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Highly Monodisperse Colloidal Magnesium Nanoparticles by Room Temperature Digestive Ripening

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Nanoclusters of 25 nm sized Mg-THF have been prepared by the solvated metal atom dispersion method. Room-temperature digestive ripening of these nanoclusters in the presence of hexadecylamine (HDA) resulted in highly monodisperse colloidal Mg-HDA nanoparticles of 2.8 ± 0.2 nm. An insight into the room-temperature digestive ripening process was obtained by studying the disintegration of clusters for various Mg:HDA ratios. The Mg colloids are quite stable with respect to precipitation of particles under Ar atmosphere. Using this procedure, pure Mg(0) nanopowders were obtained in gram scale quantities. The Mg powder precipitated from the colloid was fully hydrided at 33 bar and 118 °C. Initial desorption of H_2 from samples of MgH_2 was achieved at a remarkably low temperature, 115 °C compared to >350 °C in bulk Mg, demonstrating the importance of the size on the desorption temperatures.

Introduction

Magnesium is one of the most attractive storehouses of hydrogen because it is abundant and light in addition to its reversible and high hydrogen capacity of 7.5 wt %.[1] Synthetic chemistry of magnesium nanoparticles is quite challenging because of its high reduction potential and very large affinity toward oxygen. Magnesium nanoparticles have been prepared by different routes, e.g., electrochemical reduction,[2] sonoelectrochemistry,[3] infiltration of nanoporous carbon with molten magnesium,[4] gas phase synthesis,[5] and deposition from ethereal solutions.[6] Studies aimed at the synthesis of Mg nanoparticles of <2 nm diameter have thus far not been accomplished. This is primarily due to the difficulty in synthesizing Mg nanoparticles using conventional chemical reduction methods which offer better control over particle size. Recently, Haas and Gedanken prepared 4 nm sized Mg particles by a sonoelectrochemical method[3] while Aguey-Zinsou and Ares-Fernández obtained 5 nm Mg particles by an electrochemical route.[2]

A synthetic approach of broad appeal, solvated metal atom dispersion (SMAD) process involves growth of clusters from atoms in low-temperature matrices.[7] The synthetic protocol involves co-condensation of metal atoms and a stabilizing solvent on the walls of a reactor maintained at 77 K. This is followed by the warming up of the matrix, resulting in a slurry of metal atoms interacting with one another to form particles that grow in size. Halting of the growth is achieved by the addition of an organic ligand/surfactant. The resulting colloid consisting of polydisperse metal nanoparticles is subjected to a process termed as digestive ripening, which transforms the polydisperse sample into a highly monodisperse colloid by heating the as-prepared colloid at or near the boiling point of the solvent.[8] In this as yet not fully understood process, smaller particles grow, whereas the larger particles erode until the mixture is rendered homogeneous

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(1) (a) Schlapbach, L.; Züttel, A. *Nature (London)* **2001**, *414*, 353. (b) Grochala, W.; Edwards, P. P. *Chem. Rev.* **2004**, *104*, 1283.

(2) Aguey-Zinsou, K.-F.; Ares-Fernández, J.-R. *Chem. Mater.* **2008**, *20*, 376.

(3) Haas, I.; Gedanken, A. *Chem. Commun.* **2008**, 1795.

(4) de Jongh, P. E.; Wagemans, R. W. P.; Eggenhuisen, T. M.; Dauvillier, B. S.; Radstake, P. B.; Meeldijk, J. D.; Geus, J. W.; de Jong, K. P. *Chem. Mater.* **2007**, *19*, 6052.

(5) Kooi, B. J.; Palasantzas, G.; de Hosson, J. Th. M. *Appl. Phys. Lett.* **2006**, *89*, 161914.

(6) Aurbach, D.; Lu, Z.; Schechter, A.; Gofer, Y.; Gizbar, H.; Turgeman, R.; Cohen, Y.; Moshkovich, Levi, M. E. *Nature (London)* **2000**, *407*, 724.

(7) Klabunde, K. J.; Timms, P. L.; Skell, P. S.; Ittel, S. *Inorg. Synth.* **1979**, *19*, 59.

(8) (a) Lin, X. M.; Sorensen, C. M.; Klabunde, K. J. *J. Nanopart. Res.* **2000**, *2*, 157. (b) Klabunde, K. J.; Sorensen, C. M.; Stoeva, S. I.; Prasad, B. L. V.; Smetana, A. B.; Lin, X. M. In *Metal Nanoclusters in Catalysis and Materials Science: The Issue of Size Control, Part II Methodologies*; Corrain, C., Schmid, G., Toshima, N., Ed.; Elsevier Science: Amsterdam, 2008; Chapter 11, po 233−252.

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in size and a dynamic equilibrium is established. This treatment was successfully used for gold,[9] silver,[10] copper, and zinc[11] nanoparticles. Using this methodology, we also recently synthesized highly monodisperse colloids of copper, and zinc and Cu@ZnO[11] and Au@Pd[12] core shell nanoparticles in gram quantities. Motivated by the advantages that the SMAD method offers such as easy scale up, high reproducibility, and avoidance of tedious purification procedures and the strength of the digestive ripening process, we attempted the synthesis of Mg colloids in an effort to realize smaller sized particles.

Experimental Section

Materials. Mg turnings (99%) were purchased from Aldrich. Hexadecylamine (HDA) was dried and degassed for 12 h at 100 °C. Tetrahydrofuran (THF) and toluene were dried over sodium-benzophenone. The solvents were degassed by several freeze−pump−thaw cycles. All the glassware was dried in a hot air oven around 120 °C and evacuated in hot condition just before the use to remove trace quantities of moisture.

Instrumentation. Transmission electron microscope (TEM) bright-field images and high-resolution TEM (HRTEM) images were obtained using TECHNAI F30 electron microscope operating at 200 kV. All the TEM samples were prepared inside a N_2 filled glovebox by placing 2 μL of sample on carbon coated copper grids. The powder X-ray diffraction measurements were carried out on samples placed in 0.5 mm diameter capillaries flame-sealed under N_2 atmosphere using Philips powder X-ray diffractometer and Bruker ADVANCE X-ray diffractometer using Cu Kα radiation. FTIR spectra were obtained using Perkin-Elmer Spectrum One instrument.

Preparation of Mg-THF Nanopowders by the SMAD Method. The SMAD setup is described in detail in ref 7. The tungsten crucible coated with alumina cement was connected between two water-cooled copper electrodes and a 3000 mL reactor vessel was connected to a reactor head that is equipped with copper electrodes. The entire setup was evacuated to $2-3 \times 10^{-3}$ mbar. The crucible was heated in steps and at each step the pressure was allowed to come down to $2-3 \times 10^{-3}$ mbar. The crucible was kept under gentle heating overnight. This curing process ensures removal of moisture and other volatile impurities from the crucible. In a typical experiment, about 100 mg of Mg turnings was loaded in a tungsten crucible and the crucible was resistively heated by applying appropriate voltage between two water-cooled copper electrodes. The reactor walls were maintained at 77 K using a liquid nitrogen bath and precoated with 20 mL of THF before the metal vaporization. The voltage was increased stepwise until metal vaporization began, which was apparent by the appearance of brownish-yellow color on the white matrix. Voltage was maintained at this point for 3 h and about 80−90 mL of THF was co-condensed on the walls of reactor. During this period, the matrix turned dark brown in color. When the vaporization was complete, the liquid nitrogen dewar was removed and the matrix was warmed to room temperature under an argon atmosphere. Upon warm up, precipitation of Mg-THF powder took place. The product was stirred using a magnetic stirrer and siphoned into a Schlenk tube under argon.

Preparation of Mg-HDA-THF-Toluene Colloid by the SMAD Method. About 1.5 g of HDA was taken at the bottom of the reactor. A two-way bridge equipped with THF and toluene containing Schlenk tubes were connected at the top of the reactor. Initially, about 40 mL of toluene was condensed on the walls of the reactor which was maintained at 77 K. Next, the liquid nitrogen dewar was removed and the toluene was then allowed to melt. The dewar was placed back after the toluene melted completely and fell to the bottom of the reactor. The rest of the experiment was carried out similarly to that of Mg-THF SMAD experiment. The ratio of Mg:HDA maintained in this experiment is ~1:2 (molar ratio). The siphoned colloid was brownish black in color. THF was removed from this colloid under vacuum to yield a stable transparent colloid. The stability of the final colloid largely depends on the rate of evaporation of magnesium. The typical evaporation rate to realize stable colloids is around 30−35 mg/h.

Room-Temperature Digestive Ripening of Mg-THF Nanoparticles. From a Mg-THF colloid (30 mg of Mg) in a Schlenk tube, THF was removed partially under a vacuum. To this was added 30 mL of degassed toluene under argon. A fixed molar ratio of hexadecylamine (1:0.5 or 1:1 or 1:2 Mg:HDA) was added to this solution under vigorous stirring (18 h). Stable colloids were obtained in case of higher Mg:HDA molar ratios.

Isolation of Mg Nanopowders. A large excess (1:20 toluene: THF) of degassed THF was added to Mg-HDA-toluene colloid. The colloid was then centrifuged at 3000 rpm inside a glovebox for 2−3 h. The black precipitate that resulted was washed several times with THF to remove the excess HDA. Finally, the powders were dried and stored inside the glovebox. These powders were found to be extremely pyrophoric in nature.

Hydrogen Storage Studies. MgH_2 was synthesized from Mg-HDA nanopowder (120 mg) by reacting with hydrogen (33 bar pressure) at 118 °C for 3 h. H_2 desorption studies were carried out using a laboratory made temperature programmed desorption equipment. About 55 mg of MgH_2 powder was loaded in the sample tube between ceramic wool inside the glovebox. The desorption of H_2 was monitored from 30 to 450 °C at a ramp rate of 5 °C/min under argon flow. The H_2 gas was detected using a TCD detector which was calibrated against uptake of hydrogen for a known quantity of CuO.

Caution: *Magnesium nanopowders are highly pyrophoric! They must be handled with extreme caution, taking due care not to expose the samples to air. Safety shields must be used while operating high-pressure equipment.*

Results and Discusssion

(a) Synthesis and Characterization of Mg Nanoparticles.

Micron-sized magnesium particles have been prepared by Klabunde and co-workers using the SMAD method.[13] Imamura et al. synthesized micrometer-sized Mg-THF powders and studied the hydrogen absorption properties and found that they are more active compared to bulk Mg.[14] In these two reports, the metal to THF ratios were quite low (5 mL of THF for 1 g of Mg) and the rate of evaporation of Mg was quite fast. We prepared Mg nanoparticles using the SMAD method under carefully controlled conditions. By maintaining a higher metal to THF ratio (1

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(9) Stoeva, S.; Klabunde, K. J.; Sorensen, C. M.; Dragieva, I. *J. Am. Chem. Soc.* **2002**, *124*, 2305.

(10) Smetana, A. B.; Klabunde, K. J.; Sorensen, C. M. *J. Colloid Interface Sci.* **2005**, *284*, 521.

(11) Kalidindi, S. B.; Jagirdar, B. R. *J. Phys. Chem. C* **2008**, *112*, 4042.

(12) Jose, D.; Jagirdar, B. R. *J. Phys. Chem. C* **2008**, *112*, 10089.

(13) Klabunde, K. J.; Efner, H.-F.; Satek, L.; Donley, W. *J. Organomet. Chem.* **1974**, *71*, 309.

(14) Imamura, H.; Nobunaga, T.; Kawahigashi, M.; Tsuchiya, S. *Inorg. Chem.* **1984**, *23*, 2509.

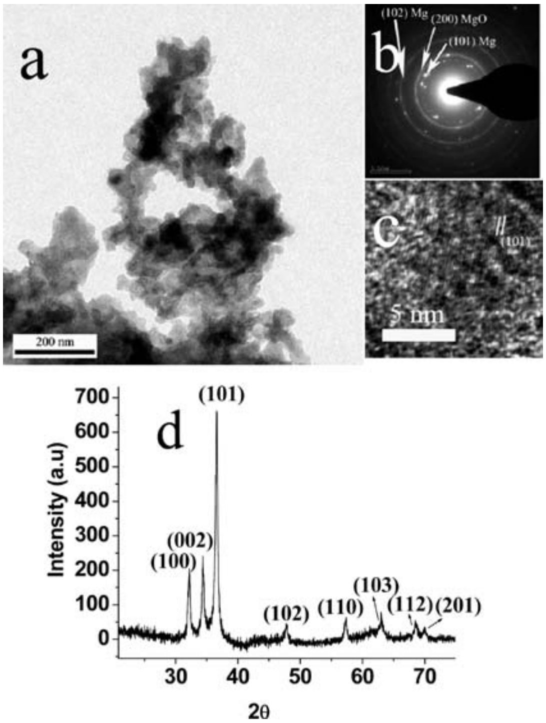

Figure 1. Mg-THF nanoparticles: (a) TEM bright-field image; (b) selected area electron diffraction pattern; (c) HRTEM image; (d) powder XRD pattern.

mL/mg) and a rate of evaporation of 30−35 mg/h, we were able to obtain Mg particles that were in the nanometer size regime. We found this rate of evaporation to be optimum. In addition, the rate of melting of the matrix has also an effect on the particle size.[15] Herein, we allowed the matrix to reach the room temperature in ∼2.5−3 h.

The colloids obtained in this manner were quite unstable toward precipitation. We noted that complete precipitation of Mg particles takes place when the matrix was warmed up to room temperature. The precipitate could be redispersed by sonication (90 W, 15 min); a TEM sample was prepared inside an inert atmosphere glovebox by placing a drop of this colloid on a copper grid. The TEM micrograph of the as-prepared Mg-THF colloid revealed the presence of completely agglomerated particles (Figure 1a). The selected area electron diffraction (SAED) pattern obtained from this agglomerated regions shows a ring pattern with some bright spots suggesting that the sample is polycrystalline having some large clusters (Figure 1b). In addition to those of Mg, we also noted rings of MgO in the SAED pattern. This is due to oxidation of the sample during the short period of exposure to air while loading the sample in the TEM instrument. The HRTEM image shows the (101) planes of hcp Mg nanoparticles (Figure 1c). The powder X-ray diffraction pattern (Figure 1d) of Mg-THF powder loaded in a 0.5 mm diameter quartz capillary tube inside the glovebox and sealed under Ar showed reflections due to pure hcp phase of Mg powder (JCPDF # 04−0770). No oxide peaks were noted in the powder pattern. The crystallite size calculated using the Scherrer equation for the (101) peak is 25 nm.

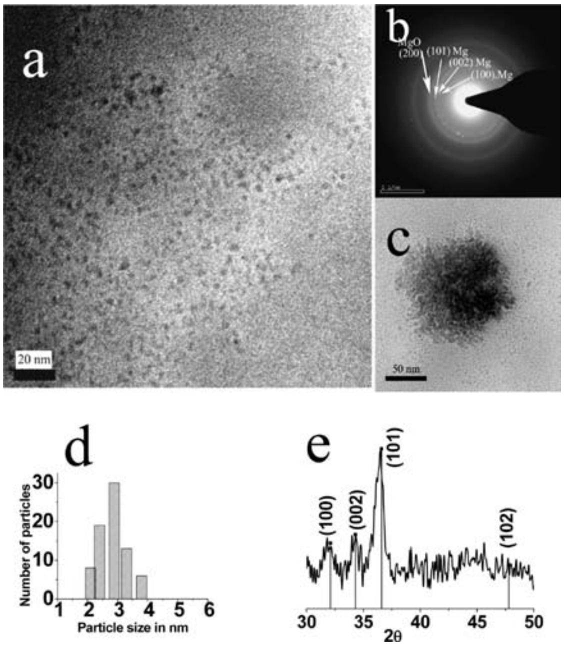

Figure 2. Mg-HDA nanoparticles: (a) TEM bright-field image; (b) SAED pattern; (c) TEM bright-field image showing the disintegration of a large cluster into smaller particles; (d) histogram showing the particle size distribution; (e) powder XRD pattern of Mg-HDA nanoparticles.

Complete precipitation of the Mg nanoparticles from the Mg-THF colloid indicates the weak capping nature of THF toward Mg clusters. The presence of a long chain ligand, e.g., hexadecylamine (HDA), could circumvent this problem of agglomeration. Therefore, we used HDA as the capping agent and placed it at the bottom of the SMAD reactor and toluene as the dispersing solvent in addition to THF. Upon warming of the matrix, stirring the colloid for an hour resulted in the complete dissolution of HDA and ripening of the colloid. A blackish brown colloid was obtained. Stable colloids (toward precipitation of Mg particles) were obtained by removal of THF under a vacuum. The resulting final colloids were found to be stable (toward precipitation) for a week. The TEM bright-field image of the Mg-HDA-toluene colloid shows the presence of highly monodisperse, well-separated, 2−4 nm sized particles (Figure 2a). This suggests that digestive ripening has taken place when the colloid was stirred in the presence of HDA at room temperature. The SAED pattern shows a ring pattern corresponding to the hcp phase of Mg and MgO (Figure 2b). Partial oxidation of Mg could not be avoided because the TEM grid had to be handled in air for a brief period during mounting in the instrument. An aliquot sampled during the digestive ripening process shows some smaller clusters/particles separating from a large cluster (Figure 2c). The powder XRD pattern of a sample taken in a capillary tube that was sealed under Ar confirmed the presence of pure Mg(0) nanoparticles (Figure 2e).

All the peaks could be assigned to hexagonal magnesium (JCPDF # 04−0770). The crystallite size calculated using the Scherrer equation is 6 nm. Magnesium particles could be precipitated from this colloid by centrifugation at 3000 rpm for 2−3 h inside a glovebox in presence of excess THF. The isolated powder was found to be highly pyrophoric.

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[15] Klabunde, K. J. In *Active Metals* Fürstner, A., Ed.; VCH Publishers: New York, 1996; Chapter 6, pp 237−275.

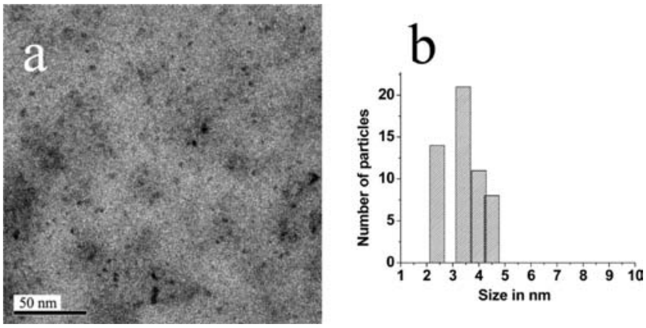

Figure 3. (a) TEM bright-field image of Mg-HDA-toluene colloid after refluxing at 120 °C for 4 h; (b) histogram showing the size distribution.

(b) Insight into Digestive Ripening Process. The fact that merely stirring of Mg-THF clusters with HDA at the bottom of the SMAD reactor resulted in a stable, highly monodisperse colloid bringing about a drastic reduction in the particle size from 25 nm to ca. 2−3 nm prompted us to probe this process in detail. Refluxing the Mg-HDA-toluene colloid at the boiling point of toluene for 3 h did not result in any further reduction in the particle size from 2−3 nm, as evidenced by TEM (Figure 3). This indicates that digestive ripening had already taken place at room temperature and refluxing the ripened sample consisting of highly monodisperse colloid will not result in further reduction in size and/or narrowing down of the size distribution.

The addition of HDA and toluene to the Mg-THF sample separately after the SMAD experiment and removal of THF in vacuo from the samples followed by stirring at room temperature for 18 h resulted in colloids. The colloids obtained by this procedure were found to be less stable (toward precipitation) in comparison to the colloids obtained in the Mg-HDA-THF-toluene SMAD experiment, which involves stirring of the colloid at the bottom of the SMAD reactor together with HDA placed in the reactor initially. This is due to the warming up of the matrix in the presence of HDA which can actually arrest the growth of Mg clusters before the colloid reaches room temperature, resulting in the formation of stable colloids. While room temperature digestive ripening of Mg colloids is unprecedented, Pileni and co-workers recently demonstrated that worm like clusters can be converted into spherical nanoclusters just by stirring with dodecanethiol for 12 h at room temperature via digestive ripening of Pd nanoparticles.[16]

Breaking down of the larger clusters into smaller particles by mere addition of HDA is due to the strong interaction of the $-NH_2$ group (Lewis base) of the capping agent, HDA with the surface atoms of Mg clusters. This favorable interaction facilitates smaller particle formation because more surface Mg atoms will be available for HDA. In addition to this effect, the long chain alkyl groups offer a lot of steric hindrance preventing agglomeration of particles thereby stabilizing the smaller clusters. A combination of these two features results in the transformation of large clusters into smaller particles via the digestive ripening process at room temperature. Strong interaction of HDA with Mg surface was evidenced by IR spectroscopy. The three N−H str bands around 3200 cm^{-1} broadened upon binding of HDA to the Mg surface (Figure 4).

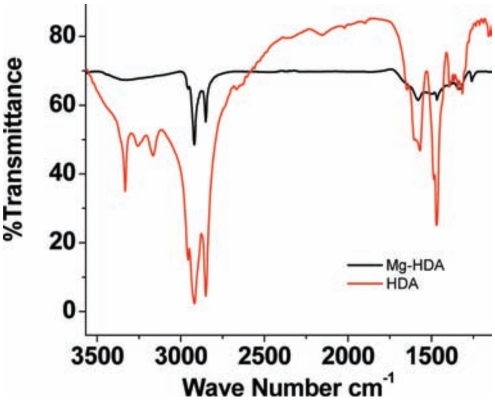

Figure 4. FTIR spectrum of HDA (red trace) and HDA-capped Mg nanoparticles (black trace).

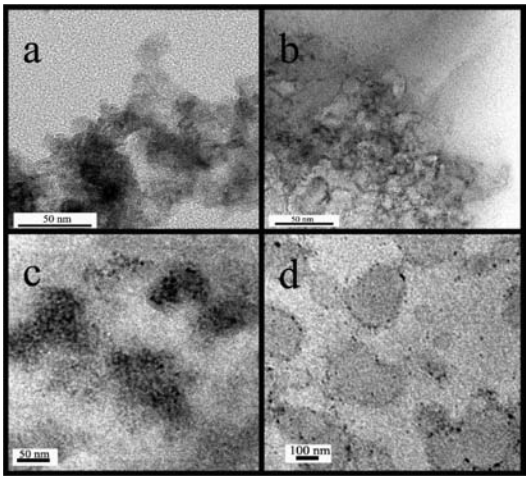

Figure 5. TEM bright-field images of Mg-HDA-toluene colloids after 2 h of ripening at room temperature for Mg:HDA molar ratio (a) 1:0, (b) 1:0.5, (c) 1:1, (d) 1:2.

To strengthen this postulate we carried out the digestive ripening of the Mg-THF nano clusters with HDA at room temperature for various molar ratios of Mg to HDA (1:0.5, 1:1, or 1:2 Mg:HDA). Samples for TEM studies were prepared inside the glovebox after 2 h of reaction time. Figure 5a shows the TEM image of the completely agglomerated clusters of Mg-toluene colloid. Disintegration of large clusters takes place after 2 h of ripening process as shown in images b and c in Figure 5 in the Mg-HDA-toluene colloids with 1:0.5 and 1:1 Mg:HDA, respectively. Digestive ripening was clearly evident in the case of 1:2 Mg:HDA sample (Figure 5d). This shows that the breakdown of the large clusters is facilitated to a greater extent by increasing the capping agent to metal ratio. A schematic representation of the room temperature digestive ripening is shown in Scheme 1.

(c) Hydrogen Storage Studies. The main impediments for use of MgH_2 in practical applications include the slow desorption kinetics and a high thermodynamic stability which results in a relatively high desorption temperature of >350 °C.[1b,17] Chen and co-workers studied the hydrogen storage properties of Mg nanowires with different diameters and found that smaller diameter nanowires exhibit faster adsorp-

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(16) Naoe, K.; Petit, C.; Pileni, M. P. *J. Phys. Chem. C* **2007**, *111*, 16249.

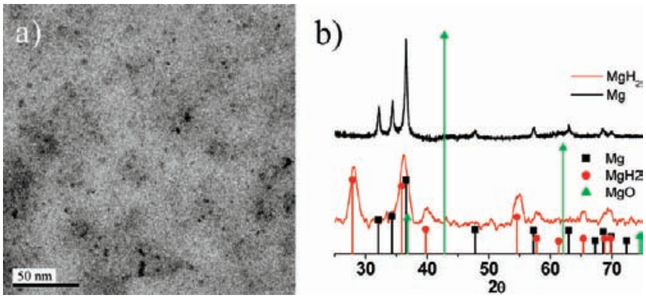
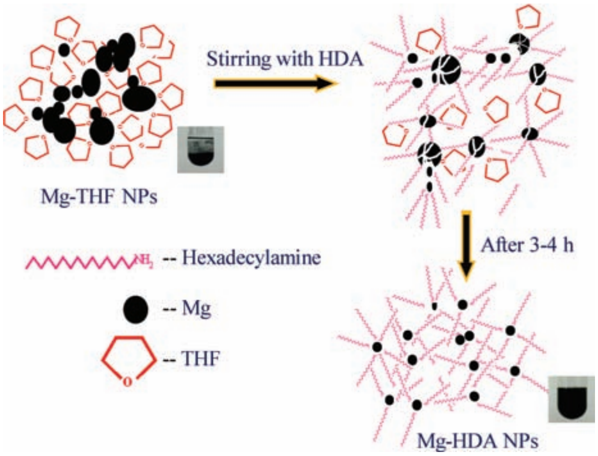


Figure 6. (a) TEM bright-field image MgH_2 nanoparticles; (b) powder XRD pattern of MgH_2 nanoparticles.

Scheme 1. Room-Temperature Digestive Ripening of Mg-THF Nanoparticles



tion/desorption kinetics.[18] Theoretical work by de Jong and co-workers and Cheung et al. indicates that the thermodynamic stability of MgH_2 decreases as the crystal grain size becomes smaller than ~2 nm.[19] Thus, the H_2 desorption temperature should drastically fall as the particle size decreases. Such size-dependent H_2 storage has recently been demonstrated by Aguey-Zinsou and Ares-Fernández in the case of Mg, and Baldé et al. in the case of sodium Alanate.[2,20] Prompted by these findings, we studied the hydrogen-storage properties of Mg-HDA nanoparticles.

We prepared MgH_2 starting from Mg-HDA nanopowder (2.8 ± 0.2 nm) and H_2 gas at a pressure of 33 bar at 118 °C. The MgH_2 thus prepared was characterized by TEM and powder XRD. The TEM image (Figure 6a) of MgH_2 particles shows that the particle size (2.0−3.5 nm) has not changed to a great extent compared to the starting Mg nanoparticles. But the carbon impurities were clearly seen as a background during TEM imaging that are from the capping agent, HDA. Because of this carbon matrix the annealing of Mg nanoparticles did not take place at 118 °C. The powder XRD pattern (Figure 6b) of the hydrogenated sample can be assigned to the β phase of MgH_2 (JCPDF#12−0697). The crystallite size calculated from the (101) peak using the Scherrer formula is 5 nm.

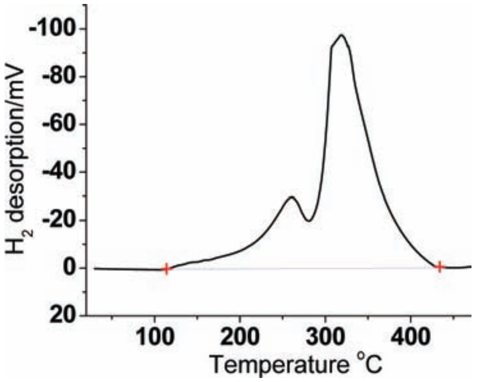


Figure 7. Temperature-programmed desorption profile of MgH_2-HDA nanoparticles at a ramp rate of 5 °C/min.

Desorption of H_2 was measured using temperature programmed desorption (TPD) over Ar gas flow at a ramp rate of 5 °C min^{-1}. The H_2 desorption was detected by a thermal conductivity detector that was calibrated against the uptake of H_2 for a known amount of CuO. Figure 7 shows the TPD profile of MgH_2. As is evident from the figure, onset of desorption of H_2 takes place at 115 °C. In light of the requirement of >350 °C for H_2 desorption from bulk magnesium, our result is significant. Even if the particle size is in the range 50−100 nm, desorption does not set in below a temperature of 200 °C.[21] Integration of the peak area of the TPD profile shows that 0.64 mmol of H_2 has desorbed (mass of Mg taken initially: 0.055 g). This is equal to 2.8 wt % H_2 in HDA-capped Mg nanoparticles (7.6 wt % in Mg). The Mg-HDA powders were washed several times (10−12 times) to remove as much of HDA as is possible from the nanoparticles' surface, so that the total hydrogen weight percent of the system after hydrogenation is not too low. This resulted in the lowering of the Mg/HDA ratio in the sample.

Even though desorption of H_2 starts at a lower temperature for Mg-HDA nanoparticles compared to that of bulk, major desorption was observed around 350 °C. This is due to sintering of Mg nanoparticles (especially in presence of lower Mg/HDA ratio) at high temperatures. According to Jong et al., MgH_2 becomes significantly unstable only for particle size below ~1.3 nm.[19a] The particle size in the present case (2−3 nm) is slightly larger to observe this effect more prominently. Further reduction in the particle size is necessary to obtain even lower desorption temperatures than what has been found in the present work. Efforts are in progress toward this direction in our laboratories.

In summary, Mg-THF nanoclusters of 25 nm have been prepared using the SMAD process by a controlled rate of evaporation of Mg metal. The as-prepared Mg-THF colloids are quite unstable and precipitation of nanoparticles took place immediately. Highly monodisperse colloids consisting of 2.8 ± 0.2 nm sized Mg particles were obtained via room-temperature digestive ripening process. As yet, such small-

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(17) Bogdanoviæ, B.; Ritter, A.; Spliethoff, B. *Angew. Chem., Int. Ed.* **1990**, *29*, 223.

(18) Li, W.; Li, C.; Ma, H.; Chen, J. *J. Am. Chem. Soc.* **2007**, *129*, 6710.

(19) (a) Wagemans, R. W. P.; van Lenthe, J. H.; de Jongh, P. E.; van Dillen, A. J.; de Jong, K. P. *J. Am. Chem. Soc.* **2005**, *127*, 16675. (b) Cheung, S.; Deng, W.-Q.; van Duin, A. C. T.; Goddard, W. A., III *J. Phys. Chem. A* **2005**, *109*, 851.

(20) Baldé, C. P.; Hereijgers, B. P. C.; Bitter, J. H.; de Jong, K. P. *J. Am. Chem. Soc.* **2008**, *130*, 6761.

(21) Shao, H.; Wang, Y.; Xu, H.; Li, X. *Mater. Sci. Eng., B* **2004**, *110*, 221.

sized colloidal Mg nanoparticles have not been reported. These colloids are quite stable with respect to either precipitation of particles or oxidation under Ar atmosphere. Using this procedure, pure Mg(0) nanopowders were obtained in gram scale quantities. The Mg powder precipitated from the colloid was fully hydrided at 33 bar and 118 °C. Initial desorption of H_2 from our samples of MgH_2 was achieved at a remarkably low temperature, 115 °C compared to >350 °C in bulk Mg, demonstrating the importance of the size on the desorption temperatures.

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Supporting Information Available: EDAX analysis and hydrogen adsorption isotherm of Mg-HDA nanoparticles (PDF). This material is available free of charge via the Internet at http://pubs.acs.org.

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