TMCS with carbonate or bicarbonate ions in the silica wet-gel to generate CO_2 gas within the pores of the wet-gel from the inside. This allows for more rapid drying of the wet-gel, with ambient pressure drying. However, improvements are still needed, in terms of economy, efficiency and the form of the aerogels produced.

It is an aim of the invention to obviate or mitigate one or more of the disadvantages associated with the prior art. A scalable method for the preparation of silica aerogels would be beneficial. A less energy intensive method would be useful, as would be a method which allows for a reduction in solvent use. A method for the preparation of silica aerogels with reduced processing/drying time would be particularly advantageous, as would a cost-effective method for producing same. A method which could be used to form controlled-shaped aerogels would be particularly beneficial.

Summary

The present disclosure relates to methods of preparing aerogels, such as silica aerogels, in which chemically driven self-pressurisation reactions occurring both inside and outside the wet-gel during production facilitates rapid drying under ambient pressure conditions. In examples, the aerogels can be prepared with fibres to allow for the preparation of controlled shaped products. Advantageously, the aerogels can be rapidly prepared under ambient pressure conditions, leading to a reduction in time costs and energy consumption, thereby facilitating scale-up of the process.

Accordingly, in a first aspect of the present disclosure there is provided a method of preparing a silica aerogel, the method comprising providing a precursor solution comprising a silicate and optionally a carbonate solution; and reacting the precursor solution with a bicarbonate and with a silylating agent, wherein the bicarbonate is in the form of a solid.

In this process, initial gelation is achieved by the reaction of the silicate with the bicarbonate, yielding a silica wet-gel shell, carbonate and water, while the silylating agent also reacts with the silicate, causing further gelation and modifying the wet-gel shell surface, while yielding HCl.

The silylating agent also reacts with the HCl produced and the carbonate, either formed as a by-product or optionally present in the precursor solution, to form CO_2. In this manner, external pressurisation occurs, i.e. at the external surface of the wet-gel shell, which facilitates rapid drying.

In addition, unreacted silylating agent and generated HCl within the pores of the wet-gel, diffuse into the wet-gel shell core, reacting with the bicarbonate to produce CO_2, thereby pressurising it from the inside and further facilitating the drying step.

The steps of reacting the precursor solution with a bicarbonate and a silylating agent may be carried out sequentially, i.e. in which the precursor solution is reacted first with a bicarbonate, and subsequently with a silylating agent; or the steps may be carried out simultaneously.

An example of the disclosure therefore relates to a method of preparing a silica aerogel, the method comprising preparing a wet-gel by providing a precursor solution comprising a silicate and optionally a carbonate solution, and reacting the precursor solution with a bicarbonate and with a silylating agent, wherein the bicarbonate is in the form of a solid; and drying the wet-gel to form a silica aerogel.

In an example of the disclosure, the step of reacting the precursor solution with the bicarbonate is carried out in the presence of fibres. The fibres can be added to the precursor solution, or to the bicarbonate powder, before the reaction takes place. This can be useful when it is required that the aerogels have a controlled shape. However, in examples, the fibres are not present, and the aerogel is produced as an uncontrolled solid. This can be subsequently pulverized or ground to form a powder or granules depending on the intended use.

In an example, the fibres are ceramic fibres, organic fibres or carbon fibres.

In an example, the fibres are ceramic fibres. Triton™ ceramic short fibres are an illustrative example of fibres which can be used, although one skilled in the art would appreciate that alternative fibres can be used.

Advantageously, when fibres are used, the method allows for the rapid production of controlled shaped reinforced aerogel composites.

The term composite is used to describe an aerogel with one or more additional components, such as the fibres which can be introduced into the aerogel structure.

In an example, the fibres surround the bicarbonate powder. The fibres can be formed into a shape around the bicarbonate powder. For example, the fibres can be shaped into a sphere or ball surrounding the bicarbonate powder. In this example, the sphere can be soaked in the precursor solution. The fibres can impart structural integrity to the resultant aerogel, allowing for the preparation of controllable hollow aerogel composites; in this example a fibre-reinforced hollow aerogel composite (FRHAC).

Additionally, in this example, the CO_2 generated at the external surface of the wet-gel shell is retained within the shaped structure until it is released via the pores of the silica wet-gel. As the gas can displace the liquid component of the gel, drying can be achieved at low temperature and pressure, making the process energy efficient.

Accordingly, the disclosure relates to a method of the production of a silica aerogel, the method comprising providing a precursor solution comprising a silicate and optionally a carbonate solution; and reacting the precursor solution with a bicarbonate and a silylating agent, wherein the bicarbonate is in the form of a solid, and wherein reacting the precursor solution with the bicarbonate is carried out in the presence of fibres.

In an example, the disclosure relates to a method of the production of a silica aerogel, the method comprising providing a precursor solution comprising a silicate and optionally a carbonate solution;

providing fibres surrounding a bicarbonate core to form a shaped structure;

reacting the precursor solution with the bicarbonate and a silylating agent, wherein the bicarbonate is in the form of a solid.

In an example, in the step of reacting the precursor solution with the bicarbonate, the shaped structure is immersed in the precursor solution. In this way, the precursor solution can soak through the fibres and react with the bicarbonate powder core.

While the fibres advantageously create a shaped structure in which the CO_2 produced is retained until it diffuses through the pores of the wet-gel, a similar effect can be achieved without the fibres, by preparing the wet-gel on a substrate. When the precursor reacts with

the bicarbonate powder, a wet-gel is formed as a layer. In this example, the CO_2 is retained between the substrate and the external surface of the wet-gel layer, until it is released via the pores of the wet-gel. Suitable substrates would be known to a person skilled in the art, and include, for example, glass, ceramic and polymer substrates.

In an example, the silicate is selected from sodium silicate, potassium silicate, lithium silicate or calcium silicate.

In an example, the silicate is sodium silicate.

Sodium silicate, also known as "waterglass" is particularly suitable for use in the present disclosure.

In an example, the carbonate is selected from sodium carbonate, potassium carbonate, calcium carbonate, magnesium carbonate, iron carbonate and ammonium carbonate.

In an example, the carbonate is sodium carbonate.

In an example, the bicarbonate is selected from sodium bicarbonate, potassium bicarbonate, calcium bicarbonate, magnesium bicarbonate, iron bicarbonate and ammonium bicarbonate.

In an example, the bicarbonate is sodium bicarbonate.

In an example, the silylating agent has the general formula R_3SiX, in which R is a C_1-C_4 alkyl or halide, and X is a halide, sulfate or sulfonate group.

The halide can be chloride, bromide or iodide.

The sulfonate can be methyl sulfonate, trifluoromethylsulfonate, benzylsulfonate or tolouenesulfonate.

The sulfate may be $-O-S(O)_2-OSiR_3$.

In an example, the silylating agent is trimethylchlorosilane (TMCS), dimethyldichlorosilane, methyltrichlorosilane and bis(trimethylsilyl)sulfate.

In an example, the silylating agent is trimethylchlorosilane (TMCS).

The method of the disclosure produces a partially dried aerogel, which could be the end product. However, in examples, one or more washing and/or drying steps may be carried out to obtain the final aerogel product.

In an example, the method comprises one or more washing steps.

In an example, the method comprises one or more drying steps. The drying step(s) can be performed by conventional means, such as in an oven or on a heat plate. Suitable heating methods would be apparent to one skilled in the art.

The drying step comprises heating under ambient pressure.

The heating may be carried out at a temperature of from 60°C to 500 °C. In an example, the heating is carried out at from 60°C to 150°C.

Temperatures around 100°C (e.g. from 80°C to 120°C) may be preferred, since the liquid phase has a boiling point no higher than 100°C.

The drying step may be performed for from 15 minutes to 24 hours. As would be understood by one skilled in the art, the length of the drying step will depend on the heating temperature, with lower temperatures requiring longer drying times. In an example, the drying step is performed for from 15 minutes to 12 hours, or from 15 minutes to 6 hours. In an example, the drying step is carried out for from 20 minutes to 3 hours, or from 30 minutes to 2 hours. When the drying temperature ranges from 60°C to 150°C, the drying time may be from 30 minutes to 12 hours, from 30 minutes to 8 hours, from 30 minutes to 6 hours or from 30 minutes to 2 hours.

Examples:

The invention will now be described by way of example only with reference to the accompanying figures, in which:

Figure 1 illustrates a schematic of a method according to an embodiment of the invention;

Figure 2 shows X-ray tomographic images of a FRHAC prepared according to an embodiment of the invention;