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Monolithic Nickel (II) Oxide Aerogels Using an Organic Epoxide: The Importance of the Counter Ion

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Monolithic Nickel (II) Oxide Aerogels Using an Organic Epoxide: The Importance of the Counter Ion

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Abstract

The synthesis and characterization of nickel (II) oxide aerogel materials prepared using the epoxide addition method is described. The addition of the organic epoxide propylene oxide to an ethanolic solution of $NiCl_2$ $6H_2O$ resulted in the formation of an opaque light green monolithic gel and subsequent drying with supercritical CO_2 gave a monolithic aerogel material of the same color. This material has been characterized using powder X-ray diffraction, electron microscopy, elemental analysis, and nitrogen adsorption/desorption analysis. The results indicate that the nickel (II) oxide aerogel has very low bulk density (98 kg/m^3 (~98 %porous)), high surface area (413 m^2/g), and has a particulate-type aerogel microstructure made up of very fine spherical particles with an open porous network. By comparison, a precipitate of $Ni_3(NO_3)_2(OH)_4$ is obtained when the same preparation is attempted with the common $Ni(NO_3)_2$ $6H_2O$ salt as the precursor. The implications of the difference of reactivity of the two different precursors are discussed in the context of the mechanism of gel formation via the epoxide addition method. The synthesis of nickel (II) oxide aerogel, using the epoxide addition method, is especially unique in our experience. It is our first example of the successful preparation of a metal oxide aerogel using a metal divalent metal ion and may have implications

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for the application of this method to the preparation of aerogels or nanoparticles of other divalent metal oxides. To our knowledge this is the first report of a monolithic pure nickel (II) oxide aerogel materials.

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1. Introduction

The use of different synthetic routes to nickel (II) oxide materials has been significant in the preparation of materials with unusual and useful properties. Different methods of syntheses can markedly alter important parameters such as particle size, particle morphology, defect concentration, crystallinity, and surface area of NiO.[1-3] Alteration of these characteristics can have dramatic effects on the behavior of the material. For example, the ac conductivity of NiO nanoparticles is six to eight orders of magnitude higher than that in a single crystal.[4]

Nickel oxide is of considerable technological interest. It is a monoxide of the $3d$ metals, which are well known to have mixed oxidation states and defects that lead to non-stoichiometry. Depending on their preparation method, they can be made with varied stoichiometries, which can affect their chemical properties significantly.[2]   Nickel (II) oxide is a semiconductor and it is also used in supported catalysts for several chemical transformations including the reformation of methane with CO_2 [5], and the oxidation of H_2S to sulfur [6]

Sol-gel methodology is a reliable and popular approach to preparing materials, especially metal oxides, with uniform and small particle sizes and varied morphologies.[7] Surprisingly, there is not a large body of work using sol-gel methods to prepare nickel (II) oxide-based materials. Much of that work involves to binary or ternary mixtures of NiO with other oxides such as silica [8] and alumina.[9-11] The primary interest in sol-gel derived material with this

structure and composition is catalytic transformations of organic molecules.[12] This type of material exhibits good activity towards oxidations of organic compounds while resisting the formation of coke on the catalyst under certain conditions.[5] There have been very limited reports on the preparation of pure nickel oxide or nickel hydroxide gels and or colloids.[13] In most instances materials of this nature were prepared using dialysis or peptization and processing to final dry aerogel material was not attempted.[14]

Aerogels have several distinct and uncommon properties that make them attractive for technical applications in many areas.[15-18] They possess high porosity (90-99%), high surface area, low density, some degree of optical transparency, and low thermal conductivity. Their unique properties are a result of their unusual microstructure, which consists of a continuous network of colloidal particles, or polymeric chains with characteristic diameters of ~10 nm, open porosity, and typical pore diameters less than 100 nm.   Aerogels have been studied for application as thermal insulators, catalysts, dielectrics, optical coatings, laser targets, waste remediation materials, sensors, pesticides, energetic materials, and as collectors for high-energy particles.[17]

Although they are technologically fascinating aerogels have limitations. In addition to the cost of supercritical processing, one must take into account synthetic routes and the final form of the material and how it functions in each application. With this in mind, aerogels are fragile materials that are easily damaged upon handling. Many aerogels are supercritically dried to aerogel powders or highly cracked and fragile monoliths that cannot be handled without severe restrictions.[16] This limits their utility in areas where processing or application dictates some structural form with moderate strength (e.g., laser target components).[18]   An exception here are SiO_2 aerogels, which can be dried to monoliths without cracking and can be handled

reasonably without damage. This property has helped to facilitate the application of silica and doped silica aerogels in many of the above-mentioned areas. The synthesis of monolithic, stable, non-silica aerogels has been found to be especially difficult. Therefore, the synthesis of robust aerogels with greater chemical range and variety would be of interest to future aerogel application studies.

We have reported on the use of 1,2-epoxides as gelation agents for the synthesis of robust monolithic iron (III) oxide, and chromium (III) oxide aerogels and xerogels from their inorganic salt precursors.[19-21]   The 1,2-epoxides have at least one three-member cyclic ether ring in their structure (see Scheme 1).   Molecules of this type have considerable ring strain and undergo irreversible ring opening reactions.[22] In addition, they are especially good at scavenging protons.
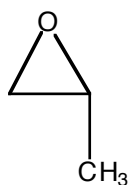
Scheme 1. Structure of the 1,2-epoxide, propylene oxide.

Attempts to apply this synthetic method to the sol-gel preparation of metal oxides other than Fe_2O_3 and Cr_2O_3 has been very successful. Aerogels of several different main group, transition metal, and lanthanide metal oxides has been achieved via this route. [20]

Typically aerogels of metal oxides are prepared using metal alkoxide precursors and most commonly are limited to SiO_2, Al_2O_3, ZrO_2, and TiO_2.[15,16] This synthetic route has proven to be an efficient, easy, and successful approach to the production of aerogels of those

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compositions. Much less sol-gel work has been published on other transition and main group metal oxides. This is largely because many of their metal alkoxides are expensive and still others are sensitive to moisture, heat, and light making their use and long-term storage difficult. In addition, some metal alkoxides are not commercially available or are difficult to obtain, thus precluding detailed studies on the preparation, characterization, and potential applications of their resulting porous metal oxides.

As we have reported, we believe this to be an important general sol-gel route to aerogels. This method is applicable in a variety of solvents with several different epoxides and makes use of stable metal ion salts, instead of alkoxides, which allow the process to be inexpensive and more facile than some previous methods to produce metal oxide aerogels. In a larger sense, we recognize that this straightforward synthetic method can be easily extended to the preparation of many other main-group and transition metal oxide porous solids from simple metal ion salts instead of metal alkoxide precursors. Although the emphasis of this paper is aerogel synthesis and characterization this method has been extended to prepare crystalline nanoparticles with unique properties. For example, Woo *et al*. have reported the synthesis of crystalline nanorods of both α-Fe_2O_3 and γ-Fe_2O_3 using the epoxide addition sol-gel reaction in combination with reverse micellular synthetic techniques.[23]

Using this approach, a variety of metal oxide aerogels are possible, however not all of our attempts have been successful. In particular, initially we were unable to achieve monolithic gel formation in any of the attempted syntheses with M^{2+} ions of the late $3d$ transition metals (M = Ni^{2+} , Co^{2+}, Zn^{2+}, and Cu^{2+}).[20] In all of those cases hydrated metal nitrate salts were used as starting materials and the results of all the syntheses were precipitates. However, we have discovered that in the case of nickel (II) the use of an alternative precursor salt ($NiCl_2$ $6H_2O$)

results in the successful synthesis of opaque light green monolithic nickel (II) oxide aerogel. This result is significant as it represents the first example of the application of this method to metal ions in the 2+ oxidation state. In this report we discuss the synthesis and characterization of nickel (II) oxide aerogel materials as well as the influence of the counter ion in the mechanism of gel formation.

2. Experimental

Nickel (II) chloride hexahydrate, $NiCl_2 \cdot 6H_2O$ (Mallinckrodt), and propylene oxide (99%; Aldrich) and nickel (II) nitrate hexahydrate, $Ni(NO_3)_2 \cdot 6H_2O$ (Aldrich), were used as received. All syntheses were performed under room conditions. In a typical experiment, 0.37 g of $NiCl_2 \cdot 6H_2O$ (1.56 mmol) was dissolved in 2.5 ml of 200 proof ethanol to give a clear light green solution. When a 1.0 g portion of propylene oxide (17 mmol) was added to the solution a rigid light green opaque gel, which spanned the entire reaction vessel, formed within 30 minutes. Alternatively, if a 0.47 g (1.6 mmol) of $Ni(NO_3)_2$ $6H_2O$ was used in this preparation the result was always a green precipitate that formed within 60 minutes after the addition of the propylene oxide.

2.1 Gel processing conditions

The wet gels were covered and allowed to age for at least 24 hours under ambient conditions. After that they were immersed in a bath of 200 proof ethanol where they were washed for ~1 week. During that time the ethanol bath solution was changed at least four times. Aerogel samples were dried in CO_2 in a Polaron™ supercritical point drier. The solvent liquid in the wet gel pores was exchanged for CO_2 (*l*) for 2-3 days, after which the temperature of the

vessel was ramped up to ~45 C, while maintaining a pressure of ~100 bars. The vessel was then depressurized at a rate of ~7 bars per hour.

2.2 Physical characterization

Surface area and pore volume and size analyses were performed by BET (Brunauer-Emmett-Teller) and BJH (Barrett-Joyner-Halenda) methods using an ASAP 2000 Surface area Analyzer (Micromeritics Instrument Corporation). Samples of approximately 0.1 – 0.2g were heated to 200 C under vacuum (10^{-5} Torr) for at least 24 hours to remove all adsorbed species. Nitrogen adsorption data was taken at five relative pressures from 0.05 to 0.20 at 77K, to calculate the surface area by BET theory.[24] All nitrogen adsorption/desorption measurements were required to equilibrate for a minimum of forty seconds before being recorded.[25] Typical nitrogen adsorption/desorption experiments took 20-50 hours to complete. For the BJH analyses average pore size and pore volume were calculated using the data points from the desorption branch of the isotherm.

The transmission electron microscopy (TEM) was performed on a Philips CM300FEG operating at 300Kev using zero loss energy filtering with a Gatan energy Imaging Filter (GIF) to remove inelastic scattering. The images where taken under BF (bright field) conditions and slightly defocused to increase contrast. The images were also recorded on a 2K x 2K CCD camera attached to the GIF. Powder X-ray diffraction (PXRD) data was collected on aerogel samples using an APD3720 PEI automatic powder diffractometer with an analyzing crystal and Cu K_α radiation was used. Samples were mounted on an aluminum plate. Elemental analyses of aerogel samples were performed by Galbraith Laboratories, Inc. of Knoxville TN.

2.3 Study of pH of aqueous Ni(II) solution during gelation.

A 0.57 M solution of Ni (II) was made by dissolving 1.8 g of $NiCl_2 \cdot 6H_2O$ (7.5 mmol) in 13 mL of 200 proof ethanol. The solution was stirred with a magnetic stir bar on a stirring plate. A glass pH electrode interfaced with a Hanna Instruments model 9020 pH meter was immersed into the solution and the resulting pH measured. Then a 4.5 g (77 mmol) aliquot of propylene oxide was added to the stirring solution and the pH was recorded at regular intervals for the next 60 minutes. An identical experiment was performed using 2.24 g of $Ni(NO_3)_2 \cdot 6H_2O$ (7.5 mmol).

3. Results

The choice of Ni (II) precursor has a dramatic effect on the synthesis of Ni (II) oxide aerogels. Using $Ni(NO_3)_2$ $6H_2O$ as the precursor no monolithic gels were obtained. Under the synthesis conditions employed here, the only product was a green precipitate. If instead, $NiCl_2$ $6H_2O$ was the precursor used the product obtained was a robust light green opaque wet gel that was subsequently dried with supercritical CO_2. Figure 1 is a photo of a one such NiO aerogel monolith.

Powder X-ray diffraction (PXRD) was used to characterize the products obtained from the synthesis of Ni (II) oxide using both the nitrate and chloride salt precursors. Figure 2 contains the diffraction patterns of the products from the different precursors. The pattern for the aerogel material made with the chloride salt has broad diffuse peaks, most likely a result of its amorphous character and the high porosity of the material. The PXRD data for the green precipitate made with the nickel (II) nitrate salt indicates that it is a crystalline material. The set of peaks form this material was successfully matched to a known compound in the JCPDS

database, $Ni_3(NO_3)_2(OH)_4$ (JCPDS #: 22-0752). This compound is a known thermal decomposition product of $Ni(NO_3)_2 \cdot 6H_2O$.[26,27]

Surface area, pore size, and pore volume are important characteristics for sol-gel derived materials. We have measured these properties for the two Ni (II) based materials described in this report. The nitrogen adsorption/desorption results for the nickel (II) oxide aerogel and the sol-gel derived $Ni_3(NO_3)_2(OH)_4$ are presented in Table I. Both samples have notable surface areas, however that of the nickel (II) oxide aerogel is much higher than that of $Ni_3(NO_3)_2(OH)_4$ (413 m^2/g to 120 m^2/g). The surface area of the aerogel material is nearly three times higher than that previously reported for NiO nanoparticles.[2] Both samples display Type II isotherms with H3 hysteresis loops. The H3 hysteresis is often seen in solids that consist of aggregates of plate-shaped particles or in adsorbents with slit-shaped pores.[24] It is interesting to note that the hysteresis in the $Ni_3(NO_3)_2(OH)_4$ material is much more pronounced than in the nickel (II) oxide aerogel.

Elemental analysis was performed on the nickel (II) oxide aerogel. The results of one of those analyses are shown in Table II. The results indicate that there is a significant amount of residual organic impurities (5.7 wt% C and 3.6 wt % H) and chloride (7.1 wt %) present in the material. The organic contaminants are most likely the result of residual epoxide, or products of the ring opening of the epoxide, or the solvent. The chloride level is higher than anticipated for a Ni (II)-oxide based material and at this time we can only speculate as to its origin.[28]

Figure 3 contains two transmission electron micrographs of nickel (II) oxide aerogel. These images provide a fine representation of the microstructure of the aerogel. The material is composed of interconnected and partially coalesced roughly spherical particles. The morphology

can be categorized as a particulate gel, which is very typical of many transition metal oxide aerogel materials.[7]

In attempt to more completely understand that processes that affect gel formation in this system we performed experiments where the pH of the precursor solution was monitored after addition of the epoxide gelation agent. The results of one such experiment are shown in the plot in Figure 4. In this experiment the pH of an ethanolic solution of $NiCl_2$ $6H_2O$ was monitored after addition of propylene oxide. Initially the pH rises in small increments, until about 3 minutes after addition. Then the pH begins to rise more rapidly; during this time a pale green colloid begins to form in the sample and near the end (~20 minutes after epoxide addition) of this portion of the plot the solution becomes slurry-like. In the final portion (> 20 minutes) of this plot the pH rise begins to flatten out a bit and at pH ~5 the sample has transformed to an opaque light green monolithic wet gel.
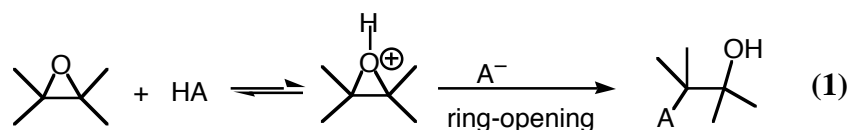
When the same experiment is performed using the $Ni(NO_3)_2$ $6H_2O$ a gradual pH rise is observed in the early portions of the experiment however, it is not as rapid as in the previous case. This may be a result of the poor nucleophilic nature of nitrate ion compared chloride, which results in less favorable kinetics for the ring opening of the epoxide. Instead of forming a monolithic gel, over the course of several hours, a compact green precipitate is formed at the bottom of the reaction vessel.

4. Discussion

In regards to the terminology nickel (II) oxide aerogel, which is utilized throughout this report, we will clarify. Since XRD analysis show no evidence of a crystalline phase and the stoichiometry determined from elemental analyses does not exactly match that of NiO we

thought identifying the aerogel as NiO could be misleading. The material is amorphous and may contain appreciable amounts of hydroxyl groups and or water so as not to overstate our knowledge of the identity of the material, at this time, we refer to it as nickel (II) oxide.

We have reported on the use of propylene oxide as a gelation agent for the synthesis of iron (III) as well as chromium (III) oxide aerogels and xerogels from their respective inorganic salt in a variety of different solvents (e.g., water, ethanol, methanol).[19-21] Mechanistically, it has been our contention that the dissolved aquocation acts as an acid (HA in (1)) and protonates the added epoxide thus, activating it towards ring-opening by a nucleophile.

$$\text{epoxide} + \text{HA} \rightleftharpoons \text{protonated epoxide} \xrightarrow[\text{ring-opening}]{A^-} \text{ring-opened product} \quad (1)$$

For the Fe (III) system a mechanistic study, using pH measurements and NMR, revealed that the added epoxide acts as an irreversible proton scavenger that induces the Fe (III) species to undergo hydrolysis and slow and uniform condensation to form an Fe (III)-oxide sol and forming substituted alcohol species as the side product of ring-opening. These processes result in uniform pH gradients in solution; hence there are little variations in the hydrolyzed species that form, and leads to aerogels with reproducible characteristics. This approach circumvents many of the problems associated with common synthetic approaches to metal oxides (e.g., direct addition of strong base).[29]

Since the protonation and ring-opening reactions are associated with the hydrolysis and condensation of metal aquo or aquo/hydroxy monomers in the sol-gel polymerization, the identity of the aquocation, and its respective acidity, can dictate the kinetics of sol particle growth and aggregation. Fine control of these parameters would allow the more control of the

microstructure and potentially, the properties of the resulting gel. In addition, variation of the epoxide can accomplish this. Although not detailed here, we have shown that the identity of the epoxide has a great deal of influence on the compositional and microstructural properties of Fe (III) oxide aerogels made via epoxide addition.[21]

Aquo cations especially those of 4+, 3+, and small 2+ ions behave as acids in solution. At its most basic level this effect is ascribed to the influence of the positive charge on the coordinated water molecules to loose a proton. The acidic properties of a given aquocation

$$[M(OH_2)_6]^{n+} + H_2O \underset{\longleftarrow}{\overset{K}{\longrightarrow}} [M(OH)(OH_2)_5]^{(n-1)+} + H_3O^+ \quad (2)$$

in solution can be measured quantitatively and expressed in an equilibrium constant K (see (2)). Precise values of acidity for aquo cation complexes are difficult to obtain and according to the literature can vary by orders of magnitude. Therefore, little reliance can be placed on the exact numbers, however qualitative comparisons of these values allow some general trends to be outlined. There is a dependence on charge, with 4+ cations typically more acidic than 3+ cations and 2+ cations being weakly acidic.[30]

In summary, the epoxide acts as simple base by undergoing the reaction shown in (1). The proton used in (1) comes from a reaction like that shown in (2). The acidic species (HA) reacting with the epoxide is likely a metal aquocation or aquo/hydroxy species and the anion (A⁻) is the precursor salt counterion. The combination of these reactions results in uniform pH increase throughout the solution and subsequent sol formation in the sample. We believe a similar process is occurring in the Ni (II) case, as the pH experiments appear to bear out (see Figure 4).

Due to its smaller formal charge the $Ni(H_2O)_6^{2+}$ species is known to be a weaker acid than its Fe(III) analog. The $Ni(H_2O)_6^{2+}$ species has been reported to have pK_a values ranging from 6.5 to 10.2, whereas $Fe(H_2O)_6^{3+}$ has values ranging from 0.8 to 2.0.[30] According to the pH data presented in Figure 4 the $Ni(H_2O)_6^{2+}$ species appears to be a strong enough acid to allow reaction (1) to proceed to the extent necessary to form a nickel (II) oxide gel. However, there is another factor to consider in the synthesis of nickel (II) oxide aerogel materials using the epoxide addition method, the counterion.

Successful preparation of opaque light green monoliths depended upon the type of precursor salt used. Monolithic gels were only achieved when $NiCl_2$ $6H_2O$ was utilized. If instead, $Ni(NO_3)_2$ $6H_2O$ was used as the starting material only green precipitates were realized. It appears that the precipitate is favored when a solution containing Ni (II) ions and nitrate ions has its pH slowly and uniformly raised.

Our previous work, involving the use of epoxides in aerogel formation has shown a dependence of gel formation on the identity of the counterion.[19] In that case, iron (III) oxide gels could not be formed in water if the nitrate salt starting material was used. A careful study suggested that in that case the nitrate was not as strong a nucleophile as the solvent water. This led to a reaction of propylene oxide with water to form propylene diol, which is catalytic with respect to proton consumption. Therefore, the pH of the solution did not increase appreciably and thus olation and oxolation of Fe (III) species to form iron (III) oxide gels did not occur. However this does not appear to be the situation with Ni (II) oxide gel formation as our pH experiments indicate that the pH of the gel synthesis solution increased, albeit more slowly than that utilizing $NiCl_2$ $6H_2O$ as the precursor salt, and a precipitate is formed within an hour.

The PXRD analysis of the green precipitate formed when the nitrate salt is used indicates that the material is the basic nickel hydroxide nitrate compound, $Ni_3(NO_3)_2(OH)_4$. This compound is a known product of the thermal decomposition of $Ni(NO_3)_2$ $6H_2O$.[26,27] Interestingly enough, it undergoes well-characterized single step decomposition to NiO and has been touted as an alternative synthetic route to NiO nanoparticles with reproducible characteristics.

Previously we had not observed gel formation for any of the late $3d$ transition metal divalent ions (Co^{2+}, Ni^{2+}, Cu^{2+}, and Zn^{2+}).[20] It was thought that the aquocations of those ions were not sufficiently strong enough acids to induce reactions **1** and **2** to occur to the extent necessary for formation of stable a gel. The results of the experiments reported here appear to contradict that. As the pH experiments indicate, the both the syntheses solutions (Ni (II) chloride and nitrate) experience a steady pH increase after epoxide addition. With the chloride salt this continues to sol and then gel formation. As the pH rises in the nitrate case it reaches a point where, because of the identity of the counterion, the formation of the basic $Ni_3(NO_3)_2(OH)_4$ species is favored and precipitation occurs.

The TEM (Figure 3) results indicate that the nickel (II) oxide aerogels are best described as particulate gels made up of nominally spherical particles of ~5-20 nm in diameter. The particles are relatively uniform in size and exhibit amorphous characteristics as they do not diffract electrons. Previous synthetic approaches to nickel (II) oxide gels have involved the precipitation of $Ni(OH)_2$ followed by dissolution in a suitable solvent and prolonged dialysis.[13,14] Alternatively, this approach to nickel (II) oxide aerogel synthesis is a straightforward, one step method that can be done at room temperature with a common inorganic Ni (II) salt.

5. Conclusions

Here we have described the successful synthesis of a pure nickel (II) based aerogel material. To our knowledge, this is the first report of this type of material. The nickel (II) oxide aerogel material displays all of the characteristics of an aerogel: 1) high surface area (413 m^2/g), 2) high porosity (~98%porous (density = 98 kg/m^3)), 3) amorphous to X-rays, and 4) extremely fine particulate microstructure and an open porous structure. The synthesis of nickel (II) oxide aerogel, using the epoxide addition method, is especially unique in our experience. It is our first example of the successful preparation of a metal oxide aerogel using a metal divalent metal ion. All previous attempts at this were unsuccessful. Precipitates were formed in all cases, no rigid gel. Our observations here, and previously, may lend some insight into the reasons for the failures. It appears that the role of the metal salt counter ion is very important. We have yet to determine the correct combination of precursor salt, solvent, and epoxide to induce gelation of additional metal oxide aerogels (e.g., CoO, CuO, or ZnO) with divalent oxidation states. Nonetheless it remains an active area of investigation in our laboratory. This method is viable for the synthesis of nickel (II) oxide aerogels and should be for the preparation of nanoparticles of NiO, with suitable post synthesis processing of the sol-gel product.

6. Acknowledgements

7. References

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Figure and Table Captions

**Figure 1.** Photograph of nickel (II) oxide aerogel made by the addition of propylene oxide to an ethanolic solution of $NiCl_2 \cdot 6H_2O$.

Figure 2. Powder X-ray diffraction data for a) nickel (II) oxide aerogel material and for b) $Ni_3(NO_3)_2(OH)_4$ prepared via the epoxide addition method. The latter compound is the product when prepared using $Ni(NO_3)_2 \cdot 6H_2O$ precursor salt dissolved in ethanol and adding propylene oxide.

Figure 3. Two transmission electron micrographs a) and b) of a nickel (II) oxide aerogel made using the $NiCl_2 \cdot 6H_2O$ precursor salt. The thick tubular structure in the lower portion of a) is a portion of the carbon used to coat the Cu TEM grid and is an artifact.

Figure 4. Plot of measured pH vs. time since addition of propylene oxide for the synthesis of a nickel (II) oxide aerogel material using the $NiCl_2 \cdot 6H_2O$ precursor salt in ethanol.

Table I. Summary of N_2 adsorption/desorption data for nickel (II) oxide aerogel and $Ni_3(NO_3)_2(OH)_4$ made via the epoxide addition method.

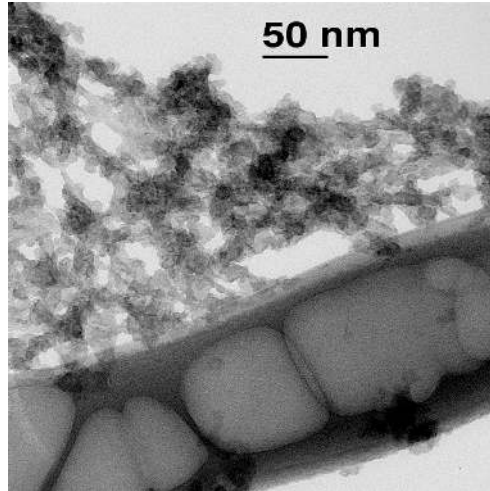**Table II.** Summary of elemental analysis data for nickel (II) oxide aerogel made via the epoxide addition method.
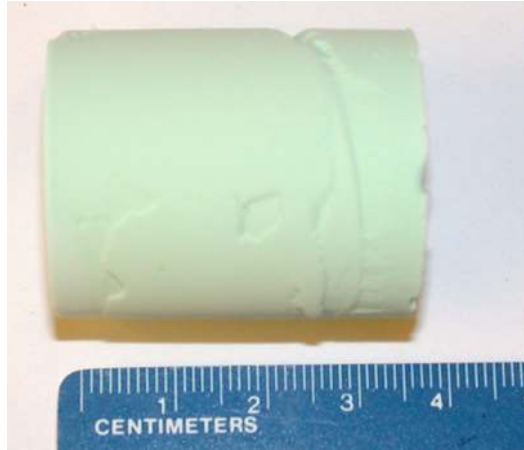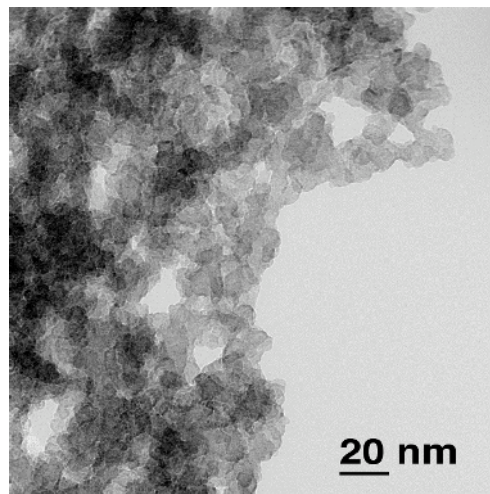
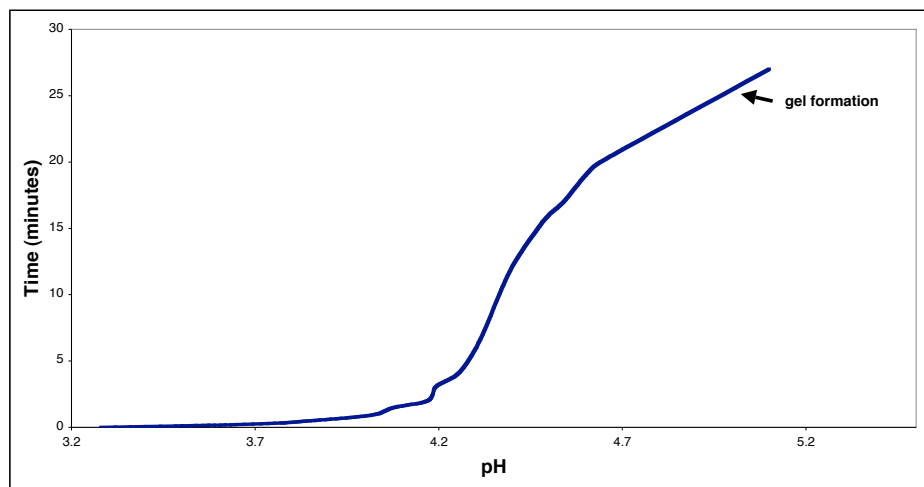**Figure 1.**
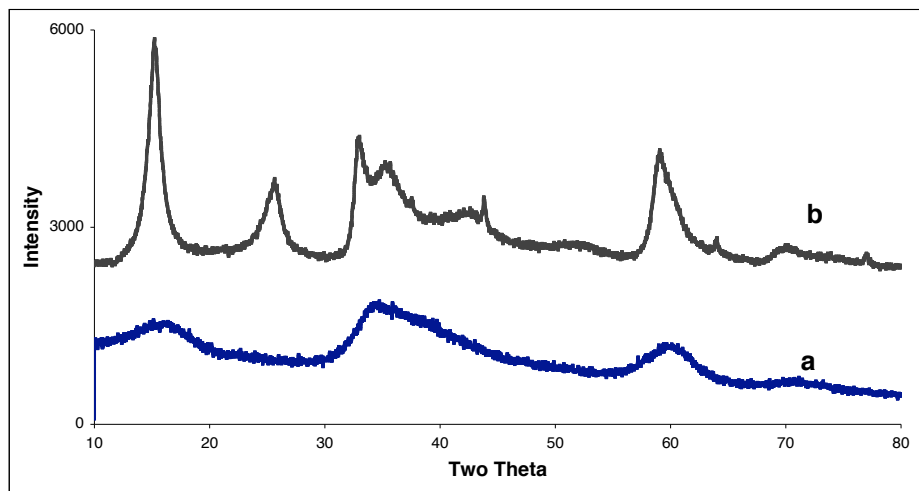
Figure 2.

a)

b)

Figure 3.

Figure 4.

Table I.

Sample	Specific surface area (m^2/g)	Pore Volume (cm^3/g)	Ave. Pore Diameter (nm)	Density (kg/m^3)
Ni (II) oxide aerogel	413	1.15	11.3	98
Ni$_3$(NO$_3$)$_2$(OH)$_4$	120	0.47	7.5	NA

Table II.

Sample	Wt.% C	Wt. % H	Wt. %Cl	Wt. % Ni
Ni (II) oxide aerogel	5.7	3.6	7.1	44.6