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# Nanocomposite particles containing semiconductor and magnetic nanocrystals: fabrication and characterization

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## ABSTRACT

In this paper several approaches to fabricate semiconductor magnetic nanocomposite are reported. Several thiol- and amino- silane cross linked molecules were used to couple and embed CdSe and $Fe_3O_4$ nanoparticles into silica particles. The resulting nanocomposites were characterized by optical spectroscopies, transmission electron microscopy, electron paramagnetic spectroscopy and fluorescence optical microscopy. The new developed nanocomposite particles posses the advantage of being both magnetic and luminescent. The chemical functionality rich surface of these new nanoparticles could enable their application in bioassays, cell separation and drug delivery.

**Keywords:** Quantum dots, magnetic nanoparticles, photoluminescence, transmission electron microscopy

## 1. INTRODUCTION

Nanoparticles have attracted great attention of many researchers working in the interface of chemistry and physics of materials. The decrease of particles size leads to significant modification of materials properties. For example, in semiconductor nanoparticles the band gap increases with decreasing particle diameter (1). In magnetic nanoparticles a transition from ferromagnetism to superparamagnetism with decreasing particle diameter is observed (2). The interesting properties of nanoparticles originate from their intermediate standing between single atoms, molecules and solids. Besides the variations in their electronic structures, the geometrical structures of nanoparticles may also change with decreasing particle diameter. The most known examples are the discovery of the cage-like structure of $C_{60}$ clusters (3) and the recent finding of a similar to fullerene type structure in binary chalcogenide semiconductor nanoparticles (4).

The first examples of preparation and studies of composites consisted of small semiconductor clusters were reported in the early 1970s (5). The rigid alumosilicate frames of zeolite crystals were used as the host for small semiconductor inclusions. Size and size distribution of guest materials were controlled via physical confinement in the regular systems of voids and channels of zeolites. The main disadvantage of this synthetic approach was the low concentration of guest materials due to blockage of channels at the first moment of the incorporation of precursor molecules. The rapid progress in semiconductor nanocrystal research, both in chemical synthesis and physical understanding has been made only in the last ten years following the pioneer work of Bawendi et al. who synthesized for the first time CdSe quantum dots using organometallic precursors (6). The method enabled controlling the size, shape and surface of these nanoparticles. The semiconductor nanoparticles showed a linear discrete absorption spectra and size-tunable photoluminescence ranging from the far infrared to deep ultraviolet range. The nanocrystal size and shape were found to be the most important parameters in controlling the optical transition energies. The exciting new optical properties of semiconductor nanoparticles enabled the application of these nanocrystals in optoelectronic circuits and as luminescent labels of biomolecules.

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Magnetic particles have a longer history. Ferrofluids is one of the common and a well known example of successful application of magnetic particles. On and off behavior of single domains in the presence and absence of magnetic field encouraged researchers to use these particles in biomedical applications like drug delivery and magnetic separation (7, 8).

Given the high utility of luminescent and magnetic particles in biological applications it is somewhat surprising that only few studies focused on the developed on nanocomposite particles that show simultaneous magnetic and luminescent properties. Hatanaka et al (9) have immobilized FTIC-avidin to the surface of magnetic nanoparticles and used them to observe magnetic patterns written on a floppy disk. Lu et al (10) prepared iron oxide silica core-shell nanoparticles and incorporated fluorescent dyes into the silica shell by covalent coupling. Laurent Levy et al (11) prepared thin silica particles that were coated with magnetic nanoparticles, and a bio-targeting group LH-LR labeled with a fluorescent dye and used these particles for cancer cells imaging. Lu and coworkers prepared up-converting fluorescent magnetic nanoparticles by covering a magnetic core with ytterbium and erbium co-doped sodium yttrium fluoride, an efficient infrared-to-visible up-conversion phosphor (12). In our laboratory we recently reported the synthesis and characterization of magnetic nanoparticles coated with semiconductor nanocrystals and their application for breast cancer cell separation from blood (13). In this paper we report new approaches for the synthesis of semiconductor – magnetic nanocomposite particles. In the first approach CdSe/ZnS quantum dots are directly attached to the functionalized surface of magnetic nanoparticles. Then, the synthesis of silica nanoparticles containing magnetic and semiconductor nanocrystals is described.

## 2. FABRICATION AND CHARACTERIZATION OF BARED SEMICONDUCTOR- MAGNETIC NANOCOMPOSITE

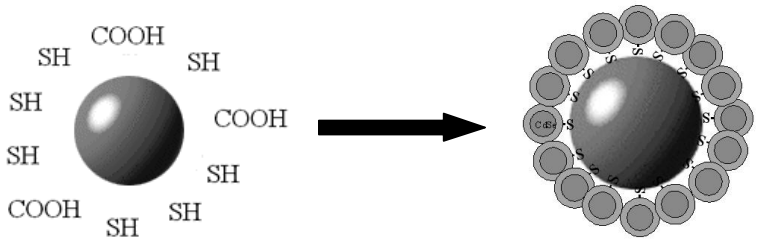
In this part we will describe the formation of luminescent and magnetic nanocomposite particles that consist of superparamagnetic core particles ($\gamma$-$Fe_2O_3$) and a layer of luminescent semiconductor quantum dots (QDs) of CdSe/ZnS on their surface. CdSe QDs are often coated with a layer of ZnS, which has larger band gap, to passivate their surface and to increase their exciton band edge emission through the termination of surface defects, which play important role in nonradiative processes. To enable miscibility of the QDs in aqueous solution, their surface was modified with carboxyl functional groups (14). Alternatively QDs were encapsulated in water miscible shells like liposomes (15), silica micelles (16) and glyconanospheres (17). QDs were also encapsulated in dendrimers (18, 19) to increase their stability against photo-oxidation and photobleaching. In our laboratory luminescent CdSe/ZnS QDs of green emission and red emission colors were synthesized based on a method developed by Peng et al., with minor modifications (20). 12.7 mg cadmium oxide and 160 mg lauric acid were mixed in a 100 ml three-neck flask. The mixture was heated to ~200 °C in a mantle to fully dissolve the cadmium oxide in the lauric acid solution. Then, 1.94 g trioctylphosphine oxide (TOPO) and 1.94 g hexadecylamine (HDA) were added to the solution. The mixture was heated to a temperature higher than 280 °C and kept under a dry nitrogen atmosphere. Upon reaching the desirable temperature the mantle was removed and 2 ml trioctylphosphine (TOP) solution containing 80 mg Selenium powder was rapidly injected into the solution under vigorous stirring. It was previously shown that the diameter of the formed QDs depends on the reaction temperature with smaller particles formed at higher temperature. The color of the mixture changed from clear colorless to yellow, orange or red depending on the exact temperature. To form ZnS coating on the CdSe QDs the mixture was cooled to ~200 °C. Then, after being kept three minutes at this temperature a solution containing 250 µl hexamethyldisllathiane ($(TMS)_2S$) and 1 ml diethylzinc ($Zn(Et)_2$) premixed in 2 ml TOP was gradually injected into the solution over a minute. The reaction mixture was kept at 180 °C and stirred for one hour. The solution was cooled to room temperature and the resulting sample of CdSe/ZnS QDs was washed three times with methanol and chloroform. The QDs showed extremely high quantum yield of about 0.7.

Polymer coated $\gamma$-$Fe_2O_3$ superparamagnetic magnetic beads were purchased from Indicia Biotech, France. The nanometric $\gamma$-$Fe_2O_3$ magnetic particles were coated with dimercapto-succinimid acid (DMSA) to stabilize and functionalize the ferrofluid. The surface of the ferrofluid was covered with free thiol (SH) and carboxyl (COOH)

residues (3:20 thiol to COOH ratio) to enable covalent coupling of various ligands to the magnetic particles (21). A TEM image (figure 2a) of the polymer coated particles shows that their magnetic core averages 10±15% nm in diameter. The hydrodynamic diameter of the particles that include the polymer layer was found to be around 20±10% nm based on dynamic light scattering measurements. The particles were fully miscible in aqueous solution and no aggregation was observed.

The coupling between the luminescent CdSe/ZnS QDs and the magnetic beads was based on thiol chemistry. Thiols (-SH) are probably the most utilized functional groups for stabilizing and modifying CdSe QDs. Thiol groups form stable bonds with metals on the surface of QDs like cadmium and zinc. However, the coupling reaction between the QDs and magnetic beads presented a difficulty since the trioctylphosphine oxide (TOPO) capped CdSe/ZnS QDs were dissolved in chloroform while the polymer-coated magnetic beads were dispersed in water. We found that running the coupling reaction in a 10:5:1 mixture of chloroform:methanol:water yielded the best nanocomposite particles with minimal aggregation. To carry out the coupling reaction we first transferred 1 ml of 1 μM CdSe/ZnS QDs into a 5 ml vial. Then we added 500 μl methanol to the solution. This was followed by the slow injection of 100 μl 0.1 μM magnetic beads water suspension to the solution under sonication and vigorous stirring. Under these reaction conditions the molar ratio between the QDs and the magnetic particles was 100:1. The excess of QDs was imperative to prevent aggregation of the magnetic beads. Under vigorous stirring the aqueous and organic phases formed an even suspension. The suspension was stirred for 1 hour to form the nanocomposite particles. The luminescent/magnetic nanoparticles were then separated from the solution by using a permanent magnet (Average cross sectional force density 16.6 $T^2/m$) and washed several times with methanol.

**Figure 1 –** Thiol and carboxyl modified $\gamma$-$Fe_2O_3$ beads are reacted with CdSe/ZnS QDs to form the luminescent/magnetic nanocomposite particles.




The sample was vortexed briefly and sonicated for 15 minutes to prevent aggregation. A representative TEM image of the nanocomposite particles is shown in figure 2b. The particles average 20 nm in diameter with a size distribution of about 15% and show minimal or no aggregation. Assuming the magnetic beads are fully covered with QDs, the maximum number of QDs per magnetic bead could be estimated based on the following equation:

$$N = 2\pi (R_{Fe} + R_{QD})^2 / \sqrt{3}\, R_{QD}^2$$

$R_{Fe}$ is the radius of the magnetic beads and $R_{QD}$ is the radius of the smaller QDs. This estimated expression is derived by dividing the surface area covered by the small QDs on the larger iron oxide particle by the area covered by a single QD on the iron oxide particle surface. The calculation assumes close packing of QDs on the magnetic particle surface and takes into consideration the gaps between the QDs. For example, for a magnetic particle diameter of 10 nm, the maximum number of QDs increases from 45 to 133 when the QDs diameter decreases from 4 to 2 nm. A high resolution TEM image of an individual magnetic bead coated with CdSe/ZnS QDs is shown in figure 2c. Gaps between the QDs can be seen, indicating imperfect coating. It also implies that the number of QDs per magnetic particle would be lower than the upper theoretical limit.

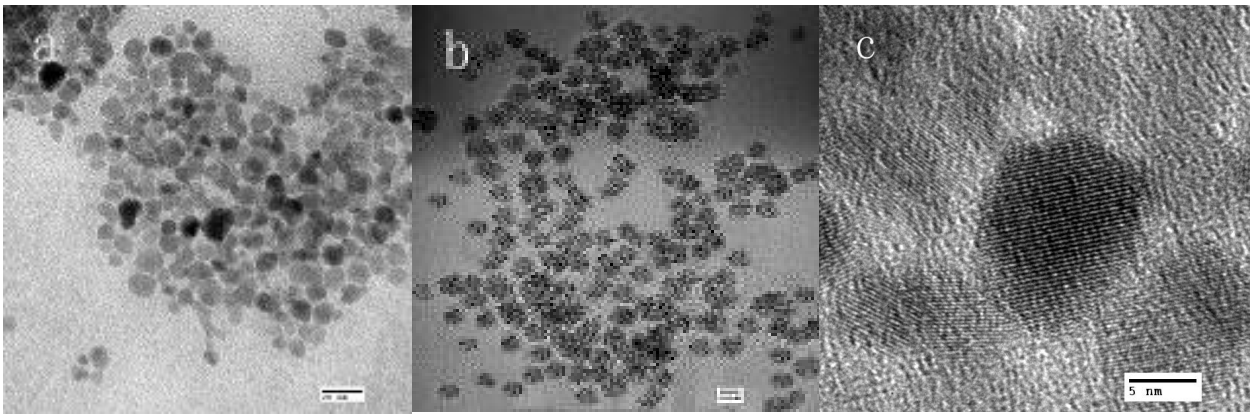



**Figure 2 –** a) A TEM image of magnetic beads ($\gamma$-$Fe_2O_3$) with polymer coating, the scale bar is 20 nm; b) A TEM image of QDs magnetic beads core-shell nanoparticles. The scale bar is 20 nm. c) A High resolution TEM image of a single magnetic bead coated with quantum dots. The scale bar is 5 nm.

An Energy Dispersed Spectrum (EDS) of the nanocomposite particles is in figure 3. The Cu peaks result from the sample grid. Spectral lines that originate from the QDs coating of the magnetic beads indicate the presence of Cd, Se, Zn, and S on the surface of the iron oxide particles. The relatively high Zn and S peaks could indicate the presence of multiple layers of ZnS on the surface of the CdSe QDs.

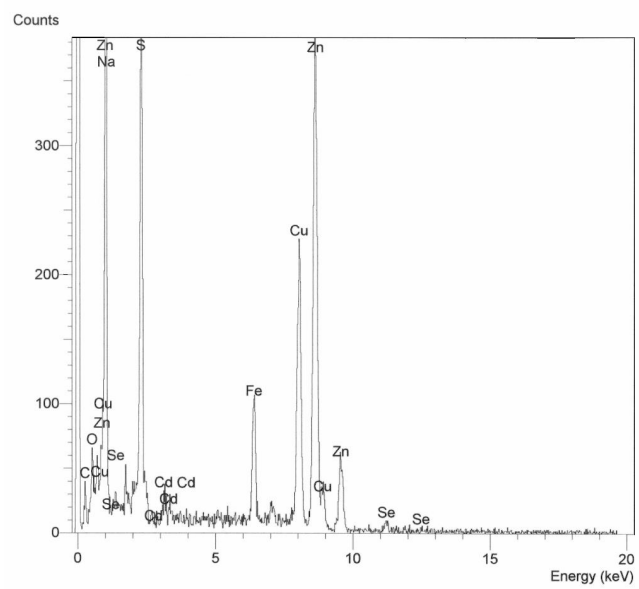



**Figure 3** – EDS (Energy Disperse Spectroscopy) spectrum of magnetic beads–QDs core-shell particles.

Luminescence images of the nanocomposite particles coated with ~3 and ~5 nm CdSe/ZnS QDs are shown in figures 4a and 4b respectively. A large signal to background ratio of over 100 is observed in these digital images. No micrometric clusters of nanocomposite particles are seen.

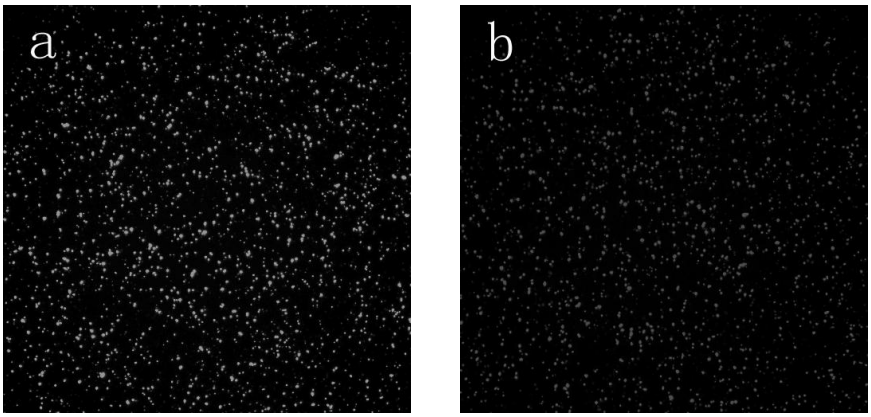



**Figure 4 –** Digital Fluorescence microscope images of magnetic beads coated with a) 3 nm and b) 5 nm CdSe/ZnS QDs

Luminescence spectra of CdSe/ZnS QDs in chloroform and CdSe/ZnS QDs- $\gamma$-$Fe_2O_3$ nanocomposite particles in aqueous solution are shown in figure 5. A slight blue shift is observed that could be attributed to a change in surface states of the QDs due to the immobilization. The emission quantum yield of the nanocomposite particles was found to be around 0.18, which is 3 times lower than the emission quantum yield of CdSe/ZnS QDs in chloroform (0.61). Luminescence lifetime of the luminescent/magnetic nanoparticles showed an excited state lifetime of 65±5 nsec. The excited state lifetime of CdSe/ZnS nanoparticles was 27±3 nsec. This excited state lifetime of the CdSe/ZnS QDs was in agreement with previous studies (21). It should be noted that the lifetime measurements yielded only approximated values since the fluorescence decay times exhibited significant variations from exponential decay curves. Nevertheless, the clear increase in excited state lifetime could be attributed to quenching interactions between the magnetic nanoparticles and the luminescent QDs or between the close packed QDs. The drop in the emission quantum yield could also be attributed to the solvent change and to possible changes in electronic density on the surface of the QDs due to their immobilization to the magnetic particles. While the ZnS capping passivates the surface of the CdSe QDs the capping is not perfect. It could allow electrons to leak to the surface of the QDs and to with the polymer coated magnetic core particle. Leakage of electrons from QDs was previously observed when the surface of CdSe/ZnS QDs was modified with mercaptoacetic acid (or MPA, MUA) (15). This resulted in a decrease in the emission quantum yield and a blue shift in the emission spectrum of the modified QDs as observed in our experiments. It should be noted however that the width of the emission peak of the QDs was not affected by the coupling of QDs to the polymer coated magnetic nanoparticles.

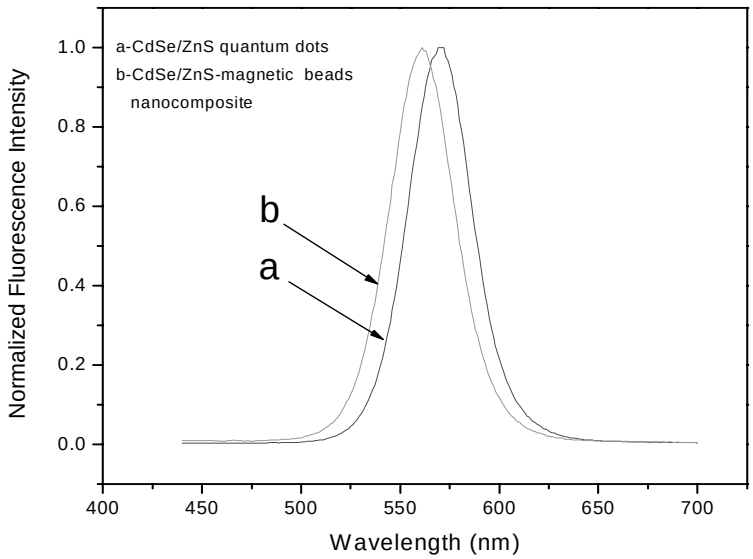



**Figure 5–** Fluorescence emission spectra. a (red): CdSe/ZnS QDs in chloroform; b (green): magnetic beads CdSe/ZnS quantum dots core-shell nanoparticles in water.

## 3. FABRICATION AND CHARACTERIZATION OF SEMICONDUCTOR – MAGNETIC NANOCOMPOSITE ENCAPSULATED INTO THE SILICA PARTICLES

### 3.1. Synthesis of Luminescent and Magnetic Silica Nanocomposite Particles using Microemulsions

In this part of our paper we describe a method to prepare silica composite particles that contain highly luminescent semiconductor CdSe-ZnS quantum dots and magnetite nanoparticles. Silica is a very versatile inorganic material, due to its inert chemical properties, optical transparency, and relative ease of functionalization with amino groups (–$NH_2$) and thiol groups (–SH). Successful incorporation of luminescent QDs into silica nanospheres has been reported. Chang et al (22) prepared silica nanospheres doped with CdS QDs using surfactant microemulsions. Kotov et al (23) used 3-mercaptopropyltrimethylsilane (MPTMS) derived CdTe QDs as seeds to synthesize silica nanospheres containing CdTe QDs. In our approach, reverse micelles were used as nanoreactors for the preparation of the QDs/magnetite-silica nanospheres. Highly luminescent water-soluble QDs capped with carboxylic acid were prepared following procedures previously reported (ref). For the microemulsion formulation, the following solutions were prepared separately: (i) 100 μL of 10% sodium silicate aqueous solution was added to a mixture of 50 μL iron oxide water soluble nanoparticles (2 mg/mL, AMRI) and 50 μL of 0.2 μM water soluble CdSe-ZnS QDs. (ii) 15 mL 0.1 M sodium bis (2-ethylhexyl) sulfosuccinate (AOT) heptane solution. Then, solution (i) was added to solution (ii) under sonication. Oleic acid (200 μL) was added and the solution was sonicated for further 30 min. The resulted nanospheres were collected by slow speed centrifugation at 2500 rpm for 10 minutes and washed 3 times with 10mL heptane. Sodium silicate was selected as the silica precursor instead of the commonly used tetraethyl orthosilicate (TEOS) because it was more miscible with the water-soluble magnetite and luminescent CdSe-ZnS QDs. Since magnetite and luminescent CdSe-ZnS QDs were previously mixed with the sodium silicate solution, the condensation of sodium silicate spontaneously trapped the magnetite and QDs nanoparticles in silica nanospheres. The oleic acid molecules served as condensation initiator and also as anionic co-surfactant with AOT to stabilize the reverse micelles. The use of sodium silicate also shortened the preparation time of the nanoparticles and the synthesis was completed in 60 minutes. The morphology of the resulted material was characterized by transmission electron microscopy (TEM) and scanning electron microscopy (SEM), as shown in figure 6a and 6b. The size distribution of composite particles was determined by size distribution of water dropet in water-oil microemulsion.

The silica nanospheres were between 0.5 and 5 μm in diameter. The large particles were removed from the sample by filtration.

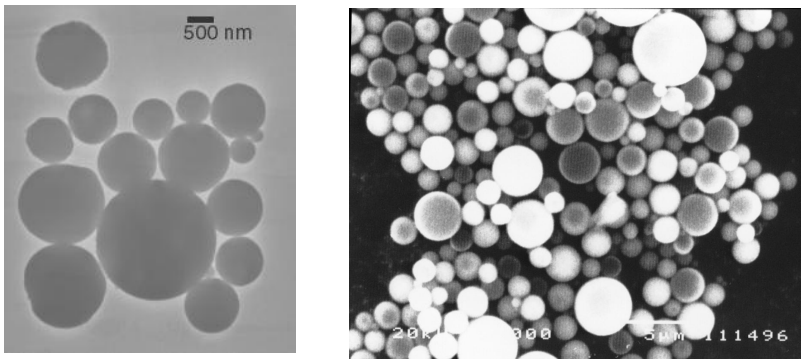



**Figure 6 – (a)** TEM image and **(b)** SEM image of. CdSe-ZnS quantum dots/magnetite-doped silica nanospheres.

The energy dispersive analysis (TEM-EDS) clearly showed the presence of Fe, Si, O, Zn, peaks. This confirmed the presence of CdSe-ZnS QDs and silica in the analyzed nanospheres (Figure 7).

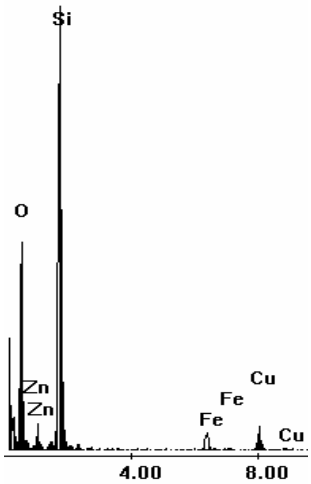



**Figure 7 -** The X-ray energy dispersive analysis (TEM-EDS) pattern of. CdSe-ZnS quantum dots/magnetite-doped silica nanospheres.

The emission properties of the QDs-doped silica nanospheres were similar to free CdSe-ZnS QDs in aqueous solution. They were highly luminescent since a large number of intense and crystalline QDs were encapsulated in each silica nanosphere. A digital fluorescence image of CdSe/ZnS luminescent quantum dots/ magnetite -doped silica nanospheres taken using a fluorescence-inverted microscope is seen in figure 8a. Each dot in the image does not represent the real size of the nanoparticles, but the light emitted for the fluorescent magnetic nanoparticles when excited at 470-490 nm. The image in Figure 8b shows the alignment of the particles under an applied magnetic field when a captative microscope-mounted magnetic yoke assembly (Molecular Probes) was placed over the sample on the inverted microscope stage. The imaging measurements show that the newly synthesized composite particles are both luminescent and magnetic. They could be detected optically while separated or manipulated using magnetic fields.

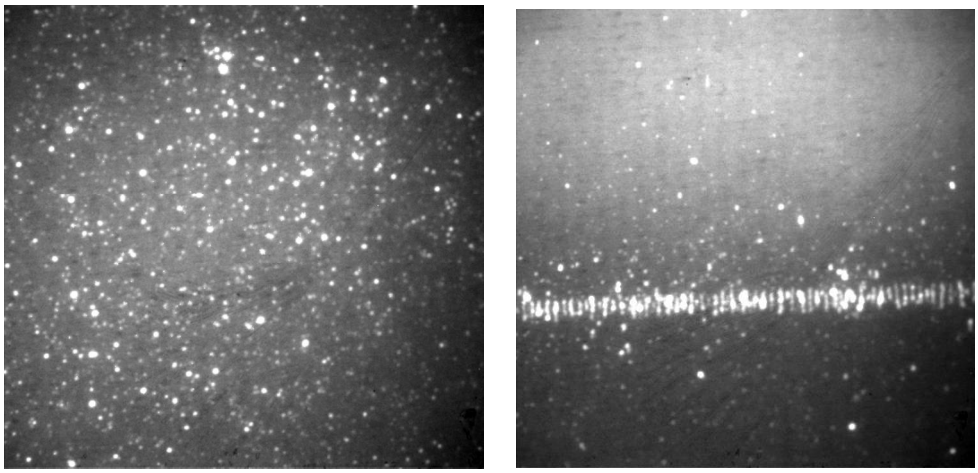


**Figure 8** – A digital luminescence image of CdSe/ZnS luminescent quantum dots/ magnetite -doped silica nanospheres in solution and oriented in a magnetic field. The image was taken through a 20X microscope objective at excitation wavelength of 460 nm.

### 3.2. Synthesis of Luminescent and Magnetic Silica Nanocomposite Particles using Co-precipitation

Another strategy of synthesizing silica nanocomposite particles that contain CdSe and $Fe_3O_4$ nanocrystals has been under development in our laboratory. CdSe quantum dots ranging between 2 and 5 nm in diameter were prepared following a method introduced by Kasuya and coworkers with slight modifications (4). Briefly, chelate complex Cd-NTA (Cadmium nitrilotriacete), obtained by dissolving 0.16 g of $CdSO_4$ *$5H_2O$ and 0.21 g of Na-NTA in 10 ml of water and 10 ml of methanol, was mixed with 0.8 ml decylamine as surfactant. The simultaneous addition of 17 ml of 1 M $Na_2SeSO_3$ with 25 ml of toluene was followed with an increase of the reaction temperature to $110^0$ C. Decylamine ligands that were bound to CdSe moved up into the toluene phase. Depending on the reaction time different size of dots were produced ranging between 2 and 5 nm in diameter. Further phase separation and centrifugation of the toluene suspension led to the formation of transparent, colored solutions which their color depended on dots size.

Commercially available ferrofluid (FF) of oleic acid stabilized $Fe_3O_4$ nanoparticles with an average diameter of 9±1 nm was used to prepare the luminescent and magnetic silica nanocomposite particles. A solution of 3 ml of CdSe quantum dots (3 nm in diameter) stabilized with decylamine in toluene was mixed with 1 ml of FF under ultrasonication for 15 min at room temperature. Then, 20 ml methanol was added to the particles mixture to wash the particles of excess of surfactant molecules and precipitate them. The suspension was left overnight at room temperature to maximize precipitation. The precipitate was with a phosphate buffer solution using repeated cycles of centrifugation at 5000 rpm for 15 min and re-suspension. Then, the washed precipitate was re-suspended in 10 ml ethanol solution containing 50 μl of aminopropyltrimethoxysilane (APTMS). Ultrasonication of this mixture led to the formation of partially transparent and colored (as original dots) solutions. The produced sample was characterized using photoluminescence spectroscopy, electron paramagnetic resonance spectroscopy, transmission electron microscopy and X-ray energy disperse spectroscopy.

The formation of silane bonds on the particles surface, which would facilitate further silanization is imperative for successfully preparing nanocomposite particles in which the silica matrix hosts the luminescent and magnetic nanocrystals. Taking into consideration the requirement of miscibility of the composite precursor in polar solvents for further silanization, the application of bifunctional organic molecules was highly desirable. In our case, quantum dots were stabilized with amino-alkane and the magnetite particles with oleic acid. In a recent report the possibility

of stabilizing magnetite particles with amino- group-containing organic ligands was demonstrated (24). Following this method we chose APTMS as a silanization precursor due to its proven ability to form dangling Si-O bonds on the surface of the particles. Figure 9 shows photoluminescence spectra of our nanoparticles: (a) CdSe-decylamine (as produced), (b) CdSe-APTMS, (c) CdSe-$Fe_3O_4$ – APTMS, and (d) CdSe-$Fe_3O_4$ – APTMS after UV irradiation. It is clearly seen, that replacement of decylamine with aminopropyltrimetothysilane led to a significant increase of the exciton band edge emission (QY of 10-20%) with slight red-shift of about 20-30 nm. While red shifts are often attributed to aggregation it appears that that aggregation does not occur since it would result in a decrease rather than in an increase in the emission quantum yield of the particles. It is possible that APTMS molecules better passivate the surface of the quantum dots and partially damp non-radiative paths that often originate from surface defects.

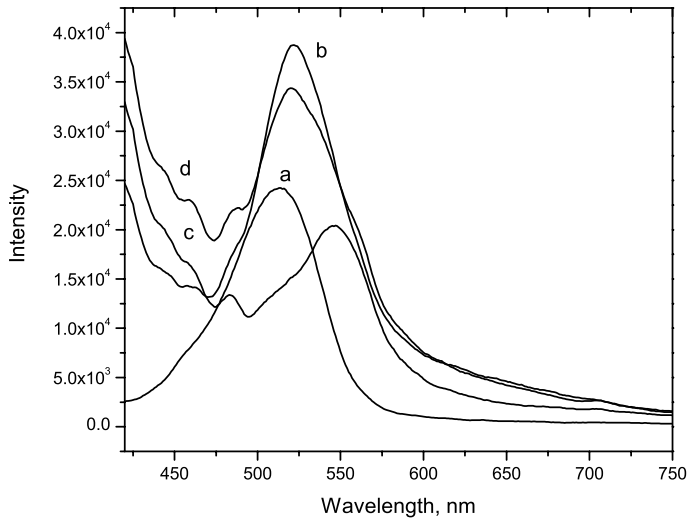



**Figure 9** – PL spectra of CdSe-decylamine (as produced) (a), CdSe-APTMS (b), CdSe-$Fe_3O_4$ – APTMS (c), CdSe-$Fe_3O_4$ – APTMS after UV irradiation (d)

Coupling of the luminescent quantum dots with magnetite particles resulted in the reduction of intensity of exciton band edge by about 3 folds, which was in agreement with our previous studies (13). However, irradiation of the nanocomposite particles using the 325 nm line of an He:Cd laser for 20 minutes restored the photoluminescence intensity to about the initial emission intensity of APTMS stabilized CdSe quantum dots.

EPR spectroscopy was used to investigate the effect of coupling the luminescent semiconductor nanoparticles to the magnetic particles. Figure 10 shows EPR spectra of (a) $Fe_3O_4$ – oleic acid , (b) $Fe_3O_4$ – APTMS, and (c) CdSe-$Fe_3O_4$ –APTMS suspensions

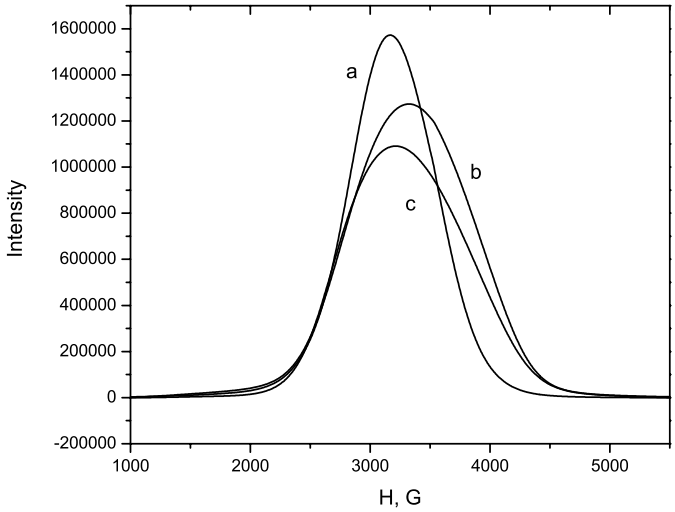



**Figure 10** – EPR spectra of (a) $Fe_3O_4$ – oleic acid, (b) $Fe_3O_4$ – APTMS, and (c) CdSe-$Fe_3O_4$ –APTMS suspensions

It is clearly seen that replacement of stabilizing ligand molecules with the semiconductor quantum dots affected the peak position of the resonance field and its half-width. This is attributed to a change in the environment of the magnetite nanoparticles. It is important to note that the resonance peak position of the CdSe-$Fe_3O_4$ – APTMS was similar to the peak position of the original ferrofluid. This was indicative of phase separation of quantum dots and magnetite particles within the silica nanocomposite particles. Figure 11 shows a TEM image of an isolated CdSe-$Fe_3O_4$ – APTMS composite particle of about 50 nm in diameter. It can be seen that the magnetite particles form dark aggregates which were surrounded by CdSe quantum dots. EDS data of the nanocomposite particle showed the following composition: Si – 31.4% (at.), Cd – 14.6% (at.), Se – 8.8% (at.), Fe – 45.3%.

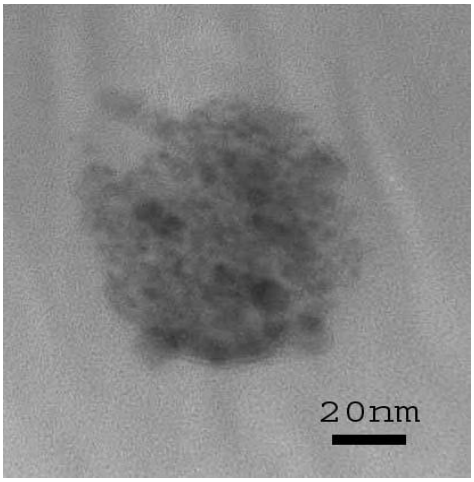



**Figure 11** – TEM image of CdSe – $Fe_3O_4$ – APTMS nanocomposite particle

## 4. SUMMARY AND CONCLUSIONS

Several approaches to synthesize semiconductor – magnetic nanocomposite were developed. Depending on original scheme of quantum dots preparation, different strategies of coupling quantum dots to magnetic particles were used. The presence of dimercapto-succinimid acid on the surface of magnetic particles surface enabled coupling of magnetite to (CdSe)ZnS core-shell nanoparticles using well documented thiol chemistry. The formed nanocomposite particles demonstrated all the advantages of both magnetic and semiconductor particles. The necessity for further functionalization of the nanocomposite particles to facilitate biomedical applications led to the formation of silica nanocomposite particles that contain luminescent semiconductor quantum dots and iron oxide magnetic nanoparticles. Two silanization strategies were used for this purpose. The first was a microemulsion approach in which physical confinement of water droplet served a crucial role in the coupling and silanization processes. The second approach involved the co-precipitation of the magnetic and luminescent nanocrystals in silica particles using bifuntional cross linkers and amino-silane chemistry. Amino-groups of APTMS replaced decylamine and oleic acid as capping ligands of the CdSe and $Fe_3O_4$ nanoparticles correspondingly. The dangling Si-O bonds polymerized and created a silane network which resulted in the formation of silica based aggregates, where quantum dots and magnetic particles were trapped in. The composition of nanocomposite was well controlled. The successful formation of silane bond precursor on the particles surfaces was achieved. Future studies will focus on further improving the physical properties of the composite nanoparticles including monodispersity, size distribution and shape. This will be accomplished by investigating the effect of replacing the currently used silica precursors with organically modified silicates on the physical properties of the nanocomposte particles and by a tighter control of reaction conditions.

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