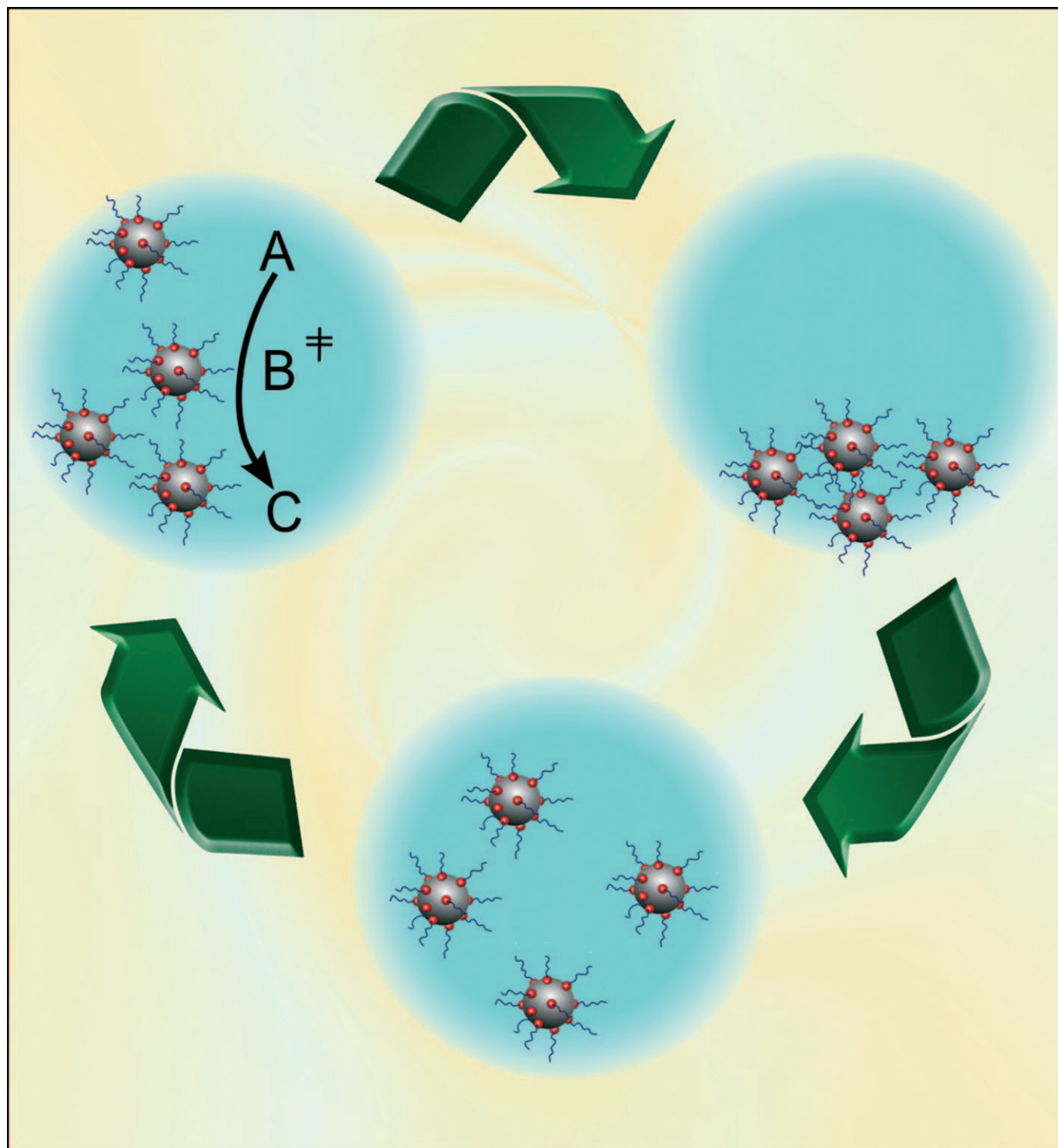


Recycling Functional Colloids and Nanoparticles

Olesya Myakonkaya,^[a] Zhiyong Hu,^[b] Muhammad Faizan Nazar,^[a, c] and Julian Eastoe*^[a]



Abstract: The stability and separation of colloids and nanoparticles has been addressed in numerous studies. Most of the work reported to date requires high cost, energy intensive approaches such as ultracentrifugation and solvent evaporation to recover the particles. At this point of time, when green science is beginning to make a real impact, it is vital to achieve efficient and effective separation and recovery of colloids to provide environmental and economic benefits. This article explores recent advances in strategies for recycling and reusing functional nanomaterials, which indicate new directions in lean engineering of high-value nanoparticles, such as Au and Pd.

Keywords: colloids · green chemistry · nanomaterials · catalyst recovery · catalyst recycling · catalyst separation

Introduction

Although nanoparticle (NP) technology has made great advances to date, realization of commercial applications has been hampered, in part due to difficulties arising from the processes of generation, recovery, and reuse of high-value nanomaterials. Numerous nanosized particles possess unique mechanical, chemical, magnetic, and optical properties, which could find potential industrial applications. However, the nanometer size also presents problems for separation, recovery, and reuse of the particulate matter. Filtration of individual NPs is essentially impossible and other classical recovery processes, such as solvent evaporation and ultracentrifugation, place high energy and high cost demands on NP processing, which compromises any possible commercial advantages. Therefore, it is particularly important to develop novel approaches to achieve reversible control over colloidal and NP stability, minimizing energy inputs, without affecting particle properties and functionality.

This work reviews simple, low-energy methods of separation, recovery, and reuse, which avoid the use of expensive equipment, or production of difficult to separate product mixtures. Importantly, the recent advances in the field of

size-selective fractionation of NPs are outlined; these are crucial for controlling the size-dependant properties of nanomaterials for specialized applications.

This article is based on extensive literature searches to capture the current state of the art. It is evident that this is an emerging field, attracting more and more interest, which is expected to grow in the near future. Although the chemistry of nanomaterial generation can be now considered as quite mature [To give an impression of the sheer scale of this work, a recent Google Scholar search on 'nanoparticle synthesis' returned over 124000 articles to date.], the field of NP manipulation, control, and recycling is more embryonic at the present time. The new concepts covered in this article concern the use of external fields, pressure, antisolvents, pH, UV light, and self-assembling colloidal solvents (as opposed to simple molecular solvents) to control NP stability: all of these approaches open up new horizons for potential applications of nanotechnology.

Separation and Recovery Concepts

Magnetic separations: Magnetic separation provides an attractive method for recycling NPs. The main advantage of this method is the stability of using an external magnetic field stimulus, which avoids additional changes in the composition of the bulk system, for example, the use of additives.

Numerous studies have been recently devoted to magnetic NPs due to their high surface area, magnetic properties, and high dispersibility in various solvents, with reference to applications in catalysis. Significantly, these NPs can be simply recycled under an external magnetic field, overcoming difficulties in product separation and catalyst contamination. Therefore, magnetic functionalization of NPs is beginning to receive increasing attention.^[1-5]

Oliveira et al. used a permanent magnet to facilitate the recovery of Au-NP catalysts.^[1] The use of magnetically recoverable supports for the immobilization of Au-NPs led to a clean, fast, and efficient separation of nanocatalysts after the reaction. The catalyst was prepared by immobilization of Au-NPs on the surface of core-shell silica-coated magnetite NPs. After oxidation reactions involving either benzyl alcohol, 1-phenylethanol, or cyclohexanol, the nanocatalysts were magnetically recovered and reused with fresh addition of substrate.^[1] The recovered particles were analyzed by atomic absorption spectroscopy (AAS), which showed negligible Au leaching. However, TEM analysis demonstrated a change in the catalyst support morphology (core-shell Fe₃O₄@SiO₂) and aggregation of Au-NPs. These observations were explained by the presence of base (K₂CO₃) in the reaction mixture.^[1]

Another study showed the application of enzyme-coated magnetic NPs as biocatalysts in the sulfoxidation of thioanisole, and subsequent recovery under an external magnetic

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field.^[5] The enzyme was immobilized on NPs, which contained an iron oxide core and a thick poly(glycidyl methacrylate) shell. The authors reported that polymer shells significantly increased the nanobio-catalyst stability, retaining catalytic activity after eleven cycles.^[5]

A magnetic nanocatalyst containing platinum supported on silica-coated magnetite NPs was successfully applied for hydrogenation of various ketones in toluene.^[2] The novel catalyst was easily separated by using an external magnetic field and recycled several times, retaining catalytic activity with a loss of only 10% after the eighth cycle (Figure 1).

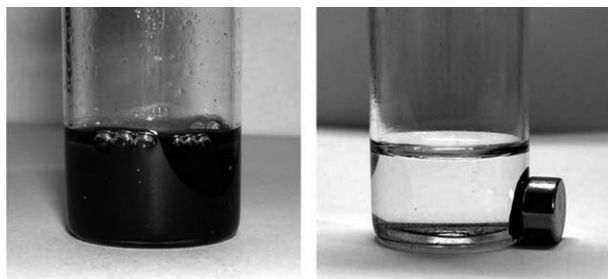


Figure 1. The Pt/SiO₂/Fe₃O₄ catalyst homogeneously suspended in toluene (left) and magnetically separated (right). Reprinted with permission from reference [2], copyright 2009, Elsevier.

Recovery of nanomaterials by using antisolvents: An interesting approach is the addition of CO₂ as an antisolvent, since it provides an inexpensive, benign, nontoxic means to efficiently control the NP stability.^[6] Drawbacks of this technique are that sophisticated and expensive infrastructure is required to use compressed CO₂. Additionally, it is essential to comply with safe high-pressure procedures when working typically above 60 bar for liquid CO₂. It was previously reported that the majority of NPs prepared in reverse micelles could be separated from the parent surfactant-rich phase and recovered by compressed CO₂. For example, NPs such as ZnS,^[7,8] Ag,^[9] TiO₂,^[10] TiO₂/SiO₂,^[11] and CdS/ZnS^[12] were separated from reverse micelles in this way.^[6]

Recent advances in the field include a combination of CO₂ and aqueous surfactant solutions.^[13] By controlling the pressure of CO₂ at a fixed temperature, it was possible to induce a phase transition of the Au–NP dispersions stabilized by the nonionic surfactant Triton X-100, separating Au–NPs from the surfactant-rich aqueous phase. TEM results demonstrated that the recovered NP sizes and shapes were similar to the freshly prepared nanomaterials with spherical diameters of around 10 nm.^[13]

Recent very interesting studies reported the use of CO₂ as an antisolvent to fractionate NPs by size.^[14] This is particularly useful to investigate the size-dependent properties of nanomaterials.^[14] Roberts et al. employed CO₂ to enhance the deposition of NP thin films from organic solvents.^[15] Ag–NPs of different size stabilized by various particle ligands were separated by controlling the CO₂ pressure to expand the majority solvent, hexane, controlling the parti-

cle–solvent interactions. This gas-expanded solvent approach was also used for size-selective NP separation, allowing fine control over monodispersity, since expansion of the organic solvent and hence its solvent strength can be simply tuned by varying the CO₂ pressure. Particle polydispersity was reduced by applying recursive fractionation, as shown in Figure 2.^[16]

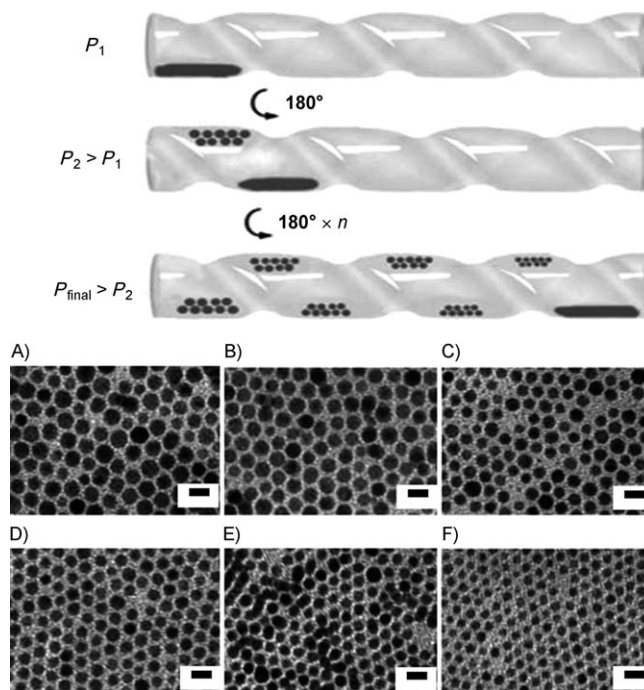


Figure 2. Top: NP size selection by recursive pressurization of the CO₂ gas-expanded liquids followed by 180° tube rotation. Bottom: TEM images of NPs separated from hexane by CO₂ pressurization from A) 0 to 500, B) 500 to 550, C) 550 to 600, D) 600 to 625, and E) 625 to 650 psi. F) Particles remaining in solution after CO₂ pressurization to 650 psi. Scale bars are 10 nm. Reprinted with permission from reference [16], copyright 2005, American Chemical Society.

A similar approach was used to fractionate polydisperse alkanethiol^[17] and dodecanethiol-stabilized Au–NPs^[18] into narrow, monodisperse fractions by employing supercritical ethane and hexane. The fractionation was proven by TEM, UV/Vis spectroscopy, and mass spectrometry analyses.

Another approach, not requiring pressure, employed liquid antisolvents, such as squalane, to recycle Au nanocatalysts, demonstrating a simple, low-energy method of solvent-quality tuning.^[19,20] Advantages of this approach over the use of CO₂ are that only standard laboratory operating conditions are necessary: no major infrastructure or hazardous working practices are needed, just readily available liquid solvents and regular laboratory glassware. Au–NPs, stabilized by sodium bis(2-ethylhexyl)sulfosuccinate (AOT) and hexaethylene glycol monododecyl ether (C₁₂E₆) were cleanly separated from octane by addition of an antisolvent (squalane, mole fraction in the solvent mixture X_{sq} = 0.483), and then redispersed in octane. The morphology and proper-

ties of the NPs were not affected by the recovery process, as shown by TEM and UV/Vis spectroscopy analyses. This approach was applied to explore application as an oxidation catalyst. Au–Pd–NP mixtures were used in these studies because they have an inherently higher reactivity than monometallic Au–NPs. Blank reactions of cinnamyl alcohol and octen-1-ol oxidation showed no reaction in the absence of NPs. After the reaction with Au–Pd nanocatalysts had occurred, the NPs were recovered by addition of squalane ($X_{\text{sq}}=0.383$), and redispersed in octane. The redispersed Au–Pd–NPs were then reused, interestingly the Au–Pd–NPs retained 50–70% of the initial activity.

Recent results obtained by using aqueous dispersions: Addition of antisolvents to NP dispersions represents a simple, inexpensive, and robust approach to separation and recovery of nanomaterials. Most of the recent studies have been focused on recycling nanomaterials from oily media, however, it is particularly important to achieve recovery in water to facilitate realization of commercial applications. This concept has been employed to control the stability of Au–NPs synthesized in aqueous solutions. Stability/instability/stability transitions of Au–NPs have been induced to separate nanomaterials from aqueous dispersions simply by the addition of THF as an antisolvent.

Au–NPs synthesized as described in reference [21] are red in color (inset a in Figure 3); these were characterized by TEM (JEOL 1200EX operating at 120 kV) and UV/Vis spectroscopy (Unicam UV2). TEM results showed the Au–NP size to be around 7–11 nm (Figure 4a). The wavelength of maximum absorbance, $\lambda_{\text{max}}=530$ nm (Figure 3a), which depends on size and shape, agrees with literature values for Au–NPs in this size range.^[22]

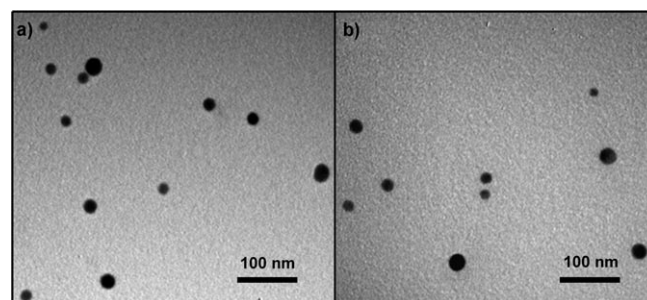


Figure 4. TEM images of the original aqueous Au–NP dispersion (a) and redispersed particles in water after separation (b).

Au–NPs were readily separated from the supporting aqueous media by the addition of THF antisolvent. The most efficient separation occurred at a THF mole fraction of $X_{\text{THF}}=0.335$, followed by extraction of the supernatant. Au–NPs were collected from the bottom of the vial and redispersed in pure water. The NPs retained the size of 7–11 nm and spherical shape, as confirmed by TEM (Figure 4b). UV/Vis spectroscopy showed that around 99% of NPs were recovered and could be redispersed after the first cycle (see Figure 3c).

The recovery procedure outlined above was repeated five times, however, 0.10 M NaCl solution (volume fraction of NaCl solution, $\phi_{\text{NaCl}}=0.082$) was also added to the NP dispersion/THF mixture to achieve maximum NP separation. This procedure resulted in 86% recovery of the NPs after a fifth cycle. Importantly, no changes in UV/Vis spectra were observed after repeated recovery/redispersion, suggesting that the recycling process had no notable effect on the NP structure or stability.

The application of an antisolvent to separate and recover NPs offers fine control over colloid stability simply by tuning the solvent quality, requiring only standard laboratory conditions and infrastructure. Significantly, this approach opens the door to recycling and reusing NPs for potential applications, leading to reversible NP stability/instability/stability transitions.

Recycling NPs by employing a colloidal solvent: Another effective concept for reversible control over NP stability has been recently introduced. By applying a background-supporting colloidal fluid (in place of a normal molecular solvent) it is possible to induce colloid stability/instability transitions to separate and recycle NPs. This approach offers a number of advantages over traditional methods, such as low cost and the ability to avoid energy-intensive pressure changes or centrifugation cycles, potentially leading to significant economical and environmental benefits.

For example, Au–NPs can be generated by using water-in-oil (w/o) microemulsions, such as those described in reference [23]. After the reduction reaction the systems in a microemulsion medium comprise Au–NPs and “reaction debris”, such as unreacted excess w/o droplets and surfactant. As shown recently, the Au–NPs can be efficiently sepa-

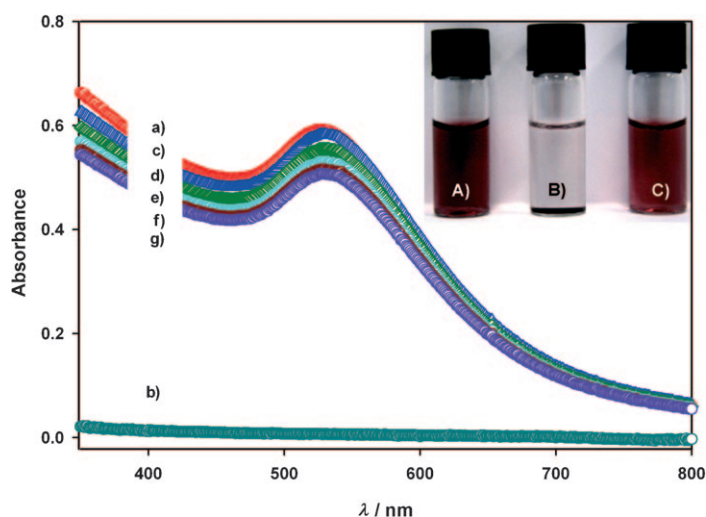


Figure 3. UV/Vis spectra of Au–NPs synthesized in water (a, vial A), the top phase of the separated dispersion after the addition of THF ($X_{\text{THF}}=0.335$) (b, vial B), and the Au–NPs redispersed in water after one to five cycles (c (vial C), d, e, f, and g, respectively). Inset: Control over Au–NP stability.

rated by inducing a phase transition of the background supporting microemulsion.^[23] NPs stabilized by (1-hexadecyl)trimethylammonium bromide (CTAB) were concentrated in the upper phase of the background water/CTAB/butanol/octane microemulsion simply by adding excess water. ¹H NMR spectroscopy showed that the upper phase mainly consisted of octane (96 wt %) and a small quantity of butanol (4 wt %). The remaining water, octane, butanol, and CTAB were found to be in the lower phase. On the other hand, UV/Vis spectroscopy demonstrated that around 80% of Au-NPs from the original microemulsion/Au-NP mixture partitioned into the upper phase. After separation, the NP-rich phase was redispersed in extra octane. TEM results confirmed that there was no change in particle size and shape—spheres, triangles, and short rods 10–40 nm in size were observed in both the original and redispersed Au-NPs dispersions.^[23]

Recently, Jiang et al. introduced an interesting approach to recovery of nanomaterials by employing the cloud point phenomenon, which permits separation of surfactant solutions into surfactant-rich (lower) and -poor (upper) phases by temperature control.^[24] The low-cost, nonionic Triton X-114 surfactant was added to aqueous dispersions of various NPs (e.g., CdS, ZnS, Fe₃O₄, TiO₂, Ag, and Au). The systems were heated to induce liquid–liquid (L–L) separations, with the NPs concentrated in the lower phases after the transition. Then the nanomaterials were redispersed in the surfactant-rich phase by the addition of NaCl (3.4 mM). The cycles were repeated ten times with no further addition of NaCl. TEM and UV/Vis spectroscopy were used to follow possible changes in NP size, shape, and stability. These studies confirmed that model NPs showed no aggregation for over two months, and no changes in particle dimensions and shape occurred.^[24]

Other interesting work has been focused on the separation and recovery of CdS and ZnS NPs by suspending them in a background-supporting colloidal fluid, which itself shows a L–L critical-type phase transition at a temperature T_c .^[25] At 62 °C the microemulsion/CdS–NP mixtures separated into two layers, with most of the NPs in the upper portion. Microemulsions containing ZnS–NPs separated into two equilibrium phases at 69 °C with ZnS–NPs concentrated in the lower phase. UV/Vis spectroscopy and AAS confirmed strong partitioning of the NPs between macroscopic phases.^[25]

A temperature-induced approach has been applied to Au–NPs synthesized in microemulsions containing wormlike micelles.^[26] The systems were cooled to induce L–L separations, and UV/Vis spectroscopy demonstrated that NPs concentrated in the upper phase. Elemental analysis showed that the majority of the surfactant and byproducts were present in the lower phase, suggesting that the NPs were separated and purified from the multicomponent reaction mixture.^[26]

Applications of pH and thermoresponsive materials: Considerable attention has been paid to designing reusable cata-

lyst systems by using thermoresponsive polymers. The most common polymer employed in these studies is poly(*N*-isopropylacrylamide) (PNIPAM) due to thermoreversible swelling transitions.^[27–30] Typically, noble-metal nanocatalysts have been stabilized by PNIPAM to form thermoresponsive nanostructures, which can be easily recovered from the catalytic reaction medium. For example, the activity of Au nanocatalysts stabilized by PNIPAM-containing copolymers was reversibly turned on and off by using temperature control in the reduction reaction of 4-nitrophenol.^[30] Blank experiments showed no reaction without the catalyst. The Au–NP separation was induced at 50 °C, concentrated nanomaterials were collected, recovered by cooling the system to 4 °C, and then reused in a second run of the reduction reaction at 25 °C.^[30]

Another interesting approach is based on pH-induced heteroaggregation of mixed positive/negative aqueous polymer microgels.^[31] The Au–NPs were incorporated into microgels containing anionic PNIPAM and cationic poly(NIPAM-co-2-vinylpyridine) (P2VP-PNIPAM). The stability/instability transitions of the background microgels were controlled by using pH, inducing flocculation of the NPs (pH 3), followed by their redispersion (pH 10). AAS showed almost complete recovery of the NPs.^[31]

Other Approaches

Another viable approach is the use of NP/microemulsion-based gel (MBG) mixtures.^[32] Numerous studies have been focused on physical and structural characterization of MBGs, which are interesting composite colloidal systems with unusual bicontinuous oil/water structures and unique transport properties.^[33–37] These MBG systems were widely used in mixtures with hydrophilic enzymes. Robinson et al. showed that immobilized lipases in MBGs retained catalytic activity with a nonpolar solvent, such as heptane.^[38] Lipases incorporated in MBGs were then recycled, retaining 75% of the activity after 30 d.

However, little work has been done on immobilization of nanocatalysts in MBGs with their further recovery and reuse. In a recent study, various inorganic NPs were doped into these thermoreversible MBGs to generate interesting and potentially useful composite systems (Figure 5).^[32] A variety of different NP types was employed and small-angle neutron scattering (SANS) showed minimal changes to the organogel structure after particle inclusion. Furthermore, UV/Vis absorption demonstrated only minimal particle aggregation in the gel matrices, suggesting that it is possible to form stable systems with potential applications in functionalized nanomaterials and catalysts. Interestingly, these NP–MBG mixtures could represent a convenient method for the reuse of high-value nanomaterials.

Another related microemulsion/gel system has been developed specifically for interesting applications in the amelioration and restoration of historical works of art.^[39] Art restoration is a delicate business, and a major challenge is to

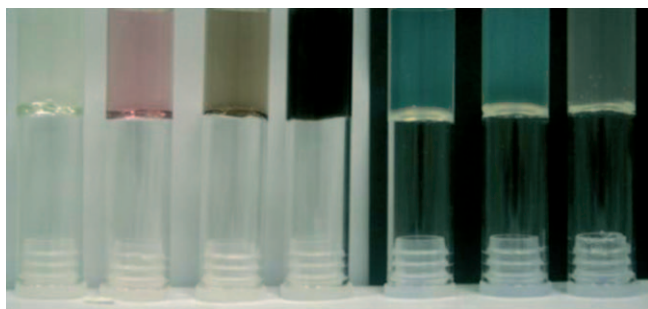


Figure 5. Various NPs dispersed in MBGs. Left to right: MBG with no NPs, gold, gold-palladium, magnetite, 25 nm silica, 80 nm silica, TiO₂ NPs. Reprinted with permission from reference [32], copyright 2010, Royal Society of Chemistry.

clean painted surface layers effectively, but importantly, without damaging them. Neat organic solvents can result in irreversible damage: penetration into sublayers cannot be easily controlled (or arrested) because the fluid solvents are readily drawn into the artwork by capillary action. However, gelled systems can significantly reduce this effect, affording a much better selectivity and control over the delicate cleaning procedure. Baglioni et al.^[39] have developed magnetically active oil-in-water microemulsions, gelled with added acrylamide ethyleneoxide polymers; surface-stabilized CoFe₂O₄-NPs entrapped in the MBG networks provide the magneto-responsive function. Figure 6 shows how the gel can be put into contact with a solid substrate (marble), which then gently invades the surface layers, and then is readily re-

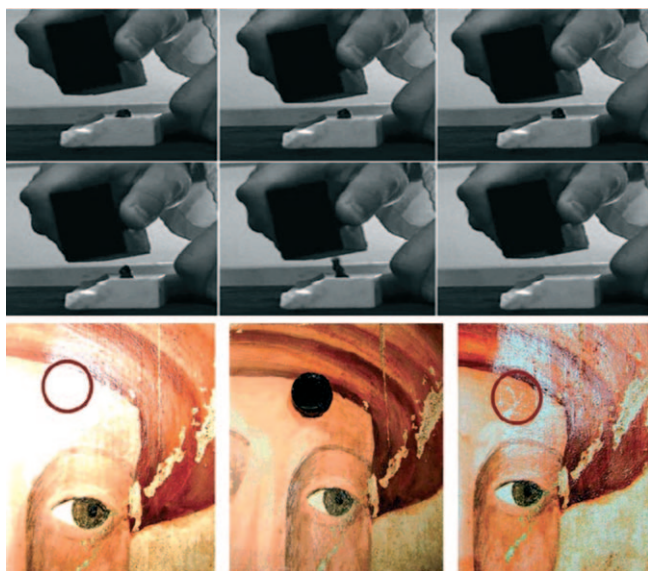


Figure 6. Magnetic MBGs for the restoration of historical works of art. Top: application and magnetic removal of MBG from a marble surface. Bottom: application of the nanomagnetic gel to a fresco painting. Left: original fresco, the circle shows the area to be treated. Center: gel treatment. Right: area cleaned after application of the magnetic MBG. Reprinted with permission from reference [39], copyright 2007, American Chemical Society.

moved by the application of an external magnetic field. Importantly, the gel and entrapped surface contaminants are removed without leaving any residue. The action and efficacy of these magnetic MBGs for cleaning a painted fresco can be seen in Figure 6. This technology^[32,39] demonstrates that complex organic colloidal structures, such as polymer-loaded microemulsions, can be used in combination with functionalized and responsive inorganic NPs to provide novel composite materials. It is anticipated there is great potential to exploit further this concept for generating advanced materials to meet the demands of challenging applications.

Recent work has also been focused on UV-induced separation of NPs in microemulsion media.^[40,41] UV-light irradiation of dispersions of Au-NPs stabilized by a photodestructible anionic surfactant C6-PAS ($X_{\text{C6-PAS}} = 10\%$), mixed with an inert nonionic C₁₂E₆^[40] and AOT/C6-PAS-stabilized silica NPs resulted in irreversible aggregation of nanomaterials.^[41] Hence, it could be possible to enhance photorecovery of inorganic NPs by using alternative photolyzable surfactants to achieve a reversible phase transition and recycle NPs.

Summary and Outlook

The principles of green chemistry require minimizing energy consumption and prevention of waste. Therefore, it is essential to develop new strategies, which allow 'clean' and reversible separation and recovery procedures. Numerous studies have been devoted to NP stability over recent decades, and the majority of published procedures require highly energetic processes, such as centrifugation or solvent evaporation.

However, a few alternative concepts to these conventional separation techniques have been recently outlined. The application of magnetic fields, pH and thermoresponsive materials, molecular antisolvents, or nanostructured colloidal solvents provide effective and efficient methodologies for recycling NPs without significant costs, time consumption, or energy demands. Nevertheless, much work has to be done to develop further novel approaches, such as applications of MBGs or photoswitchable systems to realize the full potential of NPs in commercial applications.

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