Method For Making Silica Aerogel Blanket

Technical Field

The present invention relates to a method for fabricating a silica aerogel blanket, an silica aerogel blanket obtained by such a method and the use of incinerator bottom ash in the fabrication of a silica aerogel blanket.

Background Art

Silica aerogel is one of the most common types of aerogel used for the production of aerogel-based insulation materials. Due to the fragile and brittle nature of aerogels, accompanied with its low mechanical strength, the application of the material in the industry is limited. Research has been conducted to improve its mechanical characteristics and properties via the use of fibres to further support the exterior skeletal structure of the aerogel.

Known aerogels have a processing method which consists of incorporating a sol mixture into a fibrous web in an automated roll-to-roll manner. The sol is then catalysed in order to convert it into a gel, prior to subsequent processing steps. The gelation process is then followed by an aging step, which improves the material's strength and critical performance properties, such as thermal conductivity and hydrophobicity. The final step in the process is to dry the wet gel under supercritical CO_2 conditions.

Research has proved that the addition of fibres to aerogel is able to greatly affect the cracking resistance of silica aerogel and is one of the more optimal methods to lessen the brittleness of its matrix. An aerogel blanket is a composite material made of silica aerogel and a fibrous reinforcement. This combination turns the brittle structure of a silica aerogel into a material which is durable, flexible and hydrophobic. Aerogel blankets usually consist of amorphous silica instead of crystalline silica, which reduces the risk of potential health hazards due to exposure.

The large spectrum of materials used for the manufacturing of fibres (Fig. 1) allows for alteration and formation of different parameters and properties of the resulting silica aerogel composite. Depending on the application requirements, the type of fibre reinforcement material used also differs. Polymer fibres provide a significant increase of the elasticity of the silica matrix, with the drawback being the lowering of its resistance to high temperatures. On the other hand, ceramic or glass fibres improves the thermal resistance, but it also increases the density and decreases the specific surface area of the nanocomposite.

The simplest way of aerogel blanket synthesis is through the introduction of short fibres to the solution of the silicon precursor before gelation. An important factor to note during the synthesis stage is the correct timing of gelation, so that a homogeneous structure of the composite is formed and sedimentation of the fibres does not occur. Known silica aerogel blankets are manufactured from organosilicon compounds and glass fibres through the means of supercritical drying in CO_2.

Other products, on the other hand, are based on water glass granules and polymer fibres and are made via drying at atmospheric pressure.

While the prices of the aerogel blankets thus made vary according to its specifications, types and suppliers, for an aerogel blanket measuring 30.5 cm x 38.1 cm, the price may range from US$42 to US$55, depending on the thickness of the blanket (3.5 mm to 8 mm). If the size of the aerogel blanket were to increase, the price would naturally increase as well. Other aerogel blankets range in price from US$4.89 to US$12.16, depending on the type of aerogel blanket the consumer requires (Pyrogel, Cyrogel, Spaceloft) and the thickness of the blanket (5 mm to 10 mm).

The aforementioned methods of fabrication are undoubtedly expensive, and requires the use of high-end equipment and high-quality chemicals. The fabrication methods are also complex, limiting the use of conventional aerogel blankets. In view of the limitations of current methods for preparing aerogel blankets, there is a need for development of a method that overcomes or at least ameliorates, one or more of the disadvantages described above.

Summary

In an aspect, there is provided a method of forming a silica aerogel blanket, comprising the steps of:

i) providing incineration bottom ash;

ii) extracting silica from the incineration bottom ash in the presence of a base to form a water glass;

iii) forming a silica gel from the water glass; and

iv) contacting the silica gel with fibre to form a silica aerogel blanket.

In the method as defined above, the silica aerogel blanket is fabricated by incorporating fibres into the silica aerogel made from incineration bottom ash. Advantageously, this provides a more durable and sturdy material due to its improved exterior skeletal structure. Further advantageously, the method as defined above offers an environmentally friendly and economic way of preparing silica aerogel blankets by recycling solid waste in the form of incineration bottom ash, and converting the solid waste into a high value-added product.

Further advantageously, given that the silica aerogel blanket is fabricated through a more cost-effective fabrication process, with incineration bottom ash used as the starting material, there are significant cost-savings. Moreover, the method as defined as above avoids the use of high-end equipment and expensive starting materials, making it a more affordable option for fabricating silica aerogel blankets. By employing the method as defined above, silica aerogel blankets may be advantageously fabricated in a more efficient and cost-effective manner, and this may in turn result in increased applications of silica aerogel blankets in construction, automotive, cryogenic,

aeronautic and astronautic applications, which may have previously been not possible due to high costs.

In some embodiments, the method uses the technique of microwave synthesis. In other embodiments, the method uses ion exchange chromatography to purify the water glass. In other embodiments, the method uses the technique of aging and crushing the silica gel before contacting with the fibre. Advantageously, these embodiments facilitate to significantly decreases the overall time taken to fabricate the silica aerogel blanket.

In another aspect, there is provided a silica aerogel blanket obtained by the method as defined above.

Advantageously, the silica aerogel blanket fabricated by the method as defined above offers the same properties and advantages as conventionally available aerogel blankets.

In another aspect, there is provided the use of incineration bottom ash in the fabrication of a silica aerogel blanket.

Definitions

The following words and terms used herein shall have the meaning indicated:

“Incineration bottom ash (IBA)” for the purposes of this disclosure refers to a form of ash produced in incineration facilities. IBA may include coal ash, wood chip ash, or may specifically refer to the material that is discharged from the moving grate of municipal solid waste incinerators.

“Aging” for the purposes of this disclosure refers to a phenomenon observed in solid solutions or liquid sols that describes the change of an inhomogeneous structure over time, i.e. small crystals or sol particles dissolve, and redeposit onto larger crystals or sol particles by formation of new and longer crosslinks. In the case of gels, continued gelling of the sol occurs during aging. The term “age” and “aged” should be construed accordingly.

"Alkyl" as a group or part of a group refers to a straight or branched aliphatic hydrocarbon group, preferably a C_1–C_{12} alkyl unless otherwise noted. Examples of suitable straight and branched C_1-C_6 alkyl substituents include methyl, ethyl, n-propyl, 2-propyl, n-butyl, sec-butyl, t-butyl, hexyl, heptyl, octyl and the like. The group may be a terminal group or a bridging group.

The word “substantially” does not exclude “completely” e.g. a composition which is “substantially free” from Y may be completely free from Y. Where necessary, the word “substantially” may be omitted from the definition of the invention.

Unless specified otherwise, the terms "comprising" and "comprise", and grammatical variants thereof, are intended to represent "open" or "inclusive" language such that they include recited elements but also permit inclusion of additional, unrecited elements.

As used herein, the term "about", in the context of concentrations of components of the formulations, typically means +/- 5% of the stated value, more typically +/- 4% of the stated value, more typically +/- 3% of the stated value, more typically, +/- 2% of the stated value, even more typically +/- 1% of the stated value, and even more typically +/- 0.5% of the stated value.

Throughout this disclosure, certain embodiments may be disclosed in a range format. It should be understood that the description in range format is merely for convenience and brevity and should not be construed as an inflexible limitation on the scope of the disclosed ranges. Accordingly, the description of a range should be considered to have specifically disclosed all the possible sub-ranges as well as individual numerical values within that range. For example, description of a range such as from 1 to 6 should be considered to have specifically disclosed sub-ranges such as from 1 to 3, from 1 to 4, from 1 to 5, from 2 to 4, from 2 to 6, from 3 to 6 etc., as well as individual numbers within that range, for example, 1, 2, 3, 4, 5, and 6. This applies regardless of the breadth of the range.

Certain embodiments may also be described broadly and generically herein. Each of the narrower species and subgeneric groupings falling within the generic disclosure also form part of the disclosure. This includes the generic description of the embodiments with a proviso or negative limitation removing any subject matter from the genus, regardless of whether or not the excised material is specifically recited herein.

Detailed Disclosure of Optional Embodiments

There is provided a method of forming a silica aerogel blanket, comprising the steps of:

i) providing incineration bottom ash;

ii) extracting silica from the incineration bottom ash in the presence of a base to form a water glass;

iii) forming a silica gel from the water glass; and

iv) contacting the silica gel with fibre to form a silica aerogel blanket.

The method may further comprise the step of drying the incineration bottom ash at a temperature in the range of about 50 °C to about 100 °C, about 50 °C to about 60 °C, about 50 °C to about 80 °C, about 60 °C to about 80 °C, about 60 °C to about 100 °C, or about 80 °C to about 100 °C before step ii).

The method may further comprise the step of grinding the incineration bottom ash to a particle size of less than 200 µm, less than 150 µm or less than 100 µm before step ii). The method may comprise the step of grinding the incineration bottom ash to a particle size in the range of about 1 µm to about 200 µm, about 1 µm to about 50 µm, about 1 µm to about 100 µm, about 1 µm to about 150 µm , about 50 µm to about 100 µm , about 50 µm to about 150 µm , about 50 µm to about 200 µm , about 100 µm to about 150 µm or about 150 µm to about 200 µm.

The grinding step may be performed before or after the step of drying the incineration bottom ash.

The grinding may be performed in a ball mill containing 7 to 500, 7 to 20, 7 to 50, 7 to 100, 7 to 250, 20 to 50, 20 to 100, 20 to 250, 20 to 500, 50 to 100, 50 to 250, 50 to 500, 100 to 250, 100 to 500, 200 to 500, 7 to 13, 7 to 10, 10 to 13 or 8 to 12 metal balls. The number of balls may depend on the size of the ball mill. The balls may have a diameter in the range of about 5 mm to about 10 cm, about 5 mm to about 1cm, about 5 mm to about 2 cm, about 5 mm to about 5 cm, about 1 cm to about 2 cm, about 1 cm to about 5 cm, about 1 cm to about 10 cm, about 2 cm to about 5 cm, about 2 cm to about 10 cm or about 5 cm to about 10 cm. The balls may comprise a metal, zirconia, tungsten carbide, alumina, agate, Teflon or any mixture thereof, or may be an iron-core coated with polyurethane. The ball mill may be operated a rotational speed in the range of about 200 rpm to about 400 rpm, about 200 rpm to about 300 rpm, about 300 rpm to about 400 rpm, or at about 300 rpm. The ball mill may be operated for a duration in the range of about 10 minutes to about 20 minutes, about 10 minutes to about 15 minutes, about 15 minutes to about 20 minutes, or for about 15 minutes.

The method may further comprise the step of pre-treating the incineration bottom ash with a pre-treatment acid. The pre-treatment step may be performed before the step of extracting silica from the incineration bottom ash.

The pre-treatment acid in the pre-treating step may be a strong acid. The strong acid may be hydrochloric acid, nitric acid or sulfuric acid. The strong acid may be hydrochloric acid, and may be present during the pre-treating step at a concentration in the range of about 1.5 M to about 2.5 M, about 1.5 M to about 2 M or about 2 M to about 2.5 M. The concentration of the hydrochloric acid may be about 2 M.

The pre-treatment step may further comprise the step of stirring the incineration bottom ash with the acid at room temperature at a stirring speed in the range of about 200 rpm to about 400 rpm, about 200 rpm to about 300 rpm or about 300 rpm to about 400 rpm, or at about 300 rpm, for a duration in the range of about 18 hours to about 36 hours, about 18 hours to about 24 hours, about 24 hours to about 36 hours, or for about 24 hours.

The pre-treatment step may further comprise the step of irradiating the incineration bottom ash in the presence of the acid with microwaves at a temperature in the range of about 160 °C to about 200 °C, about 160 °C to about 180 °C or about 180 °C to about 200 °C, or at about 180 °C, for a duration in the range of about 30 minutes to about 2 hours, about 30 minutes to about 1 hour, or about 1 hour to about 2 hours, or for about 1 hour.

Once the pre-treatment step is complete, the pre-treatment acid may be removed. The incineration bottom ash may then be rinsed with water, preferably deionised water, until a pH in the range of about 4 to about 7, about 4 to about 5, about 4 to about 6, about 5 to about 6, about 5 to about 7 or about 6 to about 7 is achieved.

The base in the extraction step may be a strong base. The strong base may be sodium hydroxide, potassium hydroxide, barium hydroxide, calcium hydroxide or any mixture thereof.

The extraction step may be described by the following formula (I):

$2M(OH)_a+aSiO_2 = M_2(SiO_3)_a+aH_2O$ (I),

where M may be a metal and a may be the ionic charge of the metal. M may be sodium, potassium, barium, calcium or any mixture thereof.

To perform the extraction step, Y g of incineration bottom ash having X percentage by weight of silicon (0<X<1) may be provided.

The mass of the base (m_{base}) to be used in the extraction step may be described by the following equation (Ia):

$$m_{base} = \frac{Y \times X}{30 \times a} \times MW_{base} \quad \text{(Ia)}$$

wherein MW_{base} may be the molecular weight of the base.

The volume of the base dissolved in deionised water to form a solution (V_{base}), to be used in the extraction step may be described by the following equation (Ib):

$$V_{base} = \frac{Y \times X}{30 \times a \times M_{base}} \quad \text{(Ib)},$$

wherein M_{base} may be the molar concentration of the base, typically about 2 M to about 6 M.

The strong base may be contacted with Y g of incineration bottom ash in a range of Y mL to 10Y mL of the strong base, wherein the concentration of the strong base is in the range of about 2 M to about 6 M, where Y is the mass in grams of the incineration bottom ash. The base may be sodium hydroxide, and to extract the silica, the sodium hydroxide may be contacted with X g of incineration bottom ash in a solution of $\frac{10}{7}$X g of sodium hydroxide dissolved in $\frac{X}{112}$ L of deionised water, where X is the mass in grams of the incineration bottom ash.

The extraction step may further comprise the step of refluxing the incineration bottom ash with the base at a temperature in the range of about 140 °C to about 180 °C, about 140 °C to about 160 °C, about 160 °C to about 180 °C, or at about 160 °C, for a duration in the range of about 18 hours to about 36 hours, about 18 hours to about 24 hours, about 24 hours to about 36 hours, or for about 24 hours.

The refluxing may be done using a condenser to prevent the loss of solvent or water during the extraction step.

The extraction step may further comprise the step of irradiating the incineration bottom ash in the presence of the base with microwaves at a temperature in the range of about 160 °C to about 220 °C, about 160 °C to about 180 °C or about 180 °C to about 220 °C, or at about 180 °C, for a duration in the range of about 30 minutes to about 2 hours, about 30 minutes to about 1 hour, or about 1 hour to about 2 hours, or for about 1 hour.

The extraction step may further comprise the step of reacting the incineration bottom ash in the presence of the base under pressure at a temperature in the range of about 160 °C to about 200 °C, about 160 °C to about 180 °C or about 180 °C to about 200 °C, or at about 180 °C, at a pressure in the range of about 0.5 MPa to about 2 MPa, about 0.5 MPa to about 1 MPa or about 1 MPa to about 2 MPa, or at about 1 MPa, for a duration in the range of about 30 minutes to about 4 hours, about 30 minutes to about 2 hour, or about 2 hour to about 4 hours, or for about 2 hours. During the extraction step, a plastic container may be used instead of a glass container to hold the incinerator bottom ash and the base, to prevent any reaction between the base and the glass container.

Water glass may be a solution of sodium silicate (Na_2SiO_3) in water.

The method may further comprise the step of filtering the water glass after step iii).

The filtration may be performed using an about 0.1 µm to about 12 µm, about 0.1 µm to about 0.1 µm to about 0.2 µm , about 0.1 µm to about 0.45 µm, about 0.1 µm to about 1 µm, about 0.1 µm to about 11 µm, about 0.2 µm to about 0.45 µm, about 0.2 µm to about 1 µm, about 0.2 µm to about 2 µm, about 0.2 µm to about 11 µm, about 0.2 µm to about 12 µm , about 0.45 µm to about 1 µm, about 0.45 µm to about 2 µm, about 0.45 µm to about 11 µm, about 0.45 µm to about 12 µm, about 1 µm to about 2 µm, about 1 µm to about 11 µm, about 1 µm to about 12 µm, about 2 µm to about 11 µm, about 2 µm to about 12 µm, or about 11 µm to about 12 µm, or a 0.2 µm , 2 µm, 0.45 µm, 1 µm or 11 µm filter membrane.

The method may further comprise the step of diluting the water glass in deionized water at a ratio in the range of about 1:2 to about 3:4 by volume, preferably at a ratio of about 2:3 by volume, before contacting with the fibre.

The method may further comprise the step of adding a pH-adjusting acid to the diluted water glass until the pH is in the range of about 8 to about 11, about 8 to about 9, about 8 to about 10, about 9 to about 10, about 9 to about 11 or about 10 to about 11, to form a silica gel before contacting with the fibre.

The addition of the pH-adjusting acid may be done slowly, or drop-by-drop.

The water glass may begin to form a gel as soon as the pH of the diluted water glass is adjusted to be in the range of about 8 to about 11. The silica gel may refer to the water glass as soon as it begins to form the gel. The viscosity of the silica gel may be in the range of about 20 millipoise to about 50 millipoise, about 20 millipoise to about 25 millipoise, about 20 millipoise to about 35 millipoise, about 20 millipoise to about 45 millipoise, about 25 millipoise to about 35 millipoise, about 25 millipoise to about 45 millipoise, about 25 millipoise to about 50 millipoise, about 35 millipoise to about 45 millipoise, about 35 millipoise to about 50 millipoise or about 45 millipoise to about 50 millipoise.

The pH-adjusting acid may be a strong acid, a weak acid or a mixture thereof. The strong acid may be hydrochloric acid, nitric acid, sulfuric acid or any mixture thereof. The weak acid may be selected from the group consisting of phosphoric acid, acetic acid, tartaric acid, oxalic acid and any

mixture thereof. The strong acid may be hydrochloric acid, and may be at a concentration in the range of about 0.5 M to about 5 M, about 0.5 M to about 1 M, about 0.5 M to about 2.5 M, about 0.5 M to about 4 M, about 1 M to about 2.5 M, about 1 M to about 4 M, about 1 M to about 5 M, about 2.5 M to about 4 M, about 2.5 M to about 5 M or about 4 M to about 5 M. The concentration of the acid may be about 1 M or about 4 M or both.

The method may further comprise the step of purifying the diluted water glass on a strongly acidic ion exchange resin to collect silicic acid. The strongly acidic ion exchange resin may facilitate removal of contaminant anions such as Na^+, Ca^{2+}, Ba^{2+} and Al^{3+}. The strongly acidic anion exchange resin may be Amberlite™ IR-120H, Purolite® C100, Indion 730 or a resin in the Diaion™ series. The method may further comprise the step of adding a pH-adjusting base to the silicic acid until the pH is in the range of about 3 to about 6, about 3 to about 4, about 3 to about 5, about 4 to about 5, about 4 to about 6 or about 5 to about 6, to form a silica gel.

The water glass may begin to form a gel as soon as the pH of the silicic acid is adjusted to be in the range of about 3 to about 6. The silica gel may refer to the water glass as soon as it begins to form the gel.

The pH-adjusting base may be ammonia. The ammonia may be in the form of a solution. The ammonia solution may have a concentration in the range of about 0.3 M to about 0.7 M, about 0.3 M to about 0.5 M or about 0.5 M to about 0.7 M, or at about 0.5 M.

The method may further comprise the step of aging the silica gel before or after contacting with the fibre.

The aging process may increase modulus and viscosity of the silica gel, but may reduce subsequent shrinkage of the silica gel during the drying process.

The aging step may be performed after contacting the silica gel with the fibre.

If the aging step is performed after contacting the silica gel with the fibre, the silica gel may be contacted with the fibre immediately after the diluted water glass reaches the pH in the range of about 8 to about 11 to form the aerogel blanket.

If the aging step is performed after contacting the silica gel with the fibre, the method may further comprise the step of removing a salt after aging.

The salt may be any water-soluble salt that may be present in the as-formed aerogel blanket. The salt may be a water-soluble salt formed between any one of the cations, Li^+, Na^+, K^+, NH^{4+}, Ca^{2+}, Mg^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+}, Ba^{2+}, Ti^{4+}, Zn^{2+}, Cu^{2+}, Mn^{2+}, or Cr^{3+}, and any one of the anions Cl^-, I^-, F^-, Br^-, OH^-, NO_3^-, S^{2-} or O^{2-}. There may be multiple salts.

The step of removing the salt may improve the optical transmission properties and decrease the density of the silica aerogel.

The step of salt removal may comprise the step of immersing the silica aerogel blanket in water for a duration in the range of about 1 hour to about 15 hours, about 1 hour to about 3 hours, about 1 hour to about 5 hours, about 1 hour to about 7 hours, about 1 hour to about 9 hours, about 1 hour to about 11 hours, about 1 hour to about 13 hours, about 3 hours to about 5 hours, about 3 hours to about 7 hours, about 3 hours to about 9 hours, about 3 hours to about 11 hours, about 3 hours to about 13 hours, about 3 hours to about 15 hours, about 5 hours to about 7 hours, about 5 hours to about 9 hours, about 5 hours to about 11 hours, about 5 hours to about 13 hours, about 5 hours to about 15 hours, about 7 hours to about 9 hours, about 7 hours to about 11 hours, about 7 hours to about 13 hours, about 7 hours to about 15 hours, about 9 hours to about 11 hours, about 9 hours to about 13 hours, about 9 hours to about 15 hours, about 11 hours to about 13 hours, about 11 hours to about 15 hours, about 13 hours to about 15 hours, or for about 5 hours. During this period, the water may be exchanged 1, 2, 3, 4 or 5 times. The water may be deionised water.

If the aging step is performed after contacting the silica gel with the fibre, the method may further comprise the step exchanging the solvent after aging. The step of solvent exchange may be for exchanging the water that is present the silica aerogel blanket following the salt removal step, with an organic solvent. The organic solvent may have a low surface tension which may reduce capillary pressure and prevent gel shrinkage and collapse of the pore walls formed in the aerogel. The organic solvent may also facilitate the surface modification step to occur more efficiently.

The step of solvent exchange may be performed in an exchange solvent comprising alcohol, an alkyl having 6 or more carbon atoms, acetone, xylene or any mixture thereof. The alcohol may be methanol, ethanol, isopropyl alcohol, t-butanol or any mixture thereof. The alkyl having 6 or more carbon atoms may be n-hexane, n-heptane, n-octane or any mixture thereof.

The step of solvent exchange may be performed at a temperature in the range of about 40 °C to about 60 °C, or at about 50 °C.

If the aging step is performed before contacting the silica gel with the fibre, the method may further comprise the step of crushing the aged silica gel into a powder before contacting with the fibre. The crushed powder may have a particle size in the range of about 2 µm to about 1200 µm, about 2 µm to about 10 µm, about 2 µm to about 100 µm, about 2 µm to about 200 µm, about 2 µm to about 500 µm, about 2 µm to about 1200 µm, about 10 µm to about 100 µm, about 10 µm to about 200 µm, about 10 µm to about 500 µm, about 10 µm to about 1200 µm, about 100 µm to about 200 µm, about 100 µm to about 500 µm, about 100 µm to about 1200 µm, about 200 µm to about 500 µm, about 200 µm to about 1200 µm or about 500 µm to about 1200 µm.

By crushing the aged silica gel into a powder, the step of salt removal and the step of solvent exchange may be omitted, making the overall fabrication process of the silica aerogel shorter. Further, by crushing the aged silica gel into a powder, the surface area of the aged silica gel may be increased, thereby allowing the surface modification step to be performed more efficiently.

The aging may be done in the presence of radiation, preferably UV or IR radiation**.** The UV radiation may have a wavelength in the range of about 100 nm to about 420 nm, about 100 nm to about 200 nm, about 100 nm to about 300 nm, about 200 nm to about 300 nm, about 200 nm to

about 420 nm, or about 300 nm to about 420 nm. The UV radiation may have an intensity in the range of about 5 W/cm^2 to about 15 W/cm^2, 5 W/cm^2 to about 10 W/cm^2, about 10 W/cm^2 to about 15 W/cm^2. The IR radiation may be in the range of about 720 nm to about 2400 nm, about 720 nm to about 1500 nm, about 720 nm to about 2000 nm, about 1500 nm to about 2000 nm, about 1500 nm to about 2400 nm or about 2000 nm to about 2400 nm. The IR radiation may have a n intensity in the range of about 10 W/cm^2 to about 100 W/cm^2, about 10 W/cm^2 to about 25 W/cm^2, about 10 W/cm^2 to about 50 W/cm^2, about 25 W/cm^2 to about 50 W/cm^2, about 25 W/cm^2 to about 100 W/cm^2, about 50 W/cm^2 to about 100W/cm^2.

The aging may be done for a duration in the range of about 30 minutes to about 4 hours. If the aging is done in the absence of radiation, the aging may be done for a duration in the range of about 2 hours to about 4 hours, about 2 hours to about 3 hours or about 3 hours to about 4 hours, or for about 3 hours. If the aging is done in the presence of radiation, the aging may be done for a duration in the range of 30 minutes to about 1.5 hours, about 30 minutes to about 1 hour, about 1 hour to about 1.5 hours or for about 1 hour.

Aged silica gel may refer to silica gel that has undergone the process of aging and has been substantially completely aged. The aged silica gel may have a hardness in the range of about 10 kPa to about 500 kPa, about 10 kPa to about 20 kPa, about 10 kPa to about 50 kPa, about 10 kPa to about 100 kPa, about 10 kPa to about 200 kPa, about 20 kPa to about 50 kPa, about 20 kPa to about 100 kPa, about 20 kPa to about 200 kPa, about 20 kPa to about 500 kPa, about 50 kPa to about 100 kPa, about 50 kPa to about 200 kPa, about 50 kPa to about 500 kPa, about 100 kPa to about 200 kPa, about 100 kPa to about 500 kPa or about 200 kPa to about 500 kPa.

The method may further comprise the step of surface modifying the aged silica gel before or after contacting with the fibre.

If the aging is performed after contacting with the fibre, then the surface modification is also done after contacting with the fibre. If the aging is performed before contacting with the fibre, then the surface modification is also done before contacting with the fibre.

The step of surface modification may be performed by reacting the aged silica gel with an organosilicon compound. The organosilicon compound may be trialkylsilyl halide, wherein the alkyl group may be methyl, ethyl or propyl and the halide may be chloride, bromide or iodide. The surface modification may be performed by reacting the aged silica gel with trimethylsilyl chloride (TMCS), hexamethyldislioxane (HMDSO), hexamethyldisilazane (HMDZ), trimethylmethoxysilane (TMMS), phenyltrimethoxysilane (PTMS), phenyltriethoxysilane (PTES), vinlytriethoxysilane (VTMS), methyltrimethoxysilane (MTMS) or any combination thereof.

The organosilicon compound may react with hydroxy groups on the surface of the aged silica gel to functionalise the aged silica gel with an oganosilicon group such as a trialkylsilyl group. Advantageously, the surface modification may cause the aged silica gel to become hydrophobic.

The step of surface modification may be performed after contacting the silica gel with the fibre, and the silica aerogel blanket may be immersed in a mixture of organosilicon compound, an alcohol and an alkyl having 6 or more carbon atoms, wherein:

- the molar ratio of organosilicon compound:silica is in the range of about 3:1 to about 1:1;

- the molar ratio of organosilicon compound:alcohol is in the range of about 2:1 to about 1:2; and

- the ratio of alkyl having 6 or more carbon atoms: organosilicon compound by volume is in the range of about 10:1 to about 15:1.

The step of surface modification may be performed after contacting the silica gel with the fibre, and the silica aerogel blanket may be immersed in a mixture of TMCS, ethanol and n-hexane, wherein the molar ratio of TMCS:silica may be in the range of about 3:1 to about 1:1, or at a ratio of about 2:1, the molar ratio of TMCS:ethanol may be in the range of about 2:1 to about 1:2, or at a ratio of about 1:1, and the ratio of hexane:TMCS by volume may be in the range of about 10:1 to about 15:1, or at a ratio of about 25:2.

The step of surface modification may be performed before contacting the silica gel with the fibre, and the aged silica gel may be immersed in a mixture of organosilicon compound in a surface modification solvent, wherein:

- the ratio of the surface modification solvent:aged silica gel is about 1:1 to about 6:1 by volume; and

- the molar ratio of silica:organosilicon compound is in the range of about 1:10 to about 1:2.

The step of surface modification may be performed before contacting the silica gel with the fibre, and the aged silica gel may be immersed in a mixture of organosilicon compound and a surface modification solvent, wherein the ratio of surface modification solvent:aged silica gel may be about 1:1 to about 6:1, about 1:1 to about 2:1, about 1:1 to about 3:1, about 1:1 to about 4:1, about 1:1 to about 5:1, about 2:1 to about 3:1, about 2:1 to about 4:1, about 2:1 to about 5:1, about 2:1 to about 6:1, about 3:1 to about 4:1, about 3:1 to about 5:1, about 3:1 to about 6:1, about 4:1 to about 5:1, about 4:1 to about 6:1 or about 5:1 to about 6:1 by volume.

The ratio of the alkyl having 6 or more carbon atoms:alcohol:aged silica gel may be about 10:6:3 by volume.

The step of surface modification may be performed before contacting the silica gel with the fibre, and the aged silica gel may be immersed in a mixture of TMCS and a surface modification solvent comprising IPA and n-hexane, wherein the ratio of n-hexane:IPA:aged silica gel may be about 10:6:3 by volume and the molar ratio of silica:TMCS may be in the range of about 1:10 to about 1:2, or at a ratio of about 1:4.

For the surface modification step, the surface modification solvent may comprise an alcohol, an alkyl having 6 or more carbon atoms, acetone, xylene or any mixture thereof. The alcohol may be methanol, ethanol, isopropyl alcohol, t-butanol, or any mixture thereof and the alkyl having 6 or more carbon atoms may be n-hexane, n-heptane, n-octane or any mixture thereof.

The step of surface modification may be performed at a temperature in the range of about 30 ˚C to about 50 ˚C, about 30 ˚C to about 40 ˚C or about 40 ˚C to about 50 ˚C, or at about 40 ˚C, or at about 50 ˚C for a duration in the range of about 2 hours to about 36 hours, about 2 hours to about 4 hours, about 2 hours to about 3 hours, about 3 hours to about 4 hours, about 2 hours to about 12 hours, about 2 hours to about 18 hours, about 2 hours to about 24 hours, about 12 hours to about 18 hours, about 12 hours to about 24 hours, about 12 hours to about 36 hours, about 18 hours to about 24 hours, about 18 hours to about 36 hours or about 24 hours to about 36 hours, with or without stirring.

The stirring may be performed at a stirring speed of less than about 400 rpm, less than about 300 rpm, less than about 200 rpm, less than about 100 rpm or less than about 50 rpm. The stirring may be performed at a stirring speed in the range of about 50 rpm to about 200 rpm, about 50 rpm to about 400 rpm or about 200 rpm to about 400 rpm.

If the surface modification step is performed after contacting with the fibre, then the surface modification may be performed at a temperature at about 50 ˚C for a duration of about 24 hours.

If the surface modification step is performed before contacting with the fibre, and the aged silica gel has been crushed into a powder before contacting with the fibre, then the surface modification may be performed at a temperature at about 40 ˚C for a duration of about 3 hours.

The surface modified silica gel may be separated from the surface modification solvent and dispersed in an alcohol or an alkyl having 6 or more carbon atoms before being contacted with the fibre.

The surface modified silica gel may be separated from the surface modification solvent and dispersed in IPA, ethanol or n-hexane before being contacted with the fibre.

The silica gel before surface modification may comprise water in its matrix. During the surface modification step, the water that is present in the matrix of the silica gel may be displaced and replaced with the surface modification solvent. As water is displaced from the silica gel matrix, the surface modification solvent that is not in the silica gel matrix may mix with the displaced water to form an aqueous solution. The surface modified silica gel, whereby the water has been displaced with the surface modification solvent, may therefore float on the surface of the aqueous solution, as the surface modified silica gel comprising the surface modification solvent in its matrix may become less dense than the aqueous solution. Separation of the surface modified silica gel may therefore be performed by removing the aqueous solution by layer separation, evaporation or filtration.

The surface modified silica gel may be dispersed in IPA, ethanol or hexane at a volume of about 0.5 to about 50 times the volume of the surface modified silica gel.

Surface modified silica gel may refer to silica gel that has been at least partially or substantially completely surface modified. Surface modified silica gel may be at least about 90%, about 95% or about 98% surface modified. Surface modified silica gel may be about 90% to 100%, about 95% to 100%, or about 98% to 100% surface modified. Surface modified silica gel may have been at least partially or substantially completely surface modified with trialkylsilanol.

The silica aerogel blanket may be dried at a temperature in the range of about 40 °C to about 60 °C, or at about 50 °C, for a duration in the range of about 30 minutes to about 1.5 hours, about 30 minutes to about 1 hour, about 1 hour to about 1.5 hours, or for about 1 hour, then at a temperature in the range of about 180 °C to about 220 °C, or at about 200°C, for a further duration in the range of about 30 minutes to about 1.5 hours, about 30 minutes to about 1 hour, about 1 hour to about 1.5 hours, or for about 1 hour.

Different types of fibre reinforcement may be used to reinforce a silica aerogel. The use of fibres may improve the compressive strength of the aerogel blanket along with decreasing its bulk density. The fibre may be inorganic or organic.

The silica gel may be contacted with a fibre to form a silica aerogel blanket. The contacting may be performed at a volume ratio of silica gel:fibre of 1:10 or higher. The contacting may be performed at a volume ratio of silica gel:fibre of 1:10, 1:5, 2:9, 1:4, 2:7, 1:3, 2:5, 1:2, 2:3, 1:1, 3:2, 2:1, 5:2, 3:1, 7:2, 4:1, 9:2, 5:1, 10:1, 20:1 or 50:1 or greater. The contacting may be performed at a volume ratio of silica gel:fibre in the range of about 1:10 to about 50:1, about 1:10 to about 1:5, about 1:10 to about 1:2, about 1:10 to about 1:1, about 1:10 to about 2:1, about 1:10 to about 5:1, about 1:10 to about 10:1, about 1:10 to about 25:1, about 1:5 to about 1:2, about 1:5 to about 1:1, about 1:5 to about 2:1, about 1:5 to about 5:1, about 1:5 to about 10:1, about 1:5 to about 25:1, about 1:5 to about 50:1, about 1:2 to about 1:1, about 1:2 to about 2:1, about 1:2 to about 5:1, about 1:2 to about 10:1, about 1:2 to about 25:1, about 1:2 to about 50:1, about 1:1 to about 2:1, about 1:1 to about 5:1, about 1:1 to about 10:1, about 1:1 to about 25:1, about 1:1 to about 50:1, about 2:1 to about 5:1, about 2:1 to about 10:1, about 2:1 to about 25:1, about 2:1 to about 50:1, about 5:1 to about 10:1, about 5:1 to about 25:1, about 5:1 to about 50:1, about 10:1 to about 25:1, about 10:1 to about 50:1, or about 25:1 to about 50:1.The fibre may be selected from the group consisting of glass fibres, microfibres, mineral microfibres, ceramic micro and nanofibres, nanotubes, polypropylene microfibres, aramid microfibres, carbon micro and nanofibres, carbon nanotubes and cotton nanofibres. The fibre may be glass fibre or glass microfibres.

In particular, inorganic glass or ceramic fibre reinforcement may be suitable for insulation under high temperatures.

There may be many advantages to using glass fibres. The principal advantage of using glass fibres may be their high performance per cost ratio. Further, the material itself may have many attractive properties. Besides exhibiting excellent thermal and acoustic insulating properties, glass fibres may have good impact resistance and high strength-to-weight ratio. Glass fibres are also known to be transparent to radio waves and may be used in applications involving antennas and radars.

Further, glass fibres are known to be used as reinforcement for plastic materials and has been found to increase the material's tensile strength, flex modulus, creep resistance, impact resistance, dimensional stability, heat, and chemical resistance. In this regard, composite materials reinforced with glass fibres may have useful properties as good electrical and thermal insulators.

The fibre may be in the form of separate strands or may be woven into a sheet.

The fibre may comprise fibre strands. Each fibre strand may have a length in the range of about 5 mm to about 50 mm, about 5 mm to about 10 mm, about 5 mm to about 25 mm, about 10 mm to about 25 mm, about 10 mm to about 50 mm, about 25 mm to about 50 mm. Each fibre strand may have a diameter in the range of about 3 µm to about 5 µm, about 3 µm to about 4 µm or about 4 µm to about 5 µm.

If the fibre is woven into a sheet, the sheet may have a thickness in the range of about 5 mm to about 50 mm, about 5 mm to about 10 mm, about 5 mm to about 20 mm, about 10 mm to about 20 mm, about 10 mm to about 50 mm or about 20 mm to about 50 mm.

If the fibre is woven into a sheet, the sheet may have a density in the range of about 100 kg/m^3 to about 150 kg/m^3, about 100 kg/m^3 to about 120 kg/m^3, or about 120 kg/m^3 to about 150 kg/m^3.

The fibre may be glass fibre and may comprise alumina, silica or a mixture thereof.

The method may further comprise the step of heating the fibre at a temperature in the range of about 350 °C to about 500 °C, about 350 °C to about 400 °C, about 350 °C to about 400 °C, about 400 °C to about 450 °C, about 400 °C to about 500 °C, or about 450 °C to about 500 °C before step iv).

The method may further comprise the step of washing and wringing the fibre after the heating step. The washed fibre may be wrung dry until the weight of the fibre is in the range of about 100% to about 300%, about 100% to about 200% or about 200% to about 300% of the weight of the fibre before washing.

The method may further comprise the step of placing the fibre into a mould before contacting with the silica gel.

The mould may be made of polytetrafluoroethylene, perfluoroalkoxy alkane, polyvinylidene fluoride, polypropylene, polyethylene, glass, or any mixture thereof.

The fibre may be in the form of fibre woven into a sheet and may be rolled into a cylinder before being placed into the mould.

The method of forming a silica aerogel blanket may comprise the steps of:

i) providing incineration bottom ash;

ii) drying the incineration bottom ash at a temperature in the range of about 50 °C to about 100 °C;

iii) grinding the incineration bottom ash to a particle size of less than 200 µm;

iv) pre-treating the incineration bottom ash with a pre-treatment acid, comprising the step of stirring the incineration bottom ash with the acid at room temperature at a stirring speed in the range of about 200 rpm to about 400 rpm for a duration in the range of about 18 hours to about 36 hours;

v) extracting silica from the treated incineration bottom ash in the presence of a base to form a water glass, comprising the step of refluxing the treated incineration bottom ash with the base at a temperature in the range of about 140 °C to about 180 °C for a duration in the range of about 18 hours to about 36 hours;

vi) diluting the water glass in deionized water at a ratio in the range of about 1:2 to about 3:4 by volume, before contacting with the fibre;

vii) adding a pH-adjusting acid to the diluted water glass until the pH is in the range of about 8 to about 11, to form a silica gel, before contacting with the fibre;

viii) contacting the silica gel with the fibre to form a silica aerogel blanket;

ix) aging the silica gel after contacting with the fibre;

x) removing a salt after aging;

xi) exchanging a solvent after aging; and

xii) surface modifying the aged silica gel after contacting with the fibre.

The steps may be performed in the order as indicated above.

The method of forming a silica aerogel blanket may comprise the steps of:

i) providing incineration bottom ash;

ii) drying the incineration bottom ash at a temperature in the range of about 50 °C to about 100 °C;

iii) grinding the incineration bottom ash to a particle size of less than 200 µm;

iv) pre-treating the incineration bottom ash with a pre-treatment acid, comprising the step of irradiating the incineration bottom ash in the presence of the acid with microwaves at a temperature in the range of about 160 °C to about 200 °C for a duration in the range of about 30 minutes to about 2 hours;

v) extracting silica from the treated incineration bottom ash in the presence of a base to form a water glass, comprising the step of irradiating the treated incineration bottom ash in the

presence of the base with microwaves at a temperature in the range of about 180 °C to about 220 °C for a duration of about 30 minutes to about 2 hours;

vi) diluting the water glass in deionized water at a ratio in the range of about 1:2 to about 3:4 by volume, before contacting with the fibre;

vii) purifying the water glass on a strongly acidic ion exchange resin to collect silicic acid;

viii) adding a pH-adjusting base to the silicic acid until the pH is in the range of about 3 to about 6 to form a silica gel;

ix) aging the silica gel before contacting with the fibre;

x) crushing the aged silica gel into a powder before contacting with the fibre;

xi) surface modifying the aged silica gel before contacting with the fibre; and

xii) contacting the silica gel with the fibre to form a silica aerogel blanket.

The steps may be performed in the order as indicated above.

The method comprising the steps of forming the silica precursor through a sol-gel step, crushing the aged silica gel followed by surface modification before contacting with fibre may facilitate the direct fabrication of the silica aerogel blanket without having to add any emulsifying compounds to the silica aerogel blanket which may compromise its properties. The process may be significantly more facile and straight-forward, and the dispersion of silica gel may be significantly more uniform than when the hydrophobic aerogel particles are dispersed in water.

The method may facilitate the fabrication of a silica aerogel blanket without the use of any emulsifiers. The emulsifier may be an emulsion polymer such as latex, epoxy and silane coupling agents such as γ-aminopropyltriethoxysilane (APTES), γ-glycidoxypropyltrimethoxysilane (GPTMS), γ-methacryloxypropyltrimethoxysilane (MPTMS) and vinyltriethoxysilane (VTES). There is also provided a silica aerogel blanket obtained by the method as defined above.

There is also provided the use of incineration bottom ash in the fabrication of a silica aerogel blanket. The use my comprise the method as defined above.

Brief Description of Drawings

The accompanying drawings illustrate a disclosed embodiment and serve to explain the principles of the disclosed embodiment. It is to be understood, however, that the drawings are designed for purposes of illustration only, and not as a definition of the limits of the invention.

Fig. 1

[Fig. 1] is a scheme showing the types of fibre reinforcement available for aerogel blankets known from the prior art.

Fig. 2

[Fig. 2] shows a flowchart of a typical process for fabricating an aerogel blanket from incineration bottom ash (IBA). The whole process consists of 2 steps: Step 1, fabrication of water glass solution, and step 2, fabrication of aerogel blanket.

Fig. 3

[Fig. 3] shows a schematic diagram of the steps involved in an embodiment of the process for fabricating an aerogel blanket, comprising the steps of A) tightly rolling the glass fibre (302) into a cylindrical shape (304) after the glass fibre has been heated at 450°C for an hour, so that there are no gaps in-between the layers of the rolled-up glass-fibres and that it is tightly rolled; B) placing the tightly rolled up glass fibre (304) into a PTFE mould (306) to undergo gelation and aging; C) pouring the homogenous water glass precursor (308) immediately into the PTFE mould (306) containing the glass fibre (304) before gelation of the precursor (308) within the glass beaker (310) starts and D) covering the top of the PTFE mould (306) with a lid (312) to allow the product to undergo gelation and aging (approximately 2-3 hours).

Fig. 4

[Fig.4] shows a schematic of the modified aerogel fabrication process.

Examples

Non-limiting examples of the invention will be further described in greater detail by reference to specific Examples, which should not be construed as in any way limiting the scope of the invention.

Incineration bottom ash was obtained from Sembcorp Industry (Singapore) and EnGro Co. (Singapore). Hydrochloric acid, 37%, sodium hydroxide, >97%, trimethylsilyl chloride (TMCS), >98%, n-hexane, 95%, isopropyl alcohol, 99.5%, and Amberlite® IRC120 H, hydrogen form, strongly acidic were obtained from Sigma-Aldrich (Merck Pte. Ltd., Singapore).

Example 1: Preparation Of Water Glass (Step 1)

Step 1 of the aerogel fabrication process involved the preparation of the water glass (Fig. 2). Raw incineration bottom ash (IBA) was collected from the incineration plant. Firstly, they were dried at 60 to 80°C and retrieved once they were lighter in colour or when the stench was removed. Afterwards, the IBA was checked for any metallic debris (for example nails, batteries, bolts, and other metallic objects) present and they were removed. A ball milling container containing 8-12 large metal balls, was filled with the IBA to the brim, then the planetary ball milling machine

(Planetary Mill PULVERISETTE 5 Classic Line (Fritsch (Idar-Oberstein, Germany)) was used to grind the IBA to reduce particle size at 300 rpm for approximately 15 minutes. Once accomplished, the grinded IBA was sieved using a sieving machine (Endecotts (London, UK)), through an auto-sieve using a 150 µm sieve to obtain the IBA with a particle size of less than 150 µm.

The chemical treatment was conducted by stirring 9 L of 2 M hydrochloric acid (HCl) leaching solution, in a 10 L container (Radleys (Essex, UK)) with 0.5kg of IBA after size reduction. The mixture was stirred at 300 rpm for approximately 24 hours. After the 24-hour time period, the leachate was then removed, and the residual ash was rinsed with deionised water until a pH of 5 to 6 was achieved.

The silica extraction process was carried out by reacting the sodium hydroxide solution (NaOH) with IBA after chemical treatment for 24 hours at 160°C (under reflux). The sodium hydroxide solution was prepared by dissolving $\frac{10}{7}$X g of sodium hydroxide tablets with $\frac{X}{112}$ L of deionised water, where X is the mass in grams of the IBA after chemical treatment. It should be noted that the IBA after chemical treatment should only be added after the sodium hydroxide tablets are fully dissolved in the deionised water.

The silica extraction process was carried out with a condensing unit and condenser to prevent the loss of water due to the high temperature being used. It works by condensing the evaporated water and adding it back into the mixture while the process is occurring concurrently.

It should also be noted that a plastic container was used instead of a glass container to prevent any reaction between the sodium hydroxide and glass.

After the 24-hour reaction period, the mixture was allowed to cool down until a temperature of 50°C or below, before filtration was attempted. The filtration process was executed by pouring the mixture through a filtration system with a 2 µm filter membrane. This separated the residue from the sodium silicate solution (Na_2SiO_3), which is otherwise known as water glass.

Example 2: Preparation Of Aerogel Blanket (Step 2)

Step 2 of the aerogel fabrication process involved the preparation of the aerogel blanket using the water glass prepared in Step 1 (Fig. 2). Glass fibre having a fibre diameter of 3 to 5 µm, a thickness of 5 to 50 mm, density of 100 to 150 kg/m^3 and comprising alumina and silica was first heated at 450°C for 1 hour. Then the glass fibre (302) was washed in deionized water and wrung tightly before being tightly rolled into a cylindrical shape (304) (Fig. 3A) and placed into a polytetrafluoroethylene (PTFE) mould (306) (Fig. 3B). The moist rolled-up glass fibre should have no gaps and be tightly rolled.

The water glass was diluted in deionised water at a ratio of 2:3 by volume (e.g. 20 ml of water glass to 30 ml of deionised water) and mixed well within the container (Radleys (Essex, UK)) with a stir bar. Afterwards, HCl (1M, 4M or both) was added to the water glass, drop by drop while the solution was stirring, until a pH of 8 to 11 was achieved. At the pH of 8 to 11, gelation

of the water glass would start to occur. Immediately thereafter, before the gelation of the water glass proceeded within the container (310), the water glass (308) was transferred into the mould (306) that contained the moist rolled-up glass fibre (304) (Fig. 3C), at a volume ratio of water glass:glass fibre of 3:2. Once the water glass (308) had been poured into the PTFE mould (306) and infused with the glass fibre (304), the top of the PTFE mould was tightly covered with a lid (312) (Fig. 3D) and the product was left to undergo gelation and aging for 2 to 3 hours before the steps of solvent exchange and surface modification.

After the gelation and aging, the sample was placed in water for salt removal. The aged sample was soaked in water for a total of about 12 hours, with the water changed every few hours. This removed trapped salts in the pores of the gel network. If the trapped salts are not removed, it may cause a decrease in the optical transmission properties as well as an increase in the density of the silica aerogel.

Solvent exchange was continued by soaking the sample in ethanol (C_2H_5OH) or isopropyl alcohol (IPA, C_3H_8O) at 50°C. Organic solvents have a low surface tension which reduces capillary pressure and helps prevent gel shrinkage and collapse of the pore walls. Exchange of water with organic solvent also helps the silylating surface reagent, trimethylsilyl chloride (TMCS, $[(CH_3)_3SiCl]$), to reach the pore surfaces during the surface modification step.

After solvent exchange with an organic solvent, the sample was soaked in n-hexane (C_6H_{14}) for 24 hours at 50°C in an oven (Binder (Tuttlingen, Germany)). n-hexane has been proven effective in improving the physical properties of the silica aerogel, such as surface area, pore diameter and hydrophobicity.

The sample was then soaked in a mixture of TMCS, ethanol and n-hexane for 24 hours at 50°C for surface modification. The molar ratio of TMCS/SiO_2 and TMCS/ethanol was 2:1 and 1:1 respectively, and the ratio of the hexane/TMCS by volume was 25:2. This was to obtain a hydrophobic silica aerogel, and to avoid shrinkage during the subsequent drying process at ambient pressure. Lastly, the sample was soaked in n-hexane for 24 hours at 50°C to remove unreacted TMCS.

It should be noted that the larger the sample, the longer it takes to complete the solvent exchange and surface modification processes. The sample was dried in an oven at 50°C for 1 hour, followed by 200°C for another 1 hour to ensure the complete removal of hexane, resulting in the completion of the fabrication of the silica aerogel blanket. This whole process is indicated in Fig. 2.

Example 3: Modified Fabrication Process

Fig. 4 shows a modified innovative process of fabricating aerogel blanket from the IBA. The modified process includes, in step 1M, treatment of IBA and extraction of silica through microwave reaction, and in step 2M, immersion of glass fibre after surface modification process.

Step 1M

Instead of treating the IBA by stirring it with a HCl leaching solution for 24 hours, microwave reaction was adopted, whereby the IBA was treated with the HCl leaching solution at 180˚C for 1 hour.

Further, instead of extraction of silica by refluxing the IBA in a NaOH solution at 160˚C for 24 hours, microwave extraction was adopted using a microwave digestion system Multiwave 5000 (Anton Paar (Graz, Austria)), whereby the chemically treated IBA was reacted under microwave at 180˚C for 1 hour. The Multiwave 5000 comprises 16 vessels, hence 10 g of incineration bottom ash in 50 mL of 2 M HCl solution was used in each vessel.

The yield of the water glass solution improved by 12% higher than the non-modified process, as shown in Table 1 below.

Table 1 Comparison of the water glass solution fabricated with different methods

	Fabrication process	Modified fabrication process
Method of Si Extraction	Conventional (stirring hotplate)	Microwave
Yield of water glass solution	100%	112%

Step 2M

After the dilution of the water glass in deionised water with a ratio of 2:3 by volume, the mixture was pumped through a column filled with strongly acidic ion exchange resin (Amberlite, IR-120H). The column comprised a coarse fritted disc having an internal diameter of 44 mm and a length: 600 mm. The eluent used was the mixture itself.

The silicic acid was then collected. Afterwards, NH_3 solution (0.5M) was added into the silicic acid solution, drop by drop, while the solution was stirring, until a pH of 4 to 5 was achieved. At the pH of 4 to 5, gelation of the water glass would start to occur.

By exposing the sample to radiation, i.e. UV, IR, etc., the aging process can be accelerated from 3 hours to 1 hour.

After aging, the sol-gels was crushed into powder form before being transferred into a mixed solvent solution which contains isopropyl alcohol (IPA, C_3H_8O), n-Hexane (C_6H_{14}) and trimethylsilyl chloride (TMCS, $[(CH_3)_3SiCl]$) for surface modification. The ratio of n-hexane, IPA and sample was 10:6:3 by volume and the molar ratio of silica: TMCS was 1:4. The samples were immersed in the mixed solvents with low speed stirring (<300rpm) and heating (40˚C) for 3 hours before the aerogel was separated in the upper layer. The hydrophobic silica gels were collected after removing the water at the bottom layer.

It should be noted that in the modified process, the solvent exchange process was omitted and the surface modification process was shortened due to the smaller size of the gel particle following the crushing step.

The collected hydrophobic silica gel was then dispersed in IPA or n-hexane. The glass fibre that has been heated at 450°C for 1 hour, washed in deionized water, wrung tightly and tightly rolled into a cylindrical shape was then immersed into the hydrophobic silica gel suspension for complete coating of the fibre with the silica gel. Alternatively, chopped glass fibre strands having a diameter of 3-5 µm and a length of a 5 mm to 50 mm may also be mixed with the hydrophobic silica gel suspension then moulded into a blanket. This mixing process took about 2 hours. Lastly, the samples were dried in an oven at 50˚C for 2 hours followed by a further 1 hour at 200˚C.

Table 2 below illustrates the required duration of time for each step of the aerogel blanket fabrication processes before and after modification.

Table 2. Comparison of the time duration in the aerogel blanket fabrication processes using different methods.

	Duration for fabrication process [hr]	Duration for modified fabrication process [hr]
Treatment of Glass Fibre	1	1
Physical Treatment of IBA	0.5	0.5
Chemical Treatment of IBA	24	1
Separation of Leaching Solution & IBA	0.3	0.3
Si Extraction from IBA	24	1
Separation of Water Glass solution & residue	0.3	0.3
Immersing of glass fibre into dispersed hydrophobic silica gel suspension (1)	0.2	0
Sol-gel process	0.2	0.2
Gelation & Aging	3	1
Solvent Exchange	72	0
Surface modification	24	3
Immersing of glass fibre into dispersed hydrophobic silica gel suspension (2)	0	2
Drying	3	3
Total Time Duration	152.5	13.3

Industrial Applicability

The method for fabricating a silica aerogel blanket as disclosed herein may be used to fabricate silica aerogel blankets. The silica aerogel blankets may be used in a multitude of industries and businesses. Possible applications for the silica aerogel blankets may include the oil and gas processing industry (liquefied natural gas (LNG), liquefied petroleum gas (LPG), and ethylene), offshore oil, refineries, petrochemical and gas processing, building and construction, urban heating systems, industrial furnaces, solar energy storage tanks and other energy storage, urban rail transit, electric vehicles and batteries, ultra-low temperature pipelines, marine engines, appliances, outdoor gear and apparel, transportation, military, and aerospace. Other applications include insulation of cryogenic pipes and for insulating difficult areas such as between flanged sections of vacuum-jacketed piping. In the building and construction industry, silica aerogel blankets may have a similar use to that of traditional thermal insulators.

It will be apparent that various other modifications and adaptations of the invention will be apparent to the person skilled in the art after reading the foregoing disclosure without departing from the spirit and scope of the invention and it is intended that all such modifications and adaptations come within the scope of the appended claims.

Claims

1. A method of fabricating a silica aerogel blanket, comprising the steps of:

   i) providing incineration bottom ash;

   ii) extracting silica from the incineration bottom ash in the presence of a base to form a water glass;

   iii) forming a silica gel from the water glass; and

   iv) contacting the silica gel with a fibre to form a silica aerogel blanket.

2. The method according to claim 1, further comprising the step of drying the incineration bottom ash at a temperature in the range of about 50 °C to about 100 °C before step ii) and/or the step of grinding the incineration bottom ash to a particle size of less than 200 µm before step ii).

3. The method according to claim 1 or 2, further comprising the step of pre-treating the incineration bottom ash with a pre-treatment acid before the step of extracting silica from the incineration bottom ash, wherein the pre-treatment step comprises:

   the step of stirring the incineration bottom ash with the acid at room temperature at a stirring speed in the range of about 200 rpm to about 400 rpm for a duration in the range of about 18 hours to about 36 hours; or

   the step of irradiating the incineration bottom ash in the presence of the acid with microwaves at a temperature in the range of about 160 °C to about 200 °C for a duration in the range of about 30 minutes to about 2 hours.

4. The method according to any one of claims 1 to 3, wherein the base is contacted with Y g of incineration bottom ash having X percent by weight of silicon, wherein Y is the mass of the incineration bottom ash in grams,

   wherein the mass of the base (m_{base}) to be used in the extraction step is described by the following equation (Ia):

   $$m_{base} = \frac{Y \times X}{30 \times a} \times MW_{base} \qquad \text{(Ia)}$$

   wherein MW_{base} is the molecular weight of the base; and

   the volume of the base dissolved in deionised water to form a solution (V_{base}), to be used in the extraction step is described by the following equation (Ib):

   $$V_{base} = \frac{Y \times X}{30 \times a \times M_{base}} \qquad \text{(Ib)},$$

wherein M_{base} is the molar concentration of the base.

5. The method according to claim 4, wherein the extraction step further comprises:

the step of refluxing the incineration bottom ash with the base at a temperature in the range of about 140 °C to about 180 °C for a duration in the range of about 18 hours to about 36 hours; or

the step of irradiating the incineration bottom ash in the presence of the base with microwaves at a temperature in the range of about 160 °C to about 220 °C for a duration in the range of about 30 minutes to about 2 hours; or

the step of reacting the incineration bottom ash in the presence of the base under pressure, at a temperature in the range about 160 °C to about 220 °C, a pressure in the range of about 0.5 MPa to about 2 MPa, and for a duration in the range of about 30 minutes to about 4 hours.

6. The method according to any one of the preceding claims, further comprising the step of diluting the water glass in deionized water at a ratio in the range of about 1:2 to about 3:4 by volume, before contacting with the fibre.

7. The method according to claim 6, further comprising the step of adding a pH-adjusting acid to the diluted water glass until the pH is in the range of about 8 to about 11, to form the silica gel before contacting with the fibre.

8. The method according to claim 6, further comprising the step of purifying the water glass on a strongly acidic ion exchange resin to collect silicic acid.

9. The method according to claim 8, further comprising the step of adding a pH-adjusting base to the silicic acid until the pH is in the range of about 3 to about 6 to form the silica gel.

10. The method according to any one of claims 7 to 9, further comprising the step of aging the silica gel before or after contacting with the fibre.

11. The method according to claim 10, wherein:

the aging step is performed after contacting the silica gel with the fibre, further comprising the step of removing a salt or the step of exchanging a solvent after aging; or

the aging step is performed before contacting the silica gel with the fibre, further comprising the step of crushing the aged silica gel into a powder before contacting with the fibre.

12. The method according to any one of the preceding claims, further comprising the step of surface modifying the aged silica gel before or after contacting with the fibre.

13. The method according to claim 12, wherein the step of surface modification is performed by reacting the aged silica gel with an organosilicon compound.

14. The method according to claim 12 or 13, wherein:

the step of surface modification is performed after contacting the silica gel with the fibre, and the silica aerogel blanket is immersed in a mixture of organosilicon compound, an alcohol and an alkyl having 6 or more carbon atoms, wherein:

- the molar ratio of organosilicon compound:silica is in the range of about 3:1 to about 1:1;

- the molar ratio of organosilicon compound:alcohol is in the range of about 2:1 to about 1:2; and

- the ratio of alkyl having 6 or more carbon atoms: organosilicon compound by volume is in the range of about 10:1 to about 15:1; or

the step of surface modification is performed before contacting the silica gel with the fibre, and the aged silica gel is immersed in a mixture of organosilicon compound in a surface modification solvent, wherein:

- the ratio of the surface modification solvent:aged silica gel is about 1:1 to about 6:1 by volume; and

- the molar ratio of silica:organosilicon compound is in the range of about 1:10 to about 1:2.

15. The method according to any one of claims 12 to 14, wherein the step of surface modification is performed at a temperature in the range of about 30 ˚C to about 50 ˚C for a duration in the range of about 2 hours to about 36 hours with or without stirring.

16. The method according to claim 14 or 15, wherein the surface modification is performed before contacting the silica gel with the fibre, and the surface modified silica gel is separated from the surface modification solvent and dispersed in an alcohol or an alkyl having 6 or more carbon atoms before being contacted with the fibre.

17. The method according to any one of the preceding claims, wherein the fibre is selected from the group consisting of glass microfibres, mineral microfibres, ceramic micro and nanofibres, nanotubes, polypropylene microfibres, aramid microfibres, carbon micro and nanofibres, carbon nanotubes and cotton nanofibres.

18. The method according to any one of the preceding claims, wherein the fibre is in the form of separate strands or is woven into a sheet.

19. The method according to any one of the preceding claims, further comprising the step of heating the fibre at a temperature in the range of about 350 °C to about 500 °C before step iv).

20. The method according to claim 19, further comprising the step of washing and wringing the fibre after the heating step.

21. The method according to claim 20, further comprising the step of placing the fibre into a mould before contacting with the silica gel.

22. A silica aerogel blanket obtained by the method according to any one of the preceding claims.

23. Use of incineration bottom ash in the fabrication of a silica aerogel blanket.