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Silica Aerogels: Synthesis and Experiments of a Fascinating Material for Upper Secondary School Level

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Abstract

During the last years, material scientists have developed plenty of new materials with fascinating properties. Although a lot of these innovations do not find their way into everyday life, they hold an enormous didactic potential for chemistry education by, for example, allowing a clear insight into current research in material sciences. Examples for these materials are silica-based aerogels. Although this material is not that new - the first successful synthesis was in 1931 –it is rapidly gaining in importance, especially in technical applications.[1] Reasons for this are its fascinating, exceptional physical properties such as its large surface area (500 – 1500 m^2/g), high porosity (80 - 99 %), low bulk density (0.03 – 0,3 g/cm^3) and extremely low thermal conductivity (0.005 W/mk).[2] Due to these properties, an aerogel has been recognized by Guiness World Records as the world's lightest solid with the lowest density. In addition, it was part of NASA's Stardust spacecraft acting as a particle-collecting substance.[3] Other applications are, for example, the use of aerogels as isolators or filters with new uses following. The objective of this article is to show how silica-based aerogels can be synthesized in a school setting. Furthermore, experiments are presented which illustrate the special properties of aerogels such as their low density and their unique structure caused by the small size of nanoparticles. These experiments can also be used to learn and discuss important contents from the curricula such as material characteristics (e.g. density and surface activity) or reaction mechanisms such as hydrolysis and condensation.

1. Introduction

Nowadays, the number of new materials that come on the market each month is rising continuously. But most of them play neither an important role in everyday life nor in schools, even if they have fascinating properties. So to minimize the gap between new fields of research and schools, research and development in chemistry education should facilitate insights into current research topics especially for pupils in secondary schools. From our point of view, one interesting topic to achieve such a goal is the production and the analysis of silica aerogels and their properties. These materials consist of a three-dimensional network of spherical silicon dioxide (SiO_2) nanoparticles. The resulting structure is responsible for the special characteristics of the aerogel, *e.g.* its high porosity. The materials consists of mostly air, from which the name aerogel (Greek: *aero* – air) is derived. Figure 1 shows an aerogel (left) and the structure of a silica aerogel (right).

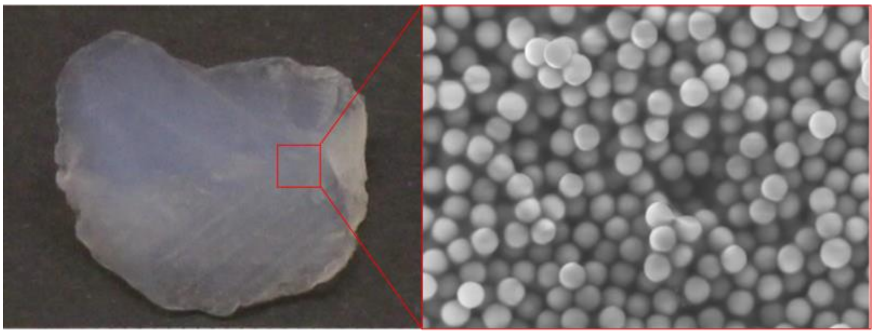

Figure 1: *Photograph (left) and a representative SEM picture of an aerogel (right).*

In recent literature, various ways of synthesising aerogel materials can be found. Our aim within this contribution is to present an aerogel synthesis which can be conducted in schools using chemicals that are (readily) available in school settings as well as cheap and harmless. Furthermore, experiments illustrating three highly interesting properties of aerogels will be highlighted.

2. Experimental Section

2.1 Production of an Aerogel

Aerogels are typically synthesized by a sol-gel-process. The precursor (alkoxysilanes) hydrolyses and condenses in the presence of a catalyst in the liquid phase, forming spherical $(SiO_2)_n$-particles. The obtained gel consists of three-dimensional connections between the particles. In order to remove the solvent in the pores of the aerogel to obtain a porous network structure, the gel is dried. Nowadays, the most effective way of drying is with supercritical carbon dioxide. Figure 2 shows the five steps and the five substances (alkoxide, alcohol, water and two catalysts) which are needed for the synthesis.

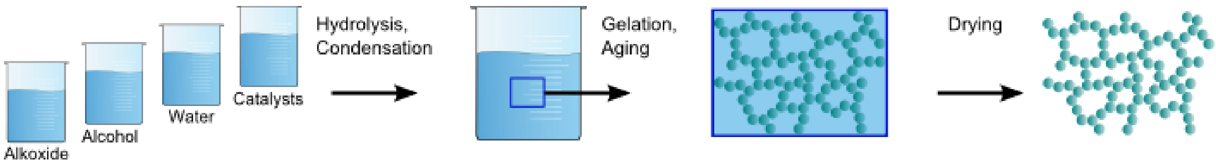

Figure 2*: Diagram of the synthesis of an aerogel (sol-gel-process) after a model by Claas.[4]*

Here, an adapted synthesis for school chemistry based on Li et al. is presented.[5]
Equipment and chemicals: beakers, Erlenmeyer flaks with fitting rubber plugs, magnetic stirrer, vessel, concentrated methyltrimethoxysilane (MTMS), concentrated tetraethoxyorthosilane (TEOS), ethanol, 0,01 M acetic acid, 0,3 M ammonium hydroxide, n-heptane.
Experimental: All the following steps have to take place at a temperature of 45°C. For the synthesis, 7,1 mL of MTMS, 1,1 mL of TEOS, 12,8 mL of ethanol and 4 mL of acetic acid are mixed and the solution is stirred for 60 minutes in an Erlenmeyer flask. The molecules hydrolyse while the flask is closed with a rubber plug and the mixture has to rest for 24 hours. For condensation, 4,5 mL of ammonium hydroxide are mixed with the solution, filled in a vessel and hermetically sealed. During the next 24 hours, the product is left to gel and age. Once the gel has fully formed, the solvent has to be removed and exchanged for another solvent (n-heptane). In the course of the following days, the solvent should be exchanged a couple of times till the ethanol has been completely removed. After another few days, the solvent needs to be evaporated after which the gel is dried.
Alternatively, in order to dry the substance, schools could also cooperate with a university or an institute able to dry the gel using supercritical carbon dioxide. This method cannot be used in schools.

2.2 Experiments on Aerogel Properties

Aerogels are made up of nano-sized particles and they possess a low density as well as an extremely low thermal conductivity. In the following section these aforementioned properties are investigated and demonstrated with the help of three different experiments.

2.2.1 Determination of the Density

There are different ways to calculate the density of a material or in this case of an aerogel. If the structure of an aerogel corresponded to a geometric figure, the density could easily be measured using its volume and mass. However, the volume of an aerogel as shown in Figure 3 (left) cannot be calculated by measuring its size.

Equipment and chemicals: cylinder, sand, aerogel, scale.

Experimental: Another possibility to obtain the volume is measuring the displaced volume of sand in a cylinder. Here, a defined amount of sand is filled into a cylinder and its volume is written down. Then, the aerogel is added to the cylinder and deeply immersed and covered with sand. The resulting volume as well as its difference to the original volume can be directly read off the cylinder (see Fig. 3 (right)).

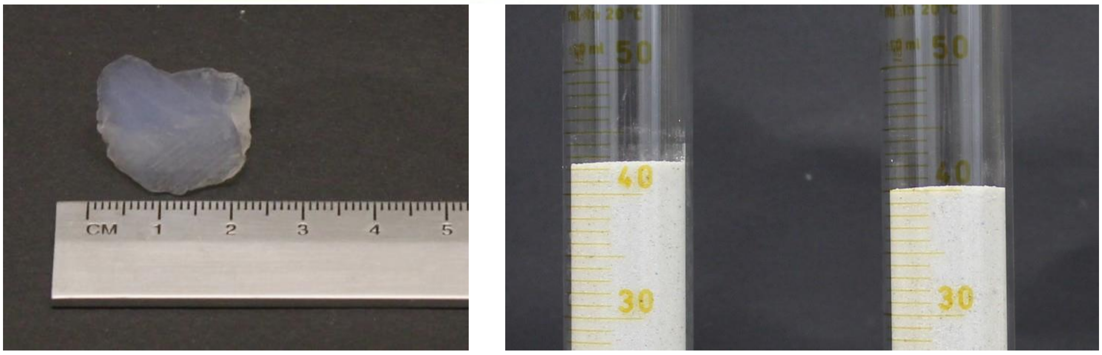

Figure 3: *The aerogel with unknown volume and density (left). In the right picture, the cylinder with the sand and the aerogel (left) and the cylinder without the aerogel (right).*

In a sample measurement as seen in Figure 3, the difference in volume is around 0,0025 L. After measuring the mass of the aerogel, 0,6723 g, the density can be calculated at approximately 0.27 g/cm^3.

2.2.3 Tyndall Effect – Estimation of Particle Size

Aerogels consist of a network of nano-sized particles. To demonstrate the dimensional size of these particles, the Tyndall effect can be used (Figure 4).

Equipment and chemicals: aerogels, laser pointer.

Figure 4: *Visualization of a laser beam inside an aerogel.*

Experimental: When laser beams are pointed at aerogels, the beam inside the aerogel can be seen. According to John Tyndall, this can be explained by the size of the particles. Colloidal particles inside of solutions or transparent solids with the same size range as the wavelength of light scatter photons creating beams of light visible to the eye.[6] This happens with aerogels since the particles in aerogels have a size in the range of some hundred nanometers.

2.2.4 Thermal Conductivity of an Aerogel

Due to their unique structure, aerogels have a low thermal conductivity. To show the difference between a thermal conductor (*eg.* a piece of metal) and an insulator (an aerogel) a simple experiment can be conducted.

Equipment and chemicals: tripod with a wire mesh, gas burner, thin sheet of copper, metal pencil sharpener, aerogel, two matches.

Experimental: The copper sheet is placed on the wire mesh. The aerogel and the metal pencil sharpener are put above and one match is placed on top of each object (see Figure 5 (left) for the experimental design). When heating the copper sheet with the gas burner, the heat is transmitted to the metal pencil sharpener, to the aerogel and therefore also to the matches. As soon as the kindling temperature of the match is reached, it catches fire. The match on top of the pencil sharpener starts burning after three minutes while the match on the aerogel stays untouched even after further heating (13 min). This demonstrates the low thermal conductivity of aerogels.

Figure 5*: Demonstration of the thermal conductivity of aerogels and metals; matches after one minute (left), after three minutes (center) and after thirteen minutes (right).*

In this case, we chose a metal pencil sharpener in order to utilize an everyday life object which is familiar to the students; it is also possible to experiment with a metal coin or other metal objects.

3. Summary and Outlook

The production and study of silica aerogels offer an enormous didactic potential. For this reason, we developed a synthesis as well as experiments for working with aerogels in a school setting. The next step would be to find other more effective ways of drying the aerogels without the use of supercritical carbon dioxide. Currently, different options are being tested.
Alternatively, an aerogel could be purchased by a school allowing students to investigate its properties without having to synthesize it first. Furthermore, a teaching unit suitable for this context could be developed in which the focus would be familiarizing students with current research. Additionally, concepts such as condensation and hydrolysis can be introduced or reinforced using the synthesis reaction of aerogels.

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