

Review Article

Sol-Gel Methods for the Assembly of Metal and Semiconductor Nanoparticles

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Abstract

The ability to assemble nanoparticles into functional structures is an important challenge that needs to be addressed for the generation of nanoparticle-based devices. Sol-gel method represents a facile yet powerful strategy for the self-assembly of metal oxides, chalcogenides, and metal-semiconductor hybrid nanoparticle systems into three-dimensionally connected porous nanostructures. In contrast to traditional oxides, where gels are formed by hydrolysis and condensation of molecular precursors, gelation in non-oxidic systems (with the exception of carbon), is achieved by condensation of the pre-formed nanoparticles. In this highlight, the application of later strategy for the assembly of chalcogenide semiconductor and noble metal nanoparticles and their intriguing physical properties is reviewed in the context of future application in catalysis, sensing, and separation technologies.

INTRODUCTION

The intriguing physical characteristics observed in nanoscale metals and semiconductors are related to the changes in electronic structure as well as the large surface-to-volume ratios, which make them vastly different from corresponding bulk solids. These unique properties have led to the development of synthetic methods that permit exquisite control over materials size, shape, and dispersity in the nanometer length scale [1,2]. However, most of the applications envisioned, and the devices that are likely to be constructed, will not be solution based or single particle based but will be formed from nanoparticle assemblies in the solid state. Hence, developing methods for the assembly of nanoparticles into solid state structures, while retaining their characteristic physical properties, is an important challenge. One classic method that has proved useful for production of nanostructures is the *sol-gel* technique, in which nanoparticles are formed and aggregated through a series of hydrolysis and condensation reactions to form a wet-gel structure [3-7]. When the wet-gel is supercritically dried, the polymeric structure can be retained, producing a material known as an *aerogel*.

Aerogels are a unique class of porous inorganic polymers that exhibit high surface areas, solid contents ranging from 1-15% by volume, and densities as little as three times that of air [5]. As such, they are ideal substrates for catalyst and sensors, and are excellent thermal insulating materials [3-7]. A schematic description of the morphology of a base-catalyzed silica aerogel is shown in (Figure 1), depicting the three-dimensionally (3-D) connected network of nanoscale building blocks. One of

the unique features of the aerogel that makes it useful for a variety of chemical applications is the continuous micro-(<2 nm) to meso-(2-50 nm) pore structure which provides a facile conduit for molecules to reach the nanostructure surface [8-10]. Nevertheless, the potential uses of aerogels have been limited to insulating or wide band gap oxidic materials (e.g. SiO_2 , Al_2O_3 , TiO_2) for many years because of restrictions in the chemical systems that can be employed for the gel formation. Under sol-gel conditions, a polymeric gel structure will form in preference to a precipitate only when hydrolysis and condensation kinetics are optimized and the metal is of moderate electronegativity [5]. Silica, alumina, and titania display ideal sol-gel characteristics, hence they have been extensively studied, while other systems, specifically conductive oxides [11-13] and non-oxidic [6] materials have been significantly less explored.

Over the last two decades, scientists have begun to extend sol-gel chemistry to non-oxidic systems. This has led to the development of carbon aerogels, the first class of non-oxide

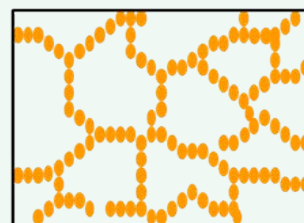


Figure 1 Pearl necklace architecture of the traditional base catalyzed silica aerogel.

aerogels prepared from pyrolysis of resorcinol-formaldehyde gel frameworks [14,15]. Later in 1997, Boilot et al. reported the synthesis of CdS (cadmium sulfide) wet-gels from thiolate coated CdS (cadmium sulfide) nanoparticles by oxidative removal of the surface ligands, *i.e. the condensation of pre-formed NP colloids* (Figure 2) [16-18]. Several research groups have successfully exploited this strategy for the construction of chalcogenide semiconductor and metal aerogels by employing thiolate or citrate stabilized pre-formed nanoparticles [19-40]. Interestingly, the resultant nanostructures display characteristic pearl necklace morphology of the nanosized constituents without the presence of intervening ligands but are low-dimensional materials with band energies (for semiconductors) that can be tuned by adjusting the density or the porosity of the gel framework [20,28]. The focus of this mini-review is to highlight the recent progress of the use of sol-gel chemistry for the assembly of metal and semiconductor nanoparticles, its advantages in comparison to other well-known nanoparticle assembling methods, and the unique physical characteristics of the sol-gel derived nanoparticle superstructures.

Semiconductor aerogels by nanoparticle condensation

Chalcogenide semiconductors have gained considerable interest from the scientific community due to their strong size, shape, and composition dependent photophysical properties [41,42]. Significant efforts have already been devoted to the assembly of such particles utilizing organic ligands, polymers, biomolecules, and templating strategies [43-46]. Despite the successes in producing ordered or non-ordered superstructures by these methods, the use of intervening ligands have been shown to moderate inter-particle interactions hindering charge transport and limiting the thermal stability of the resultant nanostructures. In contrast, a strategy involving direct cross-linking of nanoscale metals, such that intrinsic to a nanoparticle based aerogel, would result in highly conducting superstructures with tunable surface area and pore characteristics. Accordingly, the synthesis of CdS (cadmium sulfide) wet-gels by a two-step nanoparticle formation and condensation route was originally reported by Gacoin et al. (Figure 2) [16-18,47,48]. In this

approach, stable CdS nanoparticle sols were prepared by dispersing pre-formed thiolate capped particles in acetone. In a second step, partial removal of the surface ligands using a chemical oxidant (H_2O_2) resulted in transparent or opaque wet gel structures. It was also revealed that the gelation phenomena in metal chalcogenide nanoparticles rely on the kinetics of the surface thiolate oxidation, in which active sites for the nanoparticle condensation become available. If excess of oxidant is present in the sol, too many active sites are generated on the nanoparticle surface, leading to precipitation whereas, if too little oxidant is present in the sol, nanoparticles are completely passivated by the thiolates leading to a stable sol. Gacoin *et al.* suggests that at a minimum concentration of oxidant/thiolate (X_{min}) no gel is formed whereas above X_{min} , gelation occurs [16]. With increasing X_{min} , rate of the gelation/condensation increases ultimately leading to precipitation at $\sim 5X_{min}$ [16]. Hence, the density, surface area and porosity of the resulting gel networks were fine-tuned by varying the amount of oxidant (X_{min} - $5X_{min}$), type of the oxidant, and the gel aging time [23].

The generality of this route to prepare a number of other chalcogenide and pnictide gel structures based entirely on pre-formed nanoparticles was described by Brock et al. [19,21,22,33,49,50]. The gelation can be induced by a variety of oxidants such as H_2O_2 , tetranitromethane, and photo oxidation and the nanoparticles prepared by different synthetic methods have been successfully transformed into wet-gel and aerogel structures [21]. As-prepared semiconductor aerogels are monolithic, consist of a three-dimensional assembly of nanoparticles and are morphologically similar to silica aerogels prepared by base catalysis (Figure 3A). Chalcogenide aerogels produced by these route exhibit bulk densities as low as 0.07-0.35 g/cm³ and are meso-to-macroporous with BET surface areas up to ~ 250 m²/g (600 m²/g silica equivalence). Even though the nanoparticles are directly hard-wired into random three-dimensional architectures, the size dependent energetic features are mostly retained as a result of low dimensionality (high porosity) of the gel structure (Figure 3B and Table 1). For instance, the nanocrystals of CdSe (cadmium selenide) when interconnected in a colloidal wet-

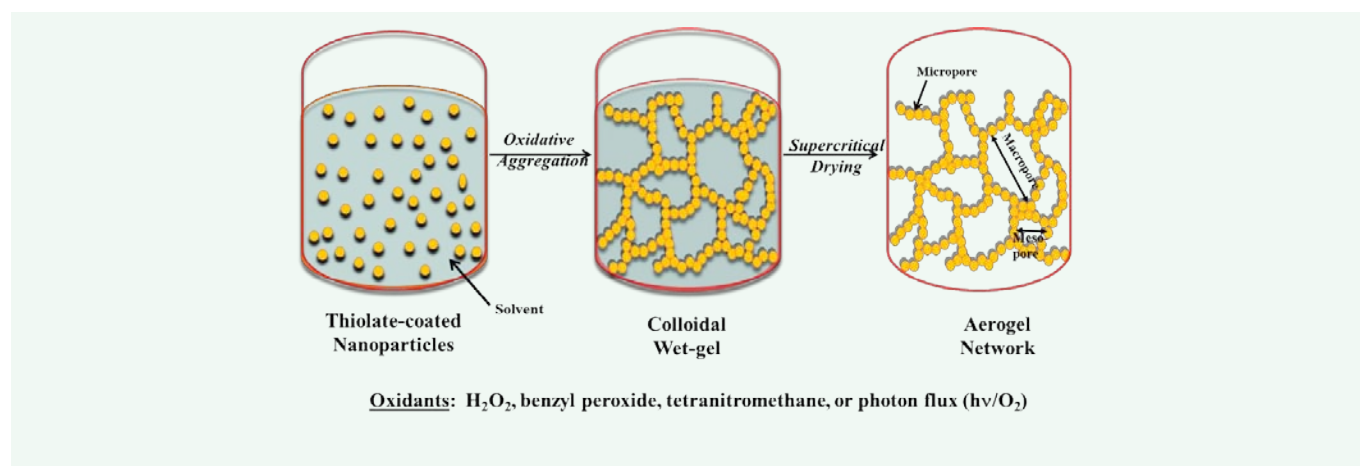


Figure 2 Schematic description of the direct self-supported assembly of metal and semiconductor nanoparticles into aerogel frameworks via nanoparticle condensation strategy. Concentrated colloidal sols are prepared in polar solvents and the gel formation is induced by oxidative removal of the surface ligands over 1-21 days. The resulting wet-gel structures are dried under supercritical conditions to form highly porous superstructures (aerogels) composed entirely of nanoscale building blocks.

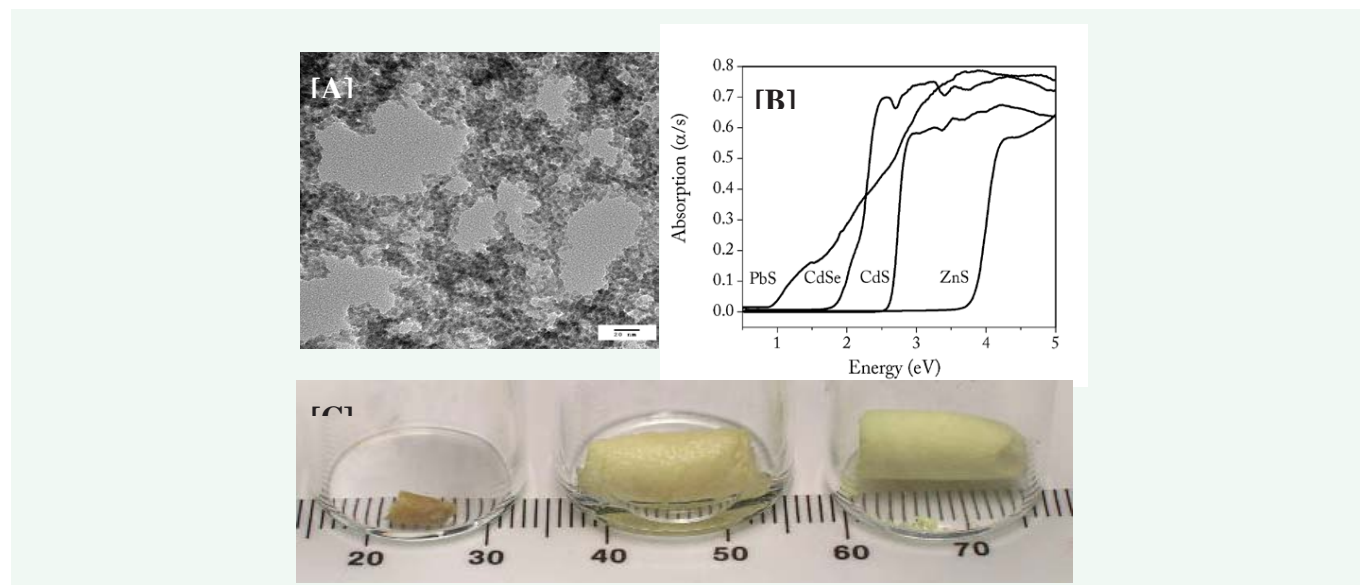


Figure 3 [A] Transmission electron micrograph of a CdSe aerogel prepared by nanoparticle condensation route showing the meso to macro porous interconnected network of colloidal nanoparticles. [B] Optical absorption spectra of chalcogenide gels prepared by nanoparticles condensation route. [C] A photograph of CdS wet-gel (center) and a xerogel (left) prepared by bench top drying and an aerogel (right) dried under supercritical conditions. Part A is reproduced with permission from 2006 American Chemical Society and part B and C are reproduced with permission from 2005 American Association for the Advancement of Science.

Table 1: Comparison of the optical bandgaps of chalcogenide semiconductor aerogels and corresponding bulk materials along with the BET surface areas, BJH average pore diameters, and cumulative pore volumes of metal and semiconductor aerogels reported to date.

Aerogel	Bandgap onset (eV)	Bulk Bandgap (eV)	BET Surface Area (m ² /g)	BJH Average Pore diameters (nm)	BJH adsorption cumulative pore volume (cm ³ /g)
PbS [19]	0.80(1)	0.37	119-141	21-45	0.79-0.94
CdSe [19]	2.19(5)	1.74	128-161	16-29	0.53-0.98
CdS [19]	2.71(2)	2.42	239-250	29-30	1.90-2.02
CdTe [34]	1.92	1.50	120-140	12.7-13.8	0.55-0.83
ZnS [19]	3.80(4)	3.54	182-202	15-30	0.40-0.86
PbTe [35]	N/A	N/A	74	19	0.34
InP [40]	2.1	1.35	195-212	7.7-8.7	0.41-0.46
Bi ₂ Te ₃ [38]	N/A	N/A	45	9	0.10
Bi _{2-x} Sb _x Te ₃ [38]	N/A	N/A	36	9	0.10
Ag/CdS [51]	N/A	N/A	73-165	3.2-3.4	0.29-0.61
Au/CdS [52]	N/A	N/A	50-81	3.14-3.62	0.20-0.21
Au/Ag [53]	N/A	N/A	48	N/A	N/A
Pt/Ag [53]	N/A	N/A	46	N/A	N/A
Pd [54]	N/A	N/A	92	N/A	N/A

gel network retain their optical band gap [21]. However, with increasing dimensionality (an increase in density or a decrease in porosity) from wet gels to aerogels to xerogels, the band gap of the inter-connected particles shows a red shift, but compared to bulk CdSe the band gap values remain significantly blue shifted (Figure 4) [20,28]. Hence, the quantum confined optical features in these gel structures has been successfully tuned as a function of the density or the porosity of nanostructures. Likewise, the band gap of the inter-connected nanoparticles can be further tuned by heating, resulting in a systematic red shift in the absorption onset with increasing annealing temperature (100-

300 °C) [19,33]. This red shift is accompanied with a crystallite size growth and a phase change from cubic to hexagonal for CdS aerogels. CdSe aerogels prepared by this approach show weak band-edge emission of their linked nanoparticles, which is attributed to the residual thiolate ligands acting as traps for the electron-hole recombination [21]. However, the thiolate ligands exchange with pyridine in the wet-gel stage results in a considerable enhancement of band edge emission. This novel approach has been successfully extended for the assembly of a variety of metal chalcogenide and pnictide based nanoparticles including but not limited to CdS [33], CdSe [19-21,28-30], CdTe

(cadmium telluride) [31,32,34], ZnS (zinc sulfide) [19,20,37], PbS (lead sulfide) [19,20], PbTe (lead telluride), [35] InP (indium phosphide) [40], Bi₂Te₃ (bismuth telluride) and Bi_{2-x}Sb_xTe₃ (bismuth antimony telluride) nanoparticles [38]. Moreover, the synthesis of Ag/CdS (silver/cadmium sulfide) [51], and Au/CdS (gold/cadmium sulfide) [52] metal-semiconductor hybrid gel structures by employing a similar strategy has also been reported. The optical bandgaps, Brunauer-Emmett-Teller (BET) surface areas, Barrett-Joyner-Halenda (BJH) average and cumulative pore volumes of the aerogels prepared from nanoparticle condensation route are listed in Table 1.

Metal aerogels by nanoparticle condensation

Intriguing physical properties observed in metal-chalcogenide and pnictide based aerogels have sparked research forefronts to investigate the potential of sol-gel chemistry for the assembly of metal nanoparticles. Accordingly, Eychmuller *et al.* has successfully utilized the nanoparticle condensation strategy for the creation of non-ordered superstructures of metal nanoparticles (Ag, Au, Pt, and Pd) [53,54]. In this approach, monometallic and heterogeneous bimetallic nanoparticles coated with citrate ligands were slowly oxidized to form monolithic gel structures composed entirely of metal nanoparticles (Figure 5). The resultant aerogels are reported to be extremely light with average densities that are two orders of magnitude lower than reported metal forms [55] and three orders of magnitude lower as compared to bulk metals. These metal aerogels are morphologically distant from chalcogenide semiconductor aerogels and appeared to be consisting of ultra-thin wire-like nanostructures with typical thickness in the same scale as precursor nanoparticles (Figure 5) [53]. Resultant bimetallic aerogels (Au/Ag and Pt/Ag) are reported to exhibit BET surface areas in the range of 46-48 m²/g. The formation of such noble metal nanoparticle based mesoporous

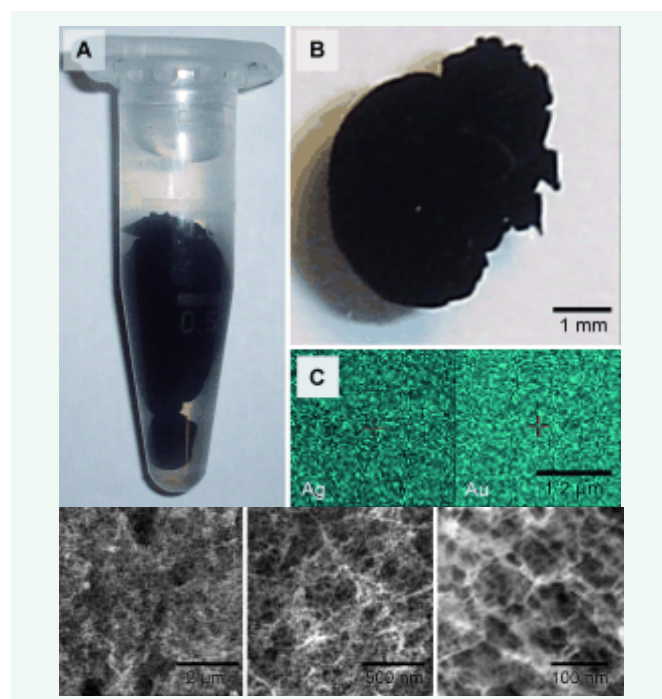


Figure 5 (a) Photograph of a heterogeneous Au/Ag hydrogel, and (b) of a piece of the corresponding aerogel produced by oxidative removal of citrate ligands from corresponding nanoparticles followed by supercritical drying. (c) Energy dispersive spectroscopy mapping of the aerogel, showing equal distributions of gold and silver. (d) SEM images of a bimetallic gold/silver aerogel at different magnifications. Reproduced with permission from 2009 *Angewandte Chemie International Edition*, Wiley Online publishers.

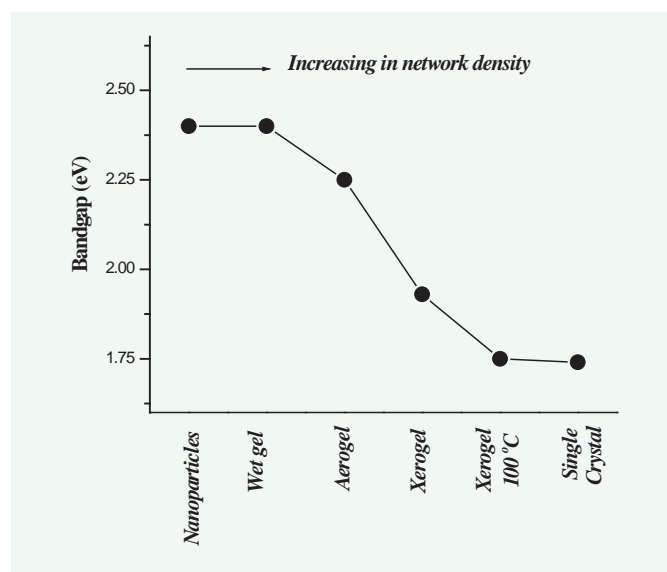


Figure 4 An illustration of the effect of network density on the optical bandgaps of CdSe gel structures. Bandgap values can be tuned in between the individual nanoparticle and bulk values by engineering the density or the porosity of the nanostructure. Reproduced with permission from 2006 *Comments on Inorganic Chemistry*, Taylor and Francis Publishers.

superstructures is an important step towards self-supported monoliths with enormously high catalytically active surfaces. When one considers that metal nanoparticles possess unique optical properties due to their pronounced surface plasmon resonance, aerogels derived from metal nanoparticles may also find future applications in nanophotonics. Very recently, the same group has reported the synthesis of Pd aerogels cross-linked with cyclodextrin, which exhibits a relatively higher surface area of 92 m²/g as well as superior catalytic activity in ethanol oxidation in fuel cell applications [54]. As a result of the synthesis of metal and semiconductor aerogels through a general synthetic approach (nanoparticle condensation), synthetic routes to metal-semiconductor hybrid gel structures have also been established (e.g. Au/CdTe (gold/cadmium telluride)) [56,57]. The control of synthetic parameters, gel drying and processing conditions has allowed the researchers to create monolithic gel structures on a time scale of minutes to hours suggesting the potential for up-scaling the synthesis for future technological applications.

To date, the understanding of the fundamental phenomena relating to: the mechanism of nanoparticle-based gel formation, the nature of the interactions between nanosized constituents, and the effect of low-dimensional connectivity on optical, electrical, and thermal transport properties of the aerogels is still in its infancy. In a previous report on gelation mechanics of metal chalcogenide aerogels (e.g. CdSe), Brock *et al.* has shown that the oxidative removal of the surface thiolates triggers the oxidation of surface selenides (Se²⁻) into diselenide bridges

(Se-Se) supporting the network formation [24]. Similarly, in a recent report from the same group suggests the gelation in pnictide-based nanoparticles (e.g. InP) is due to condensation of phosphorus oxo-anionic moieties generated at the interfaces of the particles [40]. However, such mechanistic studies on recently developed metal aerogels and metal/semiconductor hybrid gel structures have not been reported.

CONCLUSION AND PROSPECTUS

Chalcogenide, pnictide, and metallic gel structures formed via nanoparticle condensation is an exciting and emerging field of non-oxidic sol-gel chemistry. This route ensures the production of particulate gel structures by separating the nanoparticle formation from the condensation event. The ability to create such self-supported metal and semiconductor aerogels as thin films or monoliths will generate a host of new device possibilities in catalytic, sensing, and separation technologies, while their density, morphology, conductivity, surface area and porosity can be tuned by engineering the gelation kinetics as well as the physical properties of the precursor nanoparticle colloids. The direct hardwiring of the pre-formed nanoparticles into highly conducting gel frameworks with tunable surface area and porosities is a unique and creative component to this strategy that has already demonstrated superior performance in electrocatalysis [54]. These nanostructures are proven to exhibit nanoparticle connectivity, control over surface area and porosity, as well as superior charge transport properties. As such, they would be uniquely poised to tackle a number of technological issues that have not been successfully addressed by bulk materials, discrete nanoparticles, and ligand-stabilized nanoparticle superstructures. Finally, the ability to create aerogels using chalcogenide/pnictide semiconductors and noble metals provides the means to carry out the nanoparticle condensation in a larger number of other nanoparticle systems including but not limited to other catalytic metals, elemental and compound semiconductors, as well as their hybrid materials, while retaining the characteristic physical properties of individual particles. However, an area of concern for sol-gel derived nanoparticle superstructures is the poor mechanical stability of the resulting aerogels. Presently gels that are formed in solution and dried are often fragile hence; the free standing superstructures should be carefully handled during subsequent characterization and applications. To address this issue, it should also be possible to produce composite materials based on these aerogels, e.g. by polymer infiltration, to combine their physical properties with greater mechanical stability. Specific studies to test this premise are currently underway.

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