A GRAPHENE OXIDE AEROGEL

CROSS REFERENCE

[0001] The present application claims priority to Australian provisional application number 2021902883, filed 6 September 2021, the entire contents of which is incorporated herein by reference in its entirety.

FIELD OF THE INVENTION

[0002] The invention relates to a graphene oxide aerogel, and to methods and apparatus for the use of such aerogels, in particular as desiccants. However, it will be appreciated that the invention is not limited to this particular field of use.

BACKGROUND

[0003] The following discussion of the prior art is provided to place the invention in an appropriate technical context and enable the advantages of it to be more fully understood. It should be appreciated, however, that any discussion of the prior art throughout the specification should not be considered as an express or implied admission that such prior art is widely known or forms part of the common general knowledge in the field.

[0004] Nanoporous materials with large nano-sized pores and high surface area are of considerable interest worldwide for adsorption processes. Heterogeneous three dimensional porous materials such as silica gel and zeolites are widely used desiccant materials. However, issues such as large pore size distribution, low surface area to pore volume ratios, low hydrophilicity and/or poor hydrothermal stability associated with the aforementioned materials offer limitations for wide applicability.

[0005] Ideal desiccant materials for adsorption applications have a large surface area and high porosity with chemical and mechanical stability. Accordingly, there is a need to develop new desiccant materials having one or more of these desirable properties.

[0006] It is an object of the present invention to overcome or ameliorate one or more the disadvantages of the prior art, or at least to provide a useful alternative.

SUMMARY

[0007] The inventors of the present application have surprisingly developed lightweight GO-based aerogel materials which may have a highly porous structures and large surface area making them suitable for adsorption applications. These GO-based aerogels may have high water adsorption capacity with fast adsorption and desorption rates due to their unique physico-chemical properties. Moreover, the desorption process for these aerogel materials

may be completed at low temperature (50 °C) or even at room temperature with low humidity.

[0008] In a first aspect of the disclosure there is provided an aerogel comprising graphene oxide which is crosslinked with a metal ion, wherein the metal ion is selected from the group consisting of alkali metal ions, alkaline earth metal ions, transition metal ions and basic metal ions; and wherein said metal ion is not selected from the group consisting of Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zr^{4+}, Sn^{4+}, Ti^{4+}, V^{5+}, La^{3+}, Cr^{3+}, Al^{3+}, Zn^{2+} and Ce^{4+}.

[0009] The following options may be used in conjunction with the first aspect, either individually or in any combination.

[00010] The skilled person will understand that the aerogel may be any shape or size, and that its shape and/or size will depend upon the application. It may have, for example, a spherical structure, cubic structure, cylindrical structure, rectangular structure, tube-like structure, or a wire-like structure. In certain specific examples, the aerogel may be in the form of a sheet, which may, for example, be rolled up to fit into a cylindrical-shaped apparatus. In certain specific examples, the aerogel may be in the form of a cake, flakes, or a powder.

[00011] The weight ratio of graphene oxide to metal ion may be from about 500:1 to about 1:20, or it may be from about 200:1 to about 1:20, about 100:1 to about 1:20, about 50:1 to about 1:20, about 20:1 to about 1:20, about 200:1 to about 1:10, about 200:1 to about 1:5, about 100:1 to about 1:10, about 50:1 to about 1:5, about 20:1 to about 1:2, about 5:1 to about 1:2, or about 5:1 to about 1:1. In certain specific examples, the weight ratio of graphene oxide to metal ion is from about 200:1 to about 1:5. In certain specific examples, the weight ratio of graphene oxide to metal ion is from about 5:1 to about 1:1. The weight ratio of graphene oxide to metal ions may be, for example, about 500:1, 200:1, 100:1, 50:1, 20:1, 10:1, 5:1, 2:1, 1:1, 1:2, 1:5, 1:10, or 1:20.

[00012] The metal ion may be any ion capable of cross-linking the graphene oxide. In certain examples, it is selected from alkali metal ions and alkaline earth metal ions, and combinations thereof. In certain alternative examples, it is a basic metal ion or a transition metal ion. In certain examples, the metal ion is an ion selected from the group consisting of: beryllium, magnesium, calcium, strontium, lithium and barium. In certain examples, the metal ion may be an iron ion. In certain specific examples, the metal ion is selected from the group consisting of: Be^{2+}, Mg^{2+}, Ca^{2+}, Sr^{2+}, Li^{+}, and Ba^{2+}. In certain examples, the metal ion is an alkaline earth metal ion. In certain specific examples, the alkaline earth metal ion is Ca^{2+}.

[00013] Without being bound by theory, the inventors of the present application postulate that graphene oxide cross-linked by certain metal ions may provide advantageous adsorption properties in part due to the high hydration number of the cross-linking ions, which attract more water molecules around them, and/or the fast exchange rate of water molecules in the first hydration shell of the respective metal ions, which allow the water molecules to be exchanged rapidly thereby providing a fast adsorption rate for the crosslinked GO aerogels.

[00014] The graphene oxide itself (i.e. prior to being crosslinked) is an essentially two dimensional material. The size and shape of the graphene oxide may affect the properties of the aerogel. The graphene oxide may have a mean aspect ratio of at least about 20, or at least about 50, 100, 200, 500, 1000, 2000, 5000, 10^4, or 10^5. It may be from about 20 to about 10^6, or from about 10^2 to 10^6, 10^3 to 10^6, 10^4 to 10^6, 10^5 to 10^6, 20 to 10^5, 20 to 10^4, 20 to 10^3, 20 to 10^2, 10^2 to 10^3, 10^3 to 10^4, or 10^4 to 10^5. It may be for example about 20, 30, 40, 50, 100, 200, 500, 10^3, 5 x 10^3, 10^4, 5 x 10^4, 10^5, 5 x 10^5, or 10^6. The aspect ratio may be defined as the ratio of the minimum lateral dimension (i.e. in the plane of the graphene oxide) to the average non-lateral dimension (i.e. orthogonal to the plane of the graphene oxide). The graphene oxide may be non-uniform in shape, but on average may have lateral dimension at least 20 times greater than its average non-lateral dimension.

[00015] In certain examples, the graphene oxide may have an average lateral dimension of less than about 10,000 nm, 5000nm, 2000 nm, 1000 nm, 500 nm, 200 nm, 100 nm, 50 nm, or less than about 20, 10, 5, 2 or 1 nm. In certain examples, an average lateral dimension of the graphene oxide is about 500 nm or less. The graphene oxide may have an average lateral dimension of from about 0.5 nm to about 10,000 nm, or from about 1 to 500, 2 to 500, 5 to 500, 10 to 500, 20 to 500, 0.5 to 200, 0.5 to 100, 0.5 to 50, 0.5 to 20, 2 to 50, 5 to 100, or 10 to 200 nm. It may be for example about 0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, or 1000 nm. The graphene oxide may comprise particles formed from a number of sheets of laminar material. The average number of individual sheets in each particle may be 1 or may be greater than about 1, or greater than about 2, 3, 4, 5, 6, 7, 8, 9, 10, 15, 20 or 50 sheets. It may be from about 1 sheet to about 1000 sheets, or from about 1 to 500, 1 to 200, 1 to 100, 1 to 50, 5 to 100, 5 to 1000, 10 to 1000, 20 to 1000, 50 to 1000, 5 to 100, 10 to 200, or 20 to 500 sheets. It may be for example about 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80, 90, 100, 200, 500, or 100 sheets.

[00016] In certain examples, the aerogel may comprise a mixture of graphene oxide having different average lateral dimensions. For example, it may comprise a mixture of

graphene oxide having an average lateral dimension of about 5 μm, and graphene oxide having an average lateral dimension of about 300 nm (i.e. the graphene oxide may have a bimodal size distribution).

[00017] In other examples, the graphene oxide may have an average lateral dimension of more than about 200 nm, 500 nm, 1000 nm, 2000 nm, or 5000 nm. In certain examples, an average lateral dimension of the graphene oxide is about 500 nm or more. The graphene oxide may have an average lateral dimension of from about 0.2 μm to about 10 μm, or from about 0.5 to 5, 0.5 to 2, 0.5 to 1, 0.2 to 10, 0.2 to 5, or 0.2 to 0.5 μm. It may be for example about 0.5, 1, 2, 5, or 10 μm. The graphene oxide may comprise particles formed from a number of sheets of laminar material. The average number of individual sheets in each particle may be greater than about 20, 30, 40, 50, 60, 70, 80, 90, 100 or 200 sheets. It may be from about 20 sheets to about 1000 sheets, or from about 50 to 500, 50 to 200, 50 to 100, 20 to 50, 20 to 100, 20 to 200, or 20 to 500 sheets. It may be for example about 20, 25, 30, 40, 50, 60, 70, 80, 90, 100, 200, 500, or 1000 sheets.

[00018] The graphene oxide may have an average non-lateral dimension (i.e. thickness) of less than about 2000 nm, or less than about 1000, 500, 200, 100, 50, 20, 10, 5, 2, 1, or 0.5 nm. It may be from about 1 to 500, 2 to 500, 5 to 500, 10 to 500, 20 to 500, 0.5 to 200, 0.5 to 100, 0.5 to 50, 0.5 to 20, 2 to 50, 5 to 100, or 10 to 200 nm. It may be, for example, about 0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, or 1000 nm.

[00019] The carbon:oxygen ratio of the graphene oxide may be from about 0.1 to about 5, or about 0.5 to about 5, about 1 to about 4, about 1.5 to about 2.5, or about 2 to about 2.5. It may be, for example, about 0.1, 0.2, 0.5, 1, 1.5, 2, 2.1, 2.2, 2.25, 2.3, 2.35, 2.4, 2.5, 3, 4, or 5. In certain examples, the carbon:oxygen ratio of the graphene oxide is from about 0.5 to about 5. In certain specific examples, the carbon:oxygen ratio of the graphene oxide is about 2.25.

[00020] The adsorption capacity of the aerogel may be from about 5% to about 1000% at 100% relative humidity, or it may be from about 5% to about 500%, about 10% to about 500%, about 20% to about 500%, about 50% to about 500%, about 100% to about 500%, about 20% to about 400%, about 50% to about 300%, about 50% to about 250%, or about 100% to about 250%, at 100% relative humidity. In certain examples, the adsorption capacity of the aerogel is from about 20 to about 400% at 100% relative humidity. The adsorption capacity of the aerogel may be, for example, about 5, 10, 15, 20, 50, 75, 100, 110, 120, 150, 200, 250, 300, 400, 500, 600, 700, 800, 900, or 1000% at 100% relative humidity. In certain examples, the aerogel has an adsorption capacity of at least about

40% at 100% relative humidity, or it may be at least about 50, 60, 70, 80, 90, 100, 110, 120, 150, or 200% at 100% relative humidity.

[00021] The adsorption capacity of the aerogel may be from about 5% to about 500% at 50% relative humidity, or it may be from about 5% to about 200%, about 10% to about 500%, about 20% to about 500%, about 50% to about 500%, about 100% to about 500%, about 20% to about 400%, about 50% to about 300%, about 50% to about 250%, or about 100% to about 250% at 50% relative humidity. In certain examples, the adsorption capacity of the aerogel is from about 20 to about 150% at 50% relative humidity. The adsorption capacity of the aerogel may be, for example, about 5, 10, 15, 20, 50, 75, 100, 110, 120, 150, 200, 250, 300, 400, or 500% at 50% relative humidity.

[00022] The aerogel may have a density of from about 0.001 g/cm^3 to about 0.4 g/cm^3, or it may be from about 0.002 g/cm^3 to about 0.4 g/cm^3, about 0.005 g/cm^3 to about 0.4 g/cm^3, about 0.005 g/cm^3 to about 0.3 g/cm^3, about 0.005 g/cm^3 to about 0.25 g/cm^3, about 0.01 g/cm^3 to about 0.2 g/cm^3, about 0.02 g/cm^3 to about 0.2 g/cm^3, or about 0.1 g/cm^3 to about 0.2 g/cm^3, In certain examples, the density of the aerogel is from about 0.005 g/cm^3 to about 0.25 g/cm^3. The density of the aerogel may be, for example, about 0.001, 0.002, 0.005, 0.01, 0.02, 0.05, 0.1, 0.2, 0.3 or 0.4 g/cm^3.

[00023] The aerogel may have a porosity of from about 50% to about 99.9%, or from about 60% to about 99.9%, about 60% to about 99%, about 70% to about 99%, about 80% to about 99%, about 90% to about 99.9%, or about 70% to about 95%. In certain examples, the porosity of the aerogel is from about 90 to about 99.9%. The porosity of the aerogel may be, for example, about 50, 55, 60, 65, 70, 75, 80, 85, 90, 91, 92, 95, 97, 99, 99.5, or about 99.9%.

[00024] In some examples, the pore size of the aerogel can range from about 10 nm to about 500 µm. In some examples, the average pore size is at least about 20 nm, at least about 50 nm, 100 nm, 200 nm, 300 nm, 400 nm, or 500 nm. In some examples, the average pore size is at least about 1 µm, 10 µm, 20 µm, 50 µm, 100 µm, 110 µm, 120 µm, 130 µm, 140 µm, 150 µm, 160 µm, 170 µm, 180 µm, 190 µm, 200 µm, 210 µm, or 220 µm. In some examples, the average pore size may be about 500 µm or less, 400 µm or less, 300 µm or less, or 250 µm or less. Typically, the aerogel may have an average pore size from about 100 to about 250 µm, about 110 to about 220 µm, about 120 to about 210 µm, or from about 130 and 200 µm.

[00025] In certain examples, the metal ion is an Li, Na, K, Rb, Cs, Fr, Be, Mg, Ca, Sr, Ba, Ra, Sc, Y, Lu, Lr, Ti, Zr, Hf, Rf, V, Nb, Ta, Db, Cr, Mo, W, Sg, Mn, Tc, Re, Bh, Fe,

Ru, Os, Hs, Co, Rh, Ir, Mt, Ni, Pd, Pt, Ds, Cu, Ag, Au, Rg, Zn, Cd, Hg, Cn, Al, Ga, Ge, In, Sn, Sb, Tl, Pb, Bi, Po, Nh, Fl, Mc, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, or Lv ion, or a combination thereof. In certain specific examples, it is a Li, Na, K, Rb, Cs, Fr, Be, Mg, Ca, Sr, Ba, Ra, Sc, Y, Lu, Lr, Hf, Rf, Nb, Ta, Db, Mo, W, Sg, Mn, Tc, Re, Bh, Ru, Os, Hs, Rh, Ir, Mt, Pd, Pt, Ds, Ag, Au, Rg, Cd, Hg, Cn, Ga, Ge, In, Sb, Tl, Pb, Bi, Po, Nh, Fl, Mc, or Lv ion, or a combination thereof.

[00026] In certain alternative examples, the metal ion is not one or more of a Li, Na, K, Rb, Cs, Fr, Be, Mg, Ca, Sr, Ba, Ra, Sc, Y, Lu, Lr, Ti, Zr, Hf, Rf, V, Nb, Ta, Db, Cr, Mo, W, Sg, Mn, Tc, Re, Bh, Fe, Ru, Os, Hs, Co, Rh, Ir, Mt, Ni, Pd, Pt, Ds, Cu, Ag, Au, Rg, Zn, Cd, Hg, Cn, Al, Ga, Ge, In, Sn, Sb, Tl, Pb, Bi, Po, Nh, Fl, Mc, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and/or Lv ion.

[00027] In a second aspect of the disclosure there is provided a method for preparing an aerogel, said aerogel comprising graphene oxide which is crosslinked with a metal ion, wherein the metal ion is selected from the group consisting of alkali metal ions, alkaline earth metal ions, transition metal ions and basic metal ions; and wherein said metal ion is not selected from the group consisting of Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zr^{4+}, Sn^{4+}, Ti^{4+}, V^{5+}, La^{3+}, Cr^{3+}, Al^{3+}, Zn^{2+} and Ce^{4+}, the method comprising the steps of:

(a) contacting graphene oxide with a crosslinking agent in the presence of a liquid; and

(b) removing the liquid to form the aerogel.

[00028] The following options may be used in conjunction with the second aspect, either individually or in any combination.

[00029] The metal ion and/or the aerogel may be as hereinbefore described with respect to the first aspect.

[00030] The liquid may be an aqueous liquid, optionally comprising one or more salts. It may comprise water. In certain examples, the liquid is substantially water. The liquid may comprise a polar solvent. It may comprise an alcohol. It may comprise one or more volatile organic solvents. It may, for example, comprise ethanol, methanol, acetone, ethyl acetate, dichloromethane, chloroform, or propanol. The liquid may have a boiling point of below about 150°C, measured at 1 atm pressure, or below about 130°C, 110°C, 90°C or 70°C, measured at 1 atm pressure.

[00031] In certain examples, removing the liquid is by freeze-drying. The freeze-drying may be performed at from about -100°C to about -20 °C, or from about -80°C to

about -40°C, or about -100, -80, -60, -50, -40, -30, or -20 °C. The freeze drying may be performed over a period of from about 1 to about 48 hours, or from about 2 to about 24, about 3 to about 12, about 4 to about 10, or about 2 to about 5 hours. In certain specific examples, the freeze-drying step is conducted under conditions of -60°C temperature, and over a period of from about 2 to about 24 hours. Typically, vacuum levels for freeze drying are between 50mTorr and 300mTorr with 100mTorr to 200mTorr being the most common range.

[00032] In certain examples, after the contacting step, the graphene oxide is transferred to a mould prior to the freeze-drying step. The mould may be any shape or size. The skilled person will understand that the mould shape and/or size will depend upon the desired shape and/or size of the aerogel. The graphene oxide cross-linked with the metal ion in a mixture with the liquid may be transferred to a mould, and upon removal of the liquid, the mixture may form an aerogel having substantially the same volume as the mould.

[00033] In certain examples, the method comprises the steps of: providing an aqueous solution of graphene oxide at a predetermined concentration, followed by exposure to a crosslinking agent thereby providing a cross-linked graphene oxide.

[00034] In certain examples, the crosslinking agent may be added to the aqueous solution of graphene over a period of from about 5 minutes to about 12 hours, or from about 10 minutes to about 6 hours, about 30 minutes to about 5 hours, about 1 hour to about 4 hours, or about 1 hour to about 3 hours. It may be added over a period of about 5 10, 15, 20, 30, 40, or 50 minutes, or about 1, 1.5, 2, 2.5, 3, 4, 5, 6, 7, 8, 9, 10, or 12 hours. In certain specific examples, the crosslinking agent is added to the aqueous solution of graphene over a period of from about 10 minutes to about 6 hours.

[00035] In certain examples, the concentration of graphene oxide in the aqueous solution is from about 0.01 wt.% to about 30 wt.%, or about 0.02 wt.% to about 30 wt.%, about 0.05 wt.% to about 30 wt.%, about 0.05 wt.% to about 20 wt.%, about 0.05 wt.% to about 10 wt.%, about 0.05 wt.% to about 5 wt.%, about 0.05 wt.% to about 1 wt.%, or about 0.05 wt.% to about 1.5 wt.%. In certain specific examples, the concentration of graphene oxide in the aqueous solution is from about 0.01 to about 20 wt.%. In certain specific examples, the concentration of graphene oxide in the aqueous solution is from about 0.05 to about 5 wt.%. The concentration of graphene oxide in the aqueous solution may be about 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1, 2, 5, 10, 20, or 30 wt.%. In certain specific examples, the concentration of graphene oxide in the aqueous solution is about 1 wt.%.

[00036] In certain examples, the crosslinking agent includes an alkaline earth metal ion and/or an alkali metal ion. In certain specific examples, the alkaline earth metal ion and/or alkali metal ion is selected from the group consisting of: beryllium, magnesium, calcium, strontium, lithium and barium. In certain examples, the crosslinking agent is selected from the group consisting of: $CaCl_2$ and $MgCl_2$. In certain examples, the crosslinking agent is a salt of the metal ion as hereinbefore described with respect to the first aspect.

[00037] In certain examples, after the removing step, the aerogel is compressed. It may be compressed to 95% or less of its original volume, or to 90, 85, 80, 75, 70, 65, 60, 55, 50, 45, or 40% or less of its original volume. It may be compressed to about 95, 90, 85, 80, 75, 70, 65, 60, 55, 50, 45, 40, 35, 30, 25, or 20% of its original volume. In certain specific examples, the aerogel is compressed to about 50% of its original volume. It may be compressed by subjecting it to a pressure of from about 0.01 bar to about 5 bar, or from about 0.01 bar to about 2 bar, about 0.02 bar to about 1.5 bar, about 0.05 bar to about 1 bar, about 0.05 bar to about 0.5 bar, or about 0.05 bar to about 0.1 bar. It may, for example, be subjected to a pressure of about 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1, 2, 3, 4, or 5 bar.

[00038] In a third aspect of the disclosure there is provided a graphene oxide aerogel produced according to the method of the second aspect.

[00039] In certain examples, the graphene oxide aerogel may be as hereinbefore described with respect to the first aspect.

[00040] In a fourth aspect of the disclosure there is provided a method to adsorb moisture from a gas stream or an atmosphere laden with said moisture, the method comprising the step of contacting said gas stream or said atmosphere with a graphene oxide aerogel, thereby to adsorb said moisture from said gas stream or said atmosphere.

[00041] The gas stream or atmosphere may be a waste gas stream. In certain alternative examples it may be an inlet stream for a process, particularly where removing water vapour from the inlet stream is advantageous.

[00042] In certain examples, the aerogel may be housed in a porous packaging, which allows moisture to pass therethrough. The packaging may be used for a storage application, such as, for example, the storage of a dry food substance.

[00043] In certain examples, the graphene oxide aerogel is the aerogel according to the first or third aspect.

[00044] In a fifth aspect of the disclosure there is provided use of a graphene oxide aerogel to adsorb moisture from a gas stream. The gas stream may be a waste gas stream. In certain alternative examples it may be an inlet stream for a process, particularly where removing water vapour from the inlet stream is advantageous.

[00045] In certain examples, the graphene oxide aerogel is the aerogel according to the first or third aspect.

[00046] In a sixth aspect of the disclosure there is provided a method of desorbing water adsorbed onto a graphene oxide aerogel, the method comprising the step of sufficiently heating said graphene oxide aerogel thereby releasing said adsorbed water and regenerating said graphene oxide aerogel.

[00047] The heating may be at a temperature of from about 30°C to about 250 °C, or about 40°C to about 250 °C, about 40°C to about 200 °C, about 40°C to about 150 °C, about 40°C to about 100 °C, about 40°C to about 80 °C, or about 40°C to about 60 °C. It may be, for example, at a temperature of about 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 150, 200, or 250°C. In certain examples, the heating may be at room temperature.

[00048] The heating may be for a period of time from about 10 minutes to about 48 hours, or from about 20 minutes to about 48 hours, about 30 minutes to about 48 hours, about 60 minutes to about 48 hours, about 10 minutes to about 24 hours, about 10 minutes to about 12 hours, about 10 minutes to about 8 hours, about 10 minutes to about 6 hours, or about 30 minutes to about 8 hours. It may be, for example, for about 10, 20, 30, 40, or 50 minutes, or for about 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 24, 36, or 48 hours.

[00049] In certain examples, the graphene oxide aerogel is the aerogel according to the first or third aspect.

[00050] In a seventh aspect of the disclosure there is provided a method of recovering water from a graphene oxide aerogel having water adsorbed thereto, the method comprising the step of sufficiently heating said graphene oxide aerogel thereby releasing said adsorbed water, and recovering said water.

[00051] The heating may be at a temperature of from about 30°C to about 250 °C, or about 40°C to about 250 °C, about 40°C to about 200 °C, about 40°C to about 150 °C, about 40°C to about 100 °C, about 40°C to about 80 °C, or about 40°C to about 60 °C. It may be, for example, at a temperature of about 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 150, 200, or 250°C. In certain examples, the heating may be at room temperature.

[00052] The heating may be for a period of time from about 10 minutes to about 48 hours, or from about 20 minutes to about 48 hours, about 30 minutes to about 48 hours, about 60 minutes to about 48 hours, about 10 minutes to about 24 hours, about 10 minutes to about 12 hours, about 10 minutes to about 8 hours, about 10 minutes to about 6 hours, or about 30 minutes to about 8 hours. It may be, for example, for about 10, 20, 30, 40, or 50 minutes, or for about 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 24, 36, or 48 hours.

[00053] The method may comprise a step of cooling a water vapour produced by the heating process. The cooling may be performed at a temperature of about 90 °C or less, or 80, 70, 60, 50, 40, 30, 20, 10, 0, -10, -20, or -30 °C or less.

[00054] In certain examples, the graphene oxide aerogel is the aerogel according to the first or third aspect.

[00055] In an eighth aspect of the disclosure there is provided an atmospheric water generator comprising a graphene oxide aerogel.

[00056] In certain examples, the atmospheric water generator comprises an inlet for introducing a gas stream or atmosphere therein such that the gas stream or atmosphere contacts the graphene oxide aerogel. The gas stream or atmosphere may comprise moisture, and the graphene oxide aerogel may adsorb the moisture from the gas stream or atmosphere.

[00057] In certain examples, the atmospheric water generator further comprises a heating element to heat the graphene oxide aerogel and release the moisture adsorbed thereon. In certain examples, the atmospheric water generator further comprises a cooling element to cool and condense the moisture released in the heating process to form water. The atmospheric water generator may further comprise a container for collecting the condensed water.

[00058] In certain examples, the graphene oxide aerogel is the aerogel according to the first or third aspect.

BRIEF DESCRIPTION OF THE DRAWINGS

[00059] **Figure 1** shows an example GO/Ca^{2+} solution (~30 mL) that has been transferred into a glass petri dish with a diameter of 10 cm for a freeze-drying process (a) top-view; (b) side view.

[00060] **Figure 2** shows an example GO-based aerogel drying in a freeze-dryer.