Accelerated Mixed Gas Integrity Testing Of Porous Materials

CROSS-REFERENCED TO RELATED APPLICATIONS

[001] This application claims the benefit of U.S. Provisional Patent Application No.: 61/435,623, filed on ~~June 11, 2008~~, Jan. 24, 2011 the entire contents of which are incorporated by reference herein.

DESCRIPTION OF THE INVENTION

[002] **Field of the Invention**

[003] The invention relates generally to the field of validation testing of porous materials. In certain embodiments, the invention relates to validation testing porous materials using accelerated mixed gas integrity testing methods, devices and systems.

[004] **Background of the Invention**

[005] Porous materials play a significant role in a wide variety of industrial applications including processing, e.g. filtering, packaging, containing, and transporting manufactured goods and raw materials. The industrial settings in which they are used include the pharmaceutical and biotechnology industries; the oil and gas industries and the food processing and packaging industries, to name but a few.

[006] In the pharmaceutical and biotechnology industries, as well as the food processing industry, porous materials (e.g., membranes and the like) are used as filtration devices to eliminate undesirable and potentially harmful contaminants from marketable end products. Quality control and quality assurance requires that these filtration devices comply with desired performance criteria. Integrity testing of porous membranes provides a means for ensuring that a particular device meets its desired performance criteria. Typically, in the case of porous membranes, integrity testing ensures that the membrane is free of defects (e.g., free of breaches in the membrane exceeding a desired size limitation) which would impair the

WH AT IS CLAIMED IS:

1. A method of assessing the integrity of a porous material loaded into a porous material device using a compressed gas mixture and without the need for any gas component in the gas mixture to achieve a steady state concentration comprising

a) loading a wetted porous material into a device having a housing for receiving the wetted porous material, resulting in a wetted porous material having a feed side and a permeate side;

b) feeding a compressed gas mixture of two or more gas components to the feed side of the wetted porous material, wherein the feed gases have differing permeabilities in the liquid used to wet the porous material, such that one of the gases permeates through the liquid of the wetted porous material faster than the other gas or gases used;

c) flushing out the existing gas in ~~charging~~ the permeate side volume of the porous material and replacing ~~volume~~ with a gas mixture that has a composition similar to the expected composition of permeate gas generated from an integral membrane;

d) recirculating the permeate gas and venting excess permeate gas ~~and~~ while maintaining a feed side gas sweep flow:

e) assessing the concentration of the gas mixture on the permeate side ~~of~~; and

f) comparing the assessed concentration of the permeate gas with the value expected from an integral device, wherein a deviation from the expected concentration by more than predetermined amount is a signal for a non-integral porous material or porous material device.

2. The method of claim 1, wherein the liquid in (a) is water.

3. The method of claim 1, wherein at least one of the gases is $C0_2$.

4. The method of claim 1, wherein at least one of the gases is 0_2.

5. The method of claim 1, wherein the porous material comprises a hydrophilic material.

6. The method of claim 1, wherein the porous material comprises a hydrophobic material.

7. The method of claim 1, wherein the porous material comprises a membrane.

8. The method of claim ~~1~~ 7, wherein the ~~porous material is contained in a housing~~ membrane comprises a filtration device.

9. The method of claim ~~1~~ 7, wherein the ~~porous material is contained in a cartridge~~ membrane is an asymmetric membrane.

10. The method of claim 7, wherein the membrane ~~comprises a filtration device~~ is a symmetric membrane.

11. The method of claim 7, wherein the membrane is ~~an asymmetric membrane~~ comprised of a polymer.

12. The method of claim 11, wherein the polymer is PES.

13. The method of claim 7, wherein the membrane is a ~~symmetric membrane~~ flat sheet in flat plate or spiral wound formats.

14. The method of claim 7, wherein the membrane is ~~comprised of a polymer~~ a pleated sheet, in hollow fiber or tubular formats.

15. The method of claim ~~14~~ 1, wherein the ~~polymer is PES~~ porous material is contained in a housing.

16. The method of claim ~~7~~ 1, wherein the ~~membrane is a flat sheet in flat plate or spiral wound formats~~ porous material is contained in a cartridge.

17. The method of claim ~~7~~ 1, wherein the ~~membrane is a pleated sheet, in hollow fiber or tubular formats~~ porous material is a membrane comprising more than 1 layer.

18. The method of claim 1, wherein the porous material is selected from polyether sulfone, polyamide, nylon, cellulose, polytetrafluoroethylene, polysulfone, polyester, polyvinylidene fluoride, polypropylene, poly(tetrafluoroethylene-co-perfluoro(alkyl vinyl ether)), poly carbonate, polyethylene, glass fiber, polycarbonate, ceramic, and metals.

19. The method of claim 1, wherein ~~the porous material is a membrane comprising more than 1 layer~~ at least one of the gases is hexafluoroethane.

~~20. The method of claim 1 , wherein the porous material is selected from polyether sulfone. polyamide, nylon, cellulose, polytetrafluoroethylene, polysulfone. polyester, polyvinylidene fluoride, polypropylene, poly (tetrafluoroethylene-co-perfluoro(alkyl vinyl ether)), poly carbonate, polyethylene, glass fiber, polycarbonate, ceramic, and metals.~~

20. A method of assessing the integrity of a porous material loaded into a porous material device using a compressed gas mixture and without the need for any gas component in the gas mixture to achieve a steady state concentration comprising

a) loading a wetted porous material into a device having a housing for receiving the wetted porous material, resulting in a wetted porous material having a feed side and a permeate side;

b) feeding a compressed gas mixture of two or more gas components wherein at least one gas is N_2 and the other gas is O_2 to the feed side of the wetted porous material, wherein the feed gases have differing permeabilities in the liquid used to wet the porous material, such that one of the gases permeates through the liquid of the wetted porous material faster than the other gas or gases used;

c) flushing out the existing gas in the permeate side volume of the porous material and replacing with a gas mixture that has a composition similar to the expected composition of permeate gas generated from an integral membrane;

d) recirculating the permeate gas and venting excess permeate gas while maintaining a feed side gas sweep flow;

e) assessing the concentration of the gas mixture on the permeate side; and

f) comparing the assessed concentration of the permeate gas with the value expected from an integral device, wherein a deviation from the expected concentration by more than predetermined amount is a signal for a non-integral porous material or porous material device.

~~21. The method of claim 1, wherein at least one of the gasses is hexafluoroethane.~~

21. A method of assessing the integrity of a porous material loaded into a porous material device using a compressed gas mixture and without the need for any gas component in the gas mixture to achieve a steady state concentration, comprising:

a) loading a wetted porous material into a device having a housing for receiving the wetted porous material, resulting in a wetted porous material having a feed side and a permeate side;

b) feeding a compressed gas mixture of two or more gas components wherein at least one gas is hexafluoroethane and the other gas is CO_2 to the feed side of the wetted porous material, wherein the feed gases have differing permeabilities in the liquid used to wet the porous material, such that one of the gases permeates through the liquid of the wetted porous material faster than the other gas or gases used;

c) flushing out the existing gas in the permeate side volume of the porous material and replacing with a gas mixture that has a composition similar to the expected composition of permeate gas generated from an integral membrane;

d) recirculating the permeate gas and venting excess permeate gas while maintaining a feed side gas sweep flow;

e) assessing the concentration of the gas mixture on the permeate side; and

f) comparing the assessed concentration of the permeate gas with the value expected from an integral device, wherein a deviation from the expected concentration by more than predetermined amount is a signal for a non-integral porous material or porous material device.

22. A method of assessing the integrity of a porous material loaded into a porous material device using a compressed gas mixture and without the need for any gas component in the gas mixture to achieve a steady state concentration, comprising:

a) loading a wetted porous material into a device having a housing for receiving the wetted porous material, resulting in a wetted porous material having a feed side and a permeate side;

b) feeding a compressed gas mixture of two or more gas components wherein at least one gas is CO_2 and the other gas is O_2 to the feed side of the wetted porous material, wherein the feed gases have differing permeabilities in the liquid used to wet the porous material, such that one of the gases permeates through the liquid of the wetted porous material faster than the other gas or gases used;

c) flushing out the existing gas in the permeate side volume of the porous material and replacing with a gas mixture that has a composition similar to the expected composition of permeate gas generated from an integral membrane;

d) recirculating the permeate gas and venting excess permeate gas while maintaining a feed side gas sweep flow;

e) assessing the concentration of the gas mixture on the permeate side; and

f) comparing the assessed concentration of the permeate gas with the value expected from an integral device, wherein a deviation from the expected concentration by more than predetermined amount is a signal for a non-integral porous material or porous material device.