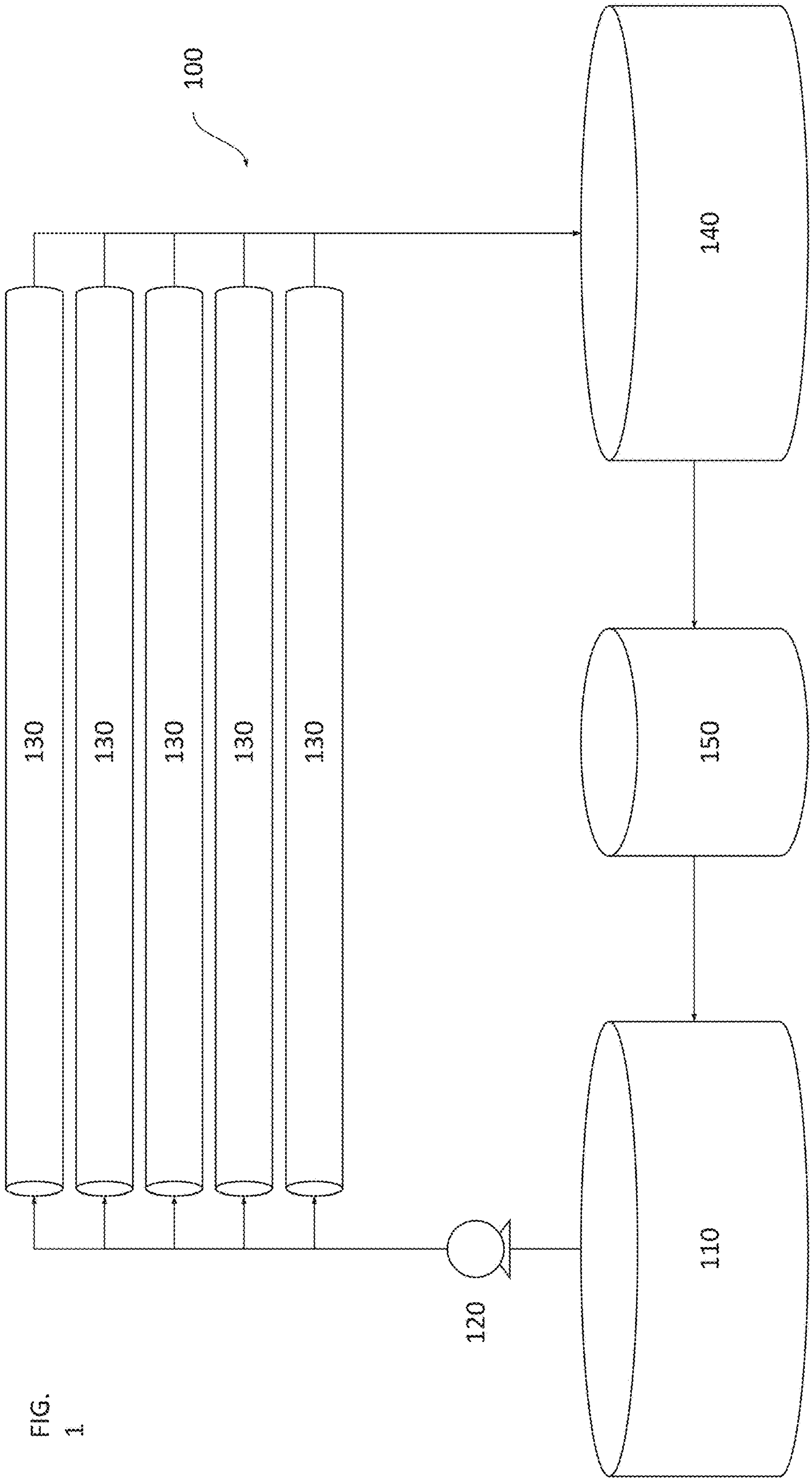


FIG. 1

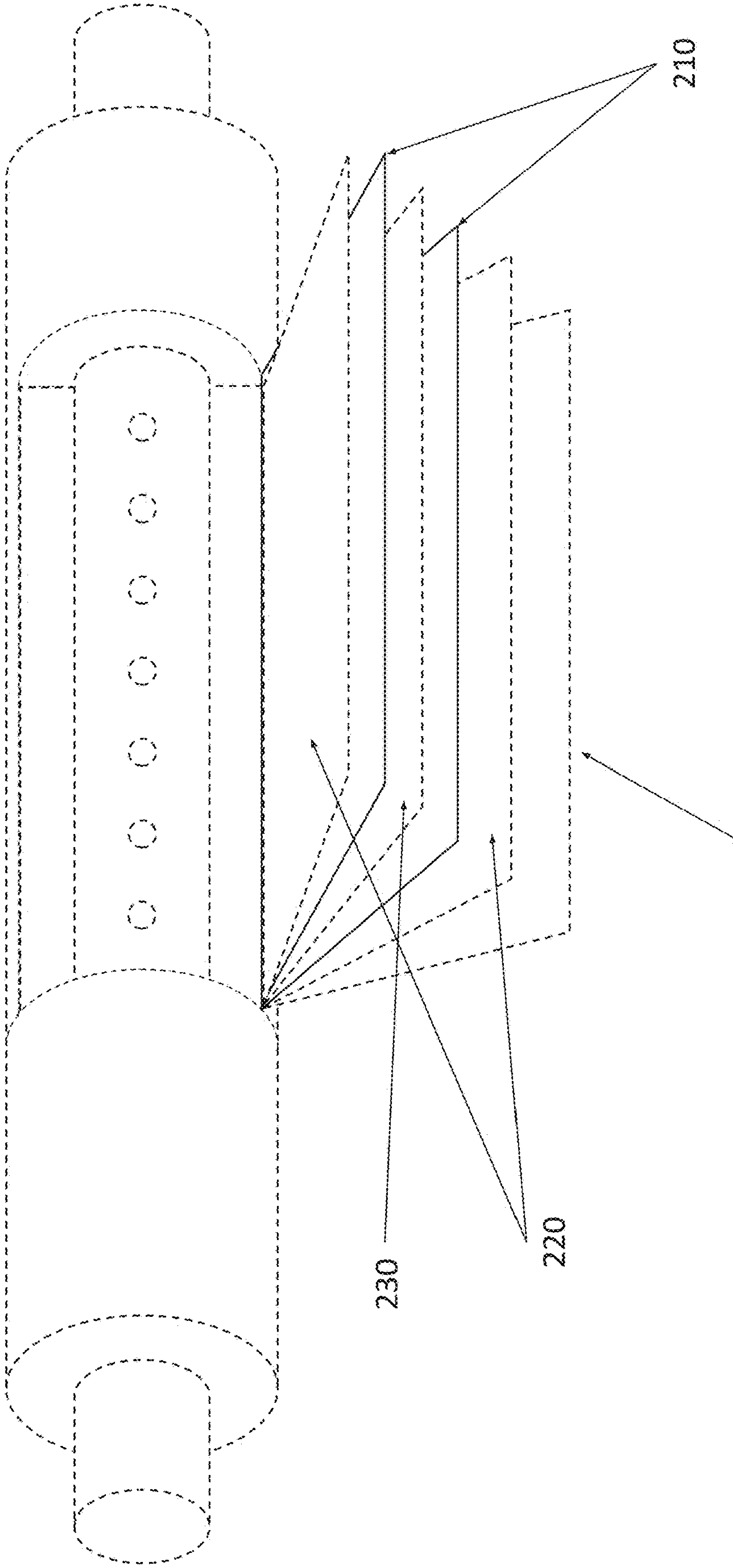


FIG. 2

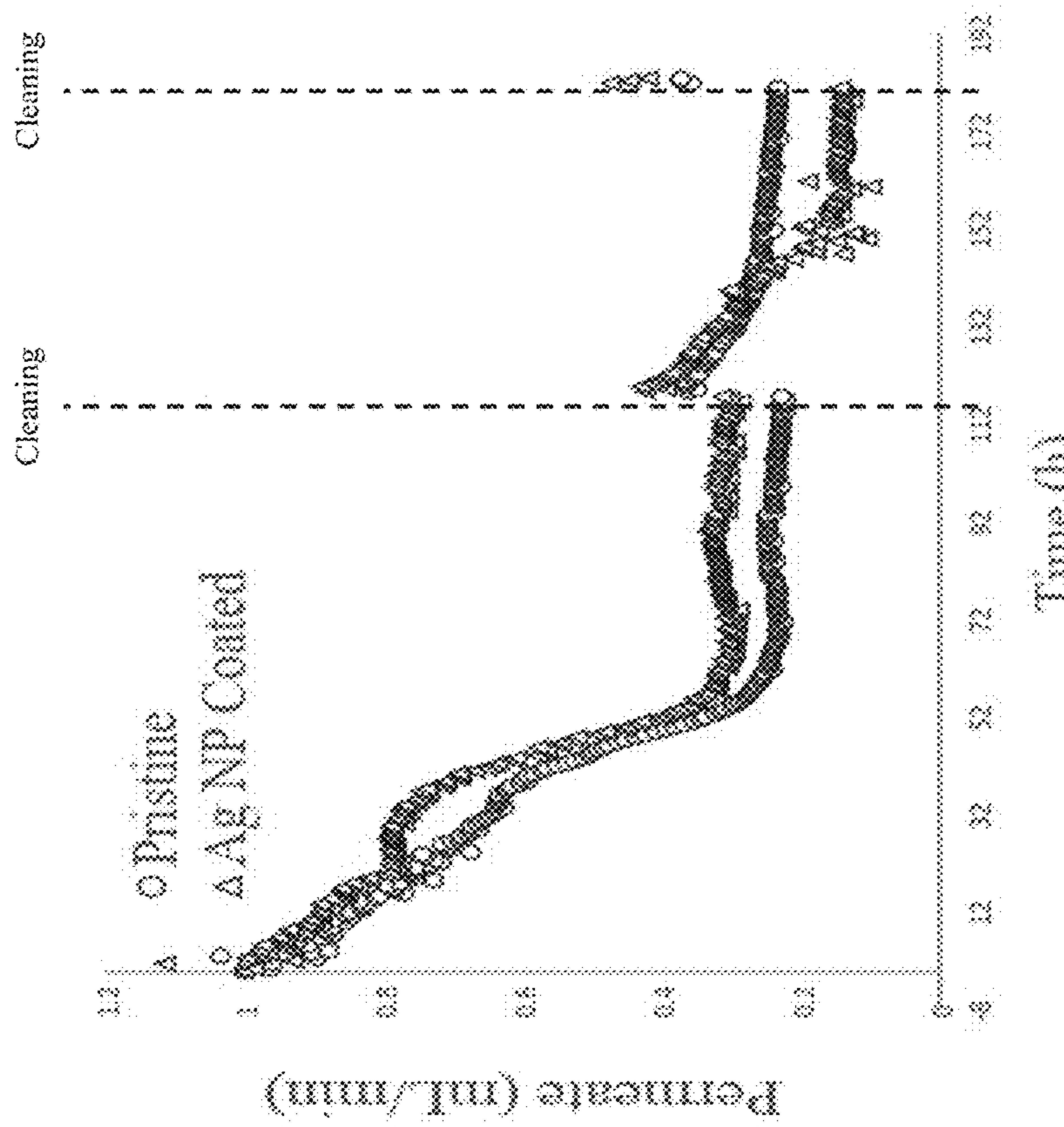

Cleaning
Cleaning
○ Pristine
Δ Ag NP Coated
Permeate (mL/min)
Time (h)

FIG. 3B

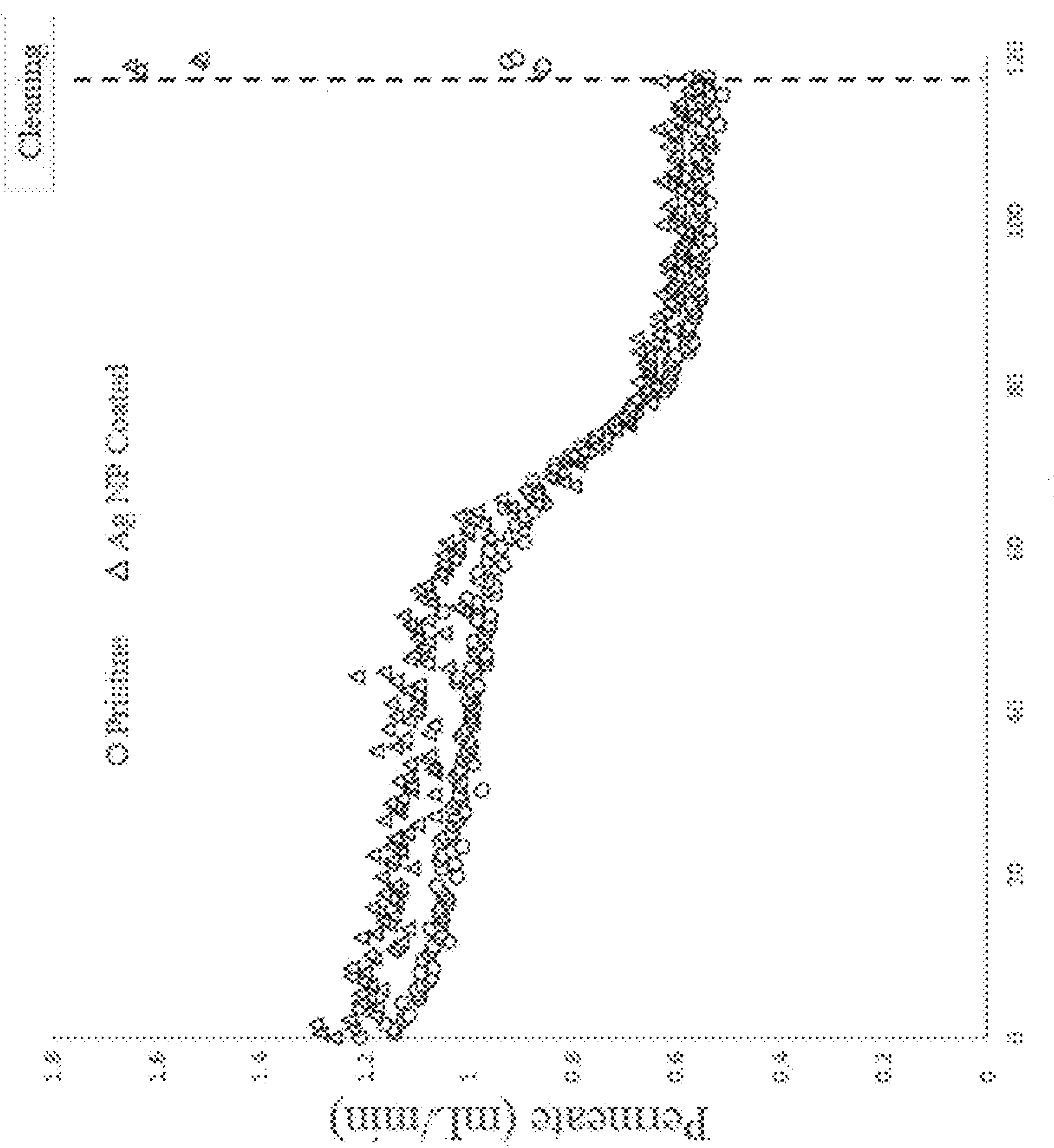

Cleaning
○ Pristine
Δ Ag NP Coated
Permeate (mL/min)
Time (h)

FIG. 3A

SYSTEM AND METHOD FOR SILVER-NANOPARTICLE-COATED MEMBRANES

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] The present application is a continuation-in-part application to PCT Application No. PCT/US21/35410, filed on Jun. 2, 2021, entitled "Passivated Silver Nanoparticle Coatings and Methods of Making the Same," which claims priority to U.S. Provisional Application No. 63/035,355, filed Jun. 5, 2020, entitled "Passivated Silver Nanoparticle Coatings and Methods of Making the Same," each of which are incorporated by reference herein in its entirety.

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH

[0002] This invention was made with government support under 1449500 awarded by the National Science Foundation and 80NSSC19C0566 awarded by the National Aeronautical and Space Administration. The government has certain rights in the invention.

BACKGROUND

1. Field of the Invention

[0003] The present disclosure relates generally to silver-nanoparticle-coated membranes and methods of making and using such membranes.

2. Description of Related Art

[0004] Silver is used as a biocide for disinfection and biofouling control in a variety of applications, including water treatment, medical supplies, medical devices, textiles, and the like. However, antimicrobial coatings containing silver nanoparticles have high leaching rates, leading to only short-term effectiveness, which increases both overall cost and the risk of biofilm development. One such application of silver nanoparticle coatings is membranes. Membranes experience biofouling which decreases lifespan. Membranes coated with silver nanoparticles have reduced biofouling, although the extent of which depends on the leaching rates of the silver nanoparticles. There has been recent exploration of in situ coatings of silver nanoparticles with a silver sulfide core-shell structure to decrease leaching rates. However, this process requires modification to be able to coat membranes. Thus, there exists a need for a method and apparatus to form silver nanoparticles with low leaching rates on a membrane.

SUMMARY

[0005] The present invention provides among other things a silver nanoparticle-coated membrane that reduces biofouling. The membranes exhibit extended antimicrobial properties that prevents the buildup that causes biofouling, which in turn prolongs the life of the membranes. The membranes may be reverse osmosis membranes, microfiltration membranes, ultrafiltration membranes, nanofiltration membranes, or any other membrane suitable for liquid processes.

[0006] In some embodiments, an antimicrobial membrane may comprise a membrane and a plurality of silver nanoparticles coupled to the membrane, wherein the plurality of silver nanoparticles have a silver sulfide core-shell structure. In some embodiments, the membrane may be a reverse osmosis membrane. In some embodiments, the membrane may be a microfiltration membrane. In some embodiments, the membrane may be an ultrafiltration membrane. In some embodiments, the membrane may be a nanofiltration membrane. In some embodiments, the plurality of silver nanoparticles may have a weight percentage of sulfur between approximately 0.1% and approximately 7%. In some embodiments, the plurality of silver nanoparticles may have a diameter of between approximately 3 nanometers (nm) and approximately 350 nm.

[0007] In some embodiments, a method for coating a membrane may comprise providing a membrane coating apparatus that may comprise a feed tank, a pump coupled to the feed tank, a membrane housing comprising a feed end, concentrate end and a permeate end, wherein the feed end is coupled to the pump, a return tank coupled to the concentrate end of the membrane housing and a plugged and unplugged permeate end and a dosing unit coupled to the return tank and the feed tank, circulating a silver nitrate solution through the membrane coating apparatus, circulating a nucleating agent solution through the membrane coating apparatus, and circulating a solution of sodium nitrate and a sulfidation agent through the membrane coating apparatus. In some embodiments, the nucleating agent may be selected from the group consisting of sodium borohydride, hydrazine, D-glucose, hyaluronic acid, and combinations thereof. In some embodiments, the nucleating agent may be sodium borohydride. In some embodiments, the sulfidation agent may be selected from the group consisting of sodium sulfide, sodium thiosulfate, thiocarbamide, thioacetamide, and combinations thereof. In some embodiments, the sulfidation agent may be sodium sulfide. In some embodiments, the silver nitrate solution may have a concentration from approximately 1 millimolar (mM) to approximately 300 mM. In some embodiments, the nucleating agent solution may have a concentration from approximately 1 mM to approximately 300 mM. In some embodiments, the sodium nitrate may have a concentration from approximately 10^{-5} M to 10^{-1} M. In some embodiments, the sulfidation agent may have a concentration from approximately 10^{-5} M to 10^{-1} M. In some embodiments, the silver nitrate solution may be cycled for a time period of at least approximately 5 minutes. In some embodiments, the nucleating agent solution may be cycled for a time period of at least approximately 2 minutes. In some embodiments, the solution of sodium nitrate and the sulfidation agent may be cycled for a time period of at least approximately 0.5 hours.

[0008] In some embodiments, a method for coating a membrane with silver nanoparticles may comprise exposing a membrane to a silver nitrate solution, exposing the membrane to a nucleating agent solution, and exposing the membrane to a solution of sodium nitrate and a sulfidation agent. In some embodiments, the nucleating agent may be selected from the group consisting of sodium borohydride, hydrazine, D-glucose, hyaluronic acid, and combinations thereof. In some embodiments, the nucleating agent may be sodium borohydride. In some embodiments, the sulfidation agent may be selected from the group consisting of sodium sulfide, sodium thiosulfate, thiocarbamide, thioacetamide, and combinations thereof. In some embodiments, the sulfidation agent may be sodium sulfide. In some embodiments, the silver nitrate solution may have a concentration from approximately 1 mM to approximately 300 mM. In some embodiments, the nucleating agent solution may have a concentration from approximately 1 mM to approximately 300 mM. In some embodiments, the sodium nitrate may have a concentration from approximately 10^{-5} M to 10^{-1} M. In some embodiments, the sulfidation agent may have a concentration from approximately 10^{-5} M to 10^{-1} M. In some embodiments, the membrane may be exposed to the silver nitrate solution for a time period of at least approximately 5 minutes. In some embodiments, the membrane may be exposed to the nucleating agent solution for a time period of at least approximately 2 minutes. In some embodiments, the membrane may be exposed to the solution of sodium nitrate and the sulfidation agent for a time period of at least approximately 0.5 hours.

[0009] In various embodiments, a method for coating a membrane with silver nanoparticles may comprise providing a membrane coating apparatus that may comprise a feed tank, a pump coupled to the feed tank, a membrane housing comprising a feed end, a concentrate end and a permeate end, wherein the feed end is coupled to the pump, a return tank coupled to the concentrate end of the membrane housing, and a plugged or unplugged permeate end and a dosing unit coupled to the return tank and the feed tank, circulating a silver nitrate solution through the membrane coating apparatus, circulating a D-glucose solution through the membrane coating apparatus, and circulating a solution of sodium nitrate and sodium sulfide through the membrane coating apparatus. In some embodiments, the silver nitrate solution may have a concentration of approximately 3 millimolar (mM). In some embodiments, the D-glucose solution may have a concentration of approximately 3 mM. In some embodiments, the sodium nitrate may have a concentration of approximately 10 mM. In some embodiments, the sodium sulfide may have a concentration of approximately 10 mM. In some embodiments, the silver nitrate solution may be cycled for a time period of at least approximately 5 minutes. In some embodiments, the D-glucose solution may be cycled for a time period of at least approximately 24 hours. In some embodiments, the solution of sodium nitrate and sodium sulfide may be cycled for a time period of at least approximately 0.5 hour.

[0010] In various embodiments, a method for coating a membrane with silver nanoparticles may comprise exposing a membrane to a silver nitrate solution, exposing the membrane to a sodium borohydride solution, and exposing the membrane to a solution of sodium nitrate and sodium sulfide. In some embodiments, the silver nitrate solution may have a concentration of approximately 3 millimolar (mM). In some embodiments, the sodium borohydride solution may have a concentration of approximately 3 mM. In some embodiments, the sodium nitrate may have a concentration of approximately 10 mM. In some embodiments, the sulfidation agent may have a concentration of approximately 10 mM. In some embodiments, the membrane may be exposed to the silver nitrate solution for a time period from approximately 5 minutes to approximately 15 minutes. In some embodiments, the membrane may be exposed to the sodium borohydride solution for a time period from approximately 2 minutes to approximately 10 minutes. In some embodiments, the membrane may be exposed to the solution of sodium nitrate and the sulfidation agent for a time period from approximately 0.5 hours to approximately 24 hours.

[0011] Aspects and applications of the invention presented here are described below in the drawings and detailed description of the invention. Unless specifically noted, it is intended that the words and phrases in the specification and the claims be given their plain, ordinary, and accustomed meaning to those of ordinary skill in the applicable arts. The inventor is fully aware that he can be his own lexicographer if desired. The inventor expressly elects, as his own lexicographers, to use only the plain and ordinary meaning of terms in the specification and claims unless he clearly states otherwise and then further, expressly sets forth the "special" definition of that term and explains how it differs from the plain and ordinary meaning. Absent such clear statements of intent to apply a "special" definition, it is the inventor's intent and desire that the simple, plain and ordinary meaning to the terms be applied to the interpretation of the specification and claims.

[0012] The inventor is also aware of the normal precepts of English grammar. Thus, if a noun, term, or phrase is intended to be further characterized, specified, or narrowed in some way, then such noun, term, or phrase will expressly include additional adjectives, descriptive terms, or other modifiers in accordance with the normal precepts of English grammar. Absent the use of such adjectives, descriptive terms, or modifiers, it is the intent that such nouns, terms, or phrases be given their plain, and ordinary English meaning to those skilled in the applicable arts as set forth above.

[0013] Further, the inventor is fully informed of the standards and application of the special provisions of 35 U.S.C. § 112(f). Thus, the use of the words "function," "means" or "step" in the Detailed Description or Description of the Drawings or claims is not intended to somehow indicate a desire to invoke the special provisions of 35 U.S.C. § 112(f), to define the invention. To the contrary, if the provisions of 35 U.S.C. § 112(f) are sought to be invoked to define the inventions, the claims will specifically and expressly state the exact phrases "means for" or "step for, and will also recite the word "function" (i.e., will state "means for performing the function of [insert function]"), without also reciting in such phrases any structure, material or act in support of the function. Thus, even when the claims recite a "means for performing the function of . . . " or "step for performing the function of . . . ," if the claims also recite any structure, material or acts in support of that means or step, or that perform the recited function, then it is the clear intention of the inventor not to invoke the provisions of 35 U.S.C. § 112(f). Moreover, even if the provisions of 35 U.S.C. § 112(f) are invoked to define the claimed inventions, it is intended that the inventions not be limited only to the specific structure, material or acts that are described in the preferred embodiments, but in addition, include any and all structures, materials or acts that perform the claimed function as described in alternative embodiments or forms of the invention, or that are well known present or later-developed, equivalent structures, material or acts for performing the claimed function.

[0014] The foregoing and other aspects, features, and advantages will be apparent to those artisans of ordinary skill in the art from the DETAILED DESCRIPTION and DRAWINGS, and from the CLAIMS.

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

[0015] A more complete understanding of the present invention may be derived by referring to the detailed description when considered in connection with the following illustrative figures. In the figures, like reference numbers refer to like elements or acts throughout the figures.

[0016] FIG. **1** depicts an exemplary membrane-coating apparatus.

[0017] FIG. **2** depicts an exemplary membrane housing cross-section.

[0018] FIGS. **3**A-**3**B depict graphs of permeate flowrate for a coated membrane and a non-coated membrane.

[0019] Elements and acts in the figures are illustrated for simplicity and have not necessarily been rendered according to any particular sequence or embodiment.

DETAILED DESCRIPTION

[0020] In the following description, and for the purposes of explanation, numerous specific details are set forth in order to provide a thorough understanding of the various aspects of the invention. It will be understood, however, by those skilled in the relevant arts, that the present invention may be practiced without these specific details. In other instances, known structures and devices are shown or discussed more generally in order to avoid obscuring the invention. In many cases, a description of the operation is sufficient to enable one to implement the various forms of the invention, particularly when the operation is to be implemented in software. It should be noted that there are many different and alternative configurations, devices and technologies to which the disclosed inventions may be applied. The full scope of the inventions is not limited to the examples that are described below.

[0021] In one application, a membrane may be coated with silver nanoparticles (Ag NPs) to reduce biofouling. The membrane may be a reverse osmosis membrane, a microfiltration membrane, an ultrafiltration membrane, a nanofiltration membrane, or any other membrane suitable for liquid processes. In some embodiments, the solutions may be circulated through a membrane coating apparatus **100** to allow for the exposure to the membrane as illustrated in FIG. **1**. In some embodiments, the membrane coating apparatus **100** may include a feed tank **110** coupled to a pump **120**. The pump **120** may transfer a solution from the feed tank to a membrane housing **130**. The membrane housing may contain the membrane that is to be coated with the nanoparticles. The membrane housing may be configured for cross flow or dead-end filtration. In some embodiments, there may be multiple membrane housings **130**. When there are multiple membrane housings **130**, the membrane housings **130** may run in series or in parallel. In some embodiments, the membrane housing **130** may feed into a return tank **140**. In other embodiments, the membrane housing **130** may feed back into the feed tank **110**. In some embodiments, the return tank **140** may feed into a dosing unit **150** to adjust a solution's concentration before returning to the feed tank **110**. In other embodiments, the return tank **140** may feed into the feed tank **110** without adjusting concentration. Parameters such as the pressure, flow rate, and temperature may vary depending on the equipment specification and the requirements of the specific membrane that is to be coated.

[0022] FIG. **2** illustrates the membrane in an exemplary reverse osmosis membrane element **200** that may be placed inside the membrane housing **130**. The membrane layers **210** are positioned between feed channel spacers **220**. In between the membrane layers is a permeate collection layer **230**. An outer wrap layer **240** is positioned outside of the outer feed channel spacer **220**.

EXAMPLES

Example 1: Coating a Membrane with Non-Sulfidated Silver Nanoparticles Using Sodium Borohydride

[0023] Set the pump to have a feed pressure of approximately 8 psi and a flow rate of approximately 945 mL/min. Circulate approximately 1 L of a 3 mM silver nitrate solution through the apparatus for approximately 10 minutes, then drain the silver nitrate solution from the apparatus. Circulate approximately 1 L of a 3 mM sodium borohydride (nucleating agent) solution through the apparatus for approximately 5 minutes or until the solution becomes dark, then drain the sodium borohydride solution from the apparatus.

Example 2: Coating a Membrane with Sulfidated Silver Nanoparticles Using Sodium Borohydride

[0024] Set the pump to have a feed pressure of approximately 8 psi and a flow rate of approximately 945 mL/min. Circulate approximately 1 L of a 3 mM silver nitrate solution through the apparatus for approximately 10 minutes, then drain the silver nitrate solution from the apparatus. Circulate approximately 1 L of a 3 mM sodium borohydride (nucleating agent) solution through the apparatus for approximately 5 minutes or until the solution becomes dark, then drain the sodium borohydride solution from the apparatus. Rinse the system by circulating approximately 1 L of deionized (DI) water through the apparatus for approximately 1 minute, then drain the DI water from the apparatus. Circulate approximately 1 L of a 10 mM solution of sodium nitrate and sodium sulfide (sulfidation agent) through the apparatus for approximately 24 hours, then drain the solution of sodium nitrate and sodium sulfide from the apparatus.

Example 3: Coating a Membrane with Non-Sulfidated Silver Nanoparticles Using D-Glucose

[0025] Set the pump to have a feed pressure of approximately 8 psi and a flow rate of approximately 945 mL/min. Circulate approximately 1 L of a 3 mM silver nitrate solution through the apparatus for approximately 10 minutes, then drain the silver nitrate solution from the apparatus. Circulate approximately 1 L of a 3 mM D-glucose (nucleating agent) solution through the apparatus for approximately 24 hours or until the solution becomes dark, then drain the D-glucose solution from the apparatus.

Example 4: Coating a Membrane with Sulfidated Silver Nanoparticles Using D-Glucose

[0026] Set the pump to have a feed pressure of approximately 8 psi and a flow rate of approximately 945 mL/min. Circulate approximately 1 L of a 3 mM silver nitrate solution through the apparatus for approximately 10 minutes, then drain the silver nitrate solution from the apparatus. Circulate approximately 1 L of a 3 mM D-glucose (nucleating agent) solution through the apparatus for approximately 24 hours or until the solution becomes dark, then drain the D-glucose solution from the apparatus. Rinse the system by circulating approximately 1 L of deionized (DI) water through the apparatus for approximately 1 minute, then drain the DI water from the apparatus. Circulate approximately 1 L of a 10 mM solution of sodium nitrate and sodium sulfide (sulfidation agent) through the apparatus for approximately

24 hours, then drain the solution of sodium nitrate and sodium sulfide from the apparatus.

Example 5: Biofouling Prevention Effectiveness of a Coated Membrane

[0027] FIGS. 3A-3B illustrate the effectiveness of coated membranes with respect to biofouling prevention. This graph shows that the membrane that had been coated with the silver nanoparticles exhibits higher permeate flowrates over time than the membrane that had not been coated. *P. aeruginosa* bacteria in secondary wastewater with a concentration of 2,500,000 CFU/mL was processed through a reverse osmosis membrane that was coated with the silver nanoparticles and through a reverse osmosis membrane that was not coated with the silver nanoparticles at a pressure of 100 psi for both FIG. 3A and FIG. 3B. FIG. 3A had a crossflow velocity of 37.8 cm/s and FIG. 3B had a crossflow of 21.4 cm/s. Table 1 below shows the total permeate collected for the datasets.

TABLE 1

Pressure (psi)	Bacteria concentration (CFU/mL)	Crossflow velocity (cm/s)	Membrane	Total permeate (L) in 5 days	Recovery after 1st cleaning (%)	Recovery after 2nd cleaning (%)
100	2.5*10^6	37.8	Pristine	98.2	30.6	NA
			Coated	106.7	39.4	NA
100	2.5*10^6	21.4	Pristine	64.2	7.7	10.7
			Coated	68	13.5	25.9

[0028] At both flowrates the total permeate collected after the initial 5 days and the percent recovery after cleaning were higher for the coated membranes than for the pristine membranes.

We claim:

1. An antimicrobial membrane, comprising:
A. a membrane; and
B. a plurality of silver nanoparticles coupled to the membrane, wherein the plurality of silver nanoparticles have a silver sulfide core-shell structure.

2. The membrane of claim 1, wherein:
A. the membrane Component A is a reverse osmosis membrane or a nanofiltration membrane;
B. the membrane Component A is a microfiltration membrane or an ultrafiltration membrane;
C. the plurality of silver nanoparticles Component B have a weight percentage of sulfur between approximately 0.1% to approximately 7%; or
D. the plurality of silver nanoparticles Component B have a diameter of between approximately 3 nanometers (nm) to approximately 350 nm

3. A method for coating a membrane with silver nanoparticles comprising:
A. providing a membrane coating apparatus comprising:
i. a feed tank;
ii. a pump coupled to the feed tank;
iii. a membrane housing comprising a feed end, concentrate end and a permeate end, wherein the feed end is coupled to the pump;
iv. a return tank coupled to the concentrate end of the membrane housing; and
v. plugged or unplugged permeate end
B. a dosing unit coupled to the return tank and the feed tank;
C. circulating a silver nitrate solution through the membrane coating apparatus;
D. circulating a nucleating agent solution through the membrane coating apparatus; and
E. circulating a solution of sodium nitrate and a sulfidation agent through the membrane coating apparatus.

4. The method of claim 3, wherein:
A. the nucleating agent (Component D) is selected from the group consisting of sodium borohydride, hydrazine, D-glucose, hyaluronic acid, and combinations thereof;
B. the nucleating agent (Component D) is sodium borohydride;
C. the sulfidation agent (Component E) is selected from the group consisting of sodium sulfide, sodium thiosulfate, thiocarbamide, thioacetamide, and combinations thereof;
D. the silver nitrate solution (Component C) has a concentration from approximately 1 millimolar (mM) to approximately 300 mM;
E. the nucleating agent solution (Component D) has a concentration from approximately 1 mM to approximately 300 mM;
F. the sodium nitrate (Component E) has a concentration from approximately 10^{-5} mM to 10^{-1} M;
G. the sulfidation agent (Component E) has a concentration from approximately 10^{-5} mM to 10^{-1} M;
H. the silver nitrate solution (Component C) is cycled for a time period of at least approximately 5 minutes;
I. the nucleating agent solution (Component D) is cycled for a time period of at least approximately 2 minutes; and
J. the solution of sodium nitrate and the sulfidation agent (Component E) is cycled for a time period of at least approximately 30 minutes.

5. A method for coating a membrane with silver nanoparticles comprising:
A. exposing a membrane to a silver nitrate solution;
B. exposing the membrane to a nucleating agent solution; and
C. exposing the membrane to a solution of sodium nitrate and a sulfidation agent.

6. The method of claim 5, wherein:
A. the nucleating agent (Component B) is selected from the group consisting of sodium borohydride, hydrazine, D-glucose, hyaluronic acid, and combinations thereof;
B. the sulfidation agent (Component C) is selected from the group consisting of sodium sulfide, sodium thiosulfate, thiocarbamide, thioacetamide, and combinations thereof;

C. the silver nitrate solution (Component A) has a concentration from approximately 1 millimolar (mM) to approximately 300 mM;

D. the nucleating agent solution (Component B) has a concentration from approximately 1 mM to approximately 300 mM;

E. the sodium nitrate (Component C) has a concentration from approximately 10^{-5} M to 10^{-1} M;

F. the sulfidation agent (Component C) has a concentration from approximately 10^{-5} M to 10^{-1} M;

G. the membrane is exposed to the silver nitrate solution (Component A) for a time period of at least approximately 5 minutes;

H. the membrane is exposed to the nucleating agent solution (Component B) for a time period of at least approximately 2 minutes; and

I. the membrane is exposed to the solution of sodium nitrate and the sulfidation agent (Component C) for a time period of at least approximately 30 minutes.

7. A method for coating a membrane with silver nanoparticles comprising:

A. providing a membrane coating apparatus comprising:

i. a feed tank;

ii. a pump coupled to the feed tank;

iii. a membrane housing comprising a feed end, concentrate end; and a permeate end, wherein the feed end is coupled to the pump;

iv. a return tank coupled to the permeate end of the membrane housing; and

v. a dosing unit coupled to the return tank and the feed tank;

B. circulating a silver nitrate solution through the membrane coating apparatus;

C. circulating a D-glucose solution through the membrane coating apparatus; and

D. circulating a solution of sodium nitrate and sodium sulfide through the membrane coating apparatus.

8. The method of claim **7**, wherein:

A. the silver nitrate solution (Component B) has a concentration of approximately 3 millimolar (mM);

B. the D-glucose solution (Component C) has a concentration of approximately 3 mM;

C. the sodium nitrate (Component D) has a concentration of approximately 10 mM;

D. the sodium sulfide (Component D) has a concentration of approximately 10 mM;

E. the silver nitrate solution (Component B) is cycled for a time period of at least approximately 5 minutes;

F. the D-glucose solution (Component C) is cycled for a time period of at least approximately 24 hours; and

G. the solution of sodium nitrate and sodium sulfide (Component D) is cycled for a time period of at least approximately 30 minutes.

9. A method for coating a membrane with silver nanoparticles comprising:

A. exposing a membrane to a silver nitrate solution;

B. exposing the membrane to a sodium borohydride solution; and

C. exposing the membrane to a solution of sodium nitrate and sodium sulfide.

10. The method of claim **9**, wherein:

A. the silver nitrate solution (Component A) has a concentration of approximately 3 millimolar (mM);

B. the sodium borohydride solution (Component B) has a concentration of approximately 3 mM;

C. the sodium nitrate (Component C) has a concentration of approximately 10 mM;

D. the sulfidation agent has a concentration of approximately 10 mM;

E. the membrane is exposed to the silver nitrate solution (Component A) for a time period from approximately 5 minutes to approximately 15 minutes;

F. the membrane is exposed to the sodium borohydride solution (Component B) for a time period from approximately 2 minutes to approximately 10 minutes; and

G. the membrane is exposed to the solution of sodium nitrate (Component C) and the sulfidation agent for a time period from approximately 30 minutes to approximately 24 hours.

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