Detection and mitigation of radionuclides in the environment: Toward a clean ecosystem

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Abstract

This research describes a straightforward approach to produce surface engineered nanomaterials for detection and mitigation of radionuclides in nuclear facilities. Specifically, pertechnetate (TcO4-) contaminants capture from aqueous environments was achieved by surface functionalization of iron oxide nanoparticles with a micelle directing surfactant, namely cetyltrimethylammonium bromide (CTAB). CTAB has a polar trimethylammonium group at one of its ends making it soluble in aqueous systems. The hydrophobic 16-carbon cetyl tail component forms an intertwining 'zipper-type" bilayer on the nanoparticle's surface. This unique micelle feature of the CTAB exposes the cationic head groups to the environment imparting a positive surface charge of the nanoparticles. A series of analytical tools were employed to characterize the nanocomposite materials and probe their ability to capture ReO4- as a model analyte, such as SEM, EDS, UV-Vis spectroscopy, DLS, and PALS. The technology can be used to remotely remediate environments by scavenging and removing radionuclide species from water sources at desired location.

Keywords: nanomaterials, Technetium-99 (Tc), iron oxide, cetyltrimethylammonium bromide (CTAB), decontamination

1. Introduction:

Radionuclides pose a serious problem to our ecosystem. Their toxicity, long half-life, and high energy discharged[1],[2] are difficult to manage. Therefore, a major effort is aimed at reducing the human and environmental risk profile through treatment, processing, and disposal of million gallons of radioactive materials.[3] Radiation detection strategies have also become increasingly important when handling radionuclides. For example, surface-enhanced Raman spectroscopy (SERS) was reported for detection of numerous analytes, including pertechnetate (TcO4-) and its speciation moieties at $\sim 10^{-7}$ M concentration levels with gold nanoparticles.[4],[5]

Nuclear materials can be found in either gaseous, liquid, or solid forms generating radioactivity of any types, such as alpha, beta, gamma rays, or X-rays.[6] As a result, developing appropriate technologies for efficient capture and handling of radionuclide requires innovative strategies. An assortment of testing protocols and materials have been developed and implemented toward identification and quantification of radioactive compounds present in nuclear waste storage facilities. Traditional approaches of capturing radionuclides rely on physical and chemical approaches that either reduces waste volume or recover valuable components. A number of adsorbents, absorbents, and ion exchange materials have been explored.[6,7,8] For example, radioactive cesium (I) ions were captured from aqueous solution through ion chelation with calixarene-crown-6 derivatives with terminal carboxyl groups attached to nano-sized magneto-ferritin molecules.[9] Gold nanoparticles treated filters were used to capture and retain radioactive 65Zn vapors generated in the tritium facilities.[1] Zero valent iron is an effective remediation agent for decontamination purposes generated in nuclear facilities.[10] We also reported earlier a waveguide for use with surface-enhanced Raman spectroscopy for enhanced detection of gas analytes, such as hydrogen isotopes.[11]

Technetium-99 (Tc), a fission product of uranium-238 and a beta emitter with a maximum energy of 292 keV, is a major contaminant at nuclear power plants. Tc has been unintentionally introduced into the environment from anthropogenic and indigenous sources, such as nuclear power plants, global weapons, nuclear storage facilities and medical applications.[12] As environmental remediation and nuclear waste treatment issues escalate, the removal or sequestration of Tc species from contaminated waters is of great concern. Most of Tc's isotopes (97Tc and 98Tc) are unstable except for 99Tc, which has a half-life of 2.11 x 10^5 years. Its most common aqueous species, pertechnetate (TcO_4^-), is very itinerant in the environment making difficult to detect and sequester. Due to its high solubility and mobility, pertechnetate can enter the food chain and cause adverse health effects to humans and environment. For example, the US Environmental Protection Agency has established its maximum contamination level of 4 millirems per year or 33.3 mBq/m^3.[13]

The majority of nuclear operations use sorption technology to reduce waste volume or to recover key valuable elements or isotopes. Ion exchange resins and reduction processes are currently the most common approaches for Tc immobilization. Although these techniques have shown to be operational, they possess major drawbacks, such as high cost, low adsorption capacity, and not specific to species of interest.[14] On the other hand, decontamination processes that relies on reduction procedures have shown to be more effective for Tc immobilization through the use of bacteria or reduction agents.[15] Reduction processes, however, possess major drawbacks as they are operationally complex making them expensive to implement and maintain. These technologies are mostly hindered by the relatively high solubility of the precipitated reduction product, Tc(IV)O2·1.6H2O in groundwater, i.e. 1.5×10^{-8} M. This greatly exceeds the EPA's maximum contaminant level. Therefore, development of efficient, simple, and deployable technologies for the detection and remediation of Tc contaminated systems are needed.

Iron oxide magnetic nanoparticles are of high interest among numerous classes of nanomaterials for contaminants removal as, in addition to having a high surface area to volume ratio, fast kinetics, strong adsorption capacities and high reactivity, they can be easily manipulated and collected with a magnet due to their magnetic properties.[16] For instance, when an external magnetic field is applied remotely at a certain distance, these nanomaterials rapidly aggregate, serving as a straightforward and cost-effective separation process to extract them from aqueous solutions.[17] We investigated earlier a porous iron material for TcO4- and perrhenate (ReO4-) sequestration from artificial groundwater. Porous iron material was significantly more effective than granular iron for both TcO4- and ReO4- sequestration under oxic conditions. The Tc removal capacity was 27.5 mg Tc/g pFe at pH ~6.8, while the Re removal capacity was 11.2 mg Re/g pFe at pH ~10.6.[15] Magnetic mesoporous silica nanoparticles were also used for the removal of toxic metal anions from water by combining the magnetic properties of iron oxide with the high adsorption capacity of mesoporous silica.[18] Although magnetic nanoparticles have been used for the removal of contaminants from aqueous media one of their limitations is their ability to aggregate due to their magnetic properties. Therefore, the stabilization of the magnetic nanoparticles by surface modification is desirable to improve their manipulation, separation and stability. Cetyltrimethylammonium bromide (CTAB), a micelle forming surfactant, was previously explored for the removal of organic and inorganic contaminants from aquatic environments or as a partitioning membrane of naphthol.[19],[20],[21] However, to date, the adsorption of technetium from groundwater using CTAB coated Fe_2O_3 has never been explored.

This study seeks to develop surface functionalized magnetic iron oxide nanoparticles with a stabilizing agent, namely cetyltrimethylammonium bromide (CTAB), and tested for the efficient removal of Tc from water resources under ambient conditions.

2. Experimental Section:

2.1. Materials. All chemicals were used as received. Iron oxide (Fe2O3) spheres, hexadecyltrimethylammonium bromide (CTAB) and sodium perrhenate (ReO4-) were purchased from Sigma Aldrich (St. Louis, MO). Aqua regia (HNO_3 & HCl) solution was used to clean all glassware. Deionized water (18.2 MΩ·cm) was obtained from an Aqua Solutions high purity filtration system (Jasper, GA) fitted with a AMTROL reverse osmosis filter (West Warwick, RI).

2.2. Surface engineering of iron oxide nanoparticles.

0.1 mL of NaOH (0.1 M) solution is added to a falcon tube containing 1 mL Fe2O3 (0.04 g/10 mL) nanoparticles solution. Then, 9 mL of CTAB (0.1 M) solution is added to the previously prepared suspension. The mixture was left to reach equilibrium for 30 minutes. The formed nanoparticles were washed 5 times with 5 mL of deionized water (DI), collected by applying an external magnetic field and dispersed in 10 mL of deionized water.

2.3. Adsorption Studies

Perrhenate (ReO4-) was used as a model contaminant due to its similar physiochemical properties (e.g., oxidation states, ionic radius and energy of hydration) to those of pertechnetate (TcO4-). Cetyltrimethylammonium bromide was used to modify the magnetic nanoparticles surface and subsequently for the adsorption of ReO_4^-. Sorption studies were performed using deionized water as matrix, at room temperature, with constant stirring (8 rpm), and using 4.0 mg of adsorbent for 10 ml of aqueous 40 mM NaReO4 solution. The solution pH was adjusted between 5-6 for all the sorption studies.

2.3. Nanoparticle's characterization

A series of analytical techniques were used to characterize the physical and chemical properties of the nanomaterials. UV-visible absorbance (UV-vis) spectra were obtained for nanoscale material samples on a tec5 MCS PDA with LS spectrometer (Oberursel, GER) at a wavelength range of

300-1000 nm. Scanning electron microscopy (SEM) and energy dispersive X-ray analyses (EDX) were obtained using a Hitachi SU8230 scanning electron microscope (Tokyo, JPN). Colloidal SEM samples were prepared by depositing 2 μL of the washed colloid on a carbon grid. SEM images were used for sizing and morphology determination for nanoscale materials with an operating Vacc and Ie of 10.0 kV & 7 μA respectively at a 5 mm distance. Sample compositional data was obtained by energy dispersive x-ray spectra using Oxford Aztec software (Abingdon, UK) with SEM collection parameters of Vacc = 15.0 kV, Ie = 7 μA, distance = 15 mm.

3. Result and Discussions:

Nanomaterials have emerged as suitable platforms to solve complex technological problems in many fields including medical, catalysis, opto-electronic, imaging arenas, and nuclear fields, including tracking nuclear species in the environment, nuclear screening, and nuclear decontamination.[22] This is because nanomaterials with dimensions between 1-100 nm display novel properties that differ from both their bulk and molecular counterparts. As a result, nanoparticles size, shape, and surface chemistry have a profound effect on their properties. Moreover, the large fraction of atoms on the particle surface results in an increased surface area available for reactions leading to an enhanced reactivity. These characteristics promotes faster reaction kinetics and higher remediation capacities.

3.1. Characterization of bare iron oxide (Fe2O3) nanoparticles:

Iron (III) oxide nanoparticles, Fe_2O_3, are an excellent candidate for use in environmental stewardship applications as they are inexpensive, non-toxic, and broadly accessible. An added benefit of these nanoparticles are their magnetic properties. Given that the maghemite phase, γ-Fe_2O_3, is ferrimagnetic, and the hematite phase, α-Fe2O3, is weakly ferromagnetic, these nanostructures can be easily manipulated with external magnetic fields, i.e. magnet.

Scanning electron microscopy (SEM) was used to investigate the morphology of the magnetic iron (III) oxide nanoparticles. The SEM studies reveal that bare Fe_2O_3 nanoparticles are spherical in shape with smooth and distinct geometry. The outer diameter of these nanostructures is approximatively 46 ± 5 nm. The UV−visible spectroscopy (UV−vis) analysis was collected in aqueous solution, and a distinctive absorption peak was recorded at 353nm. The spectrum is consistent with the behavior of dispersed iron oxide nanoparticles.[23]

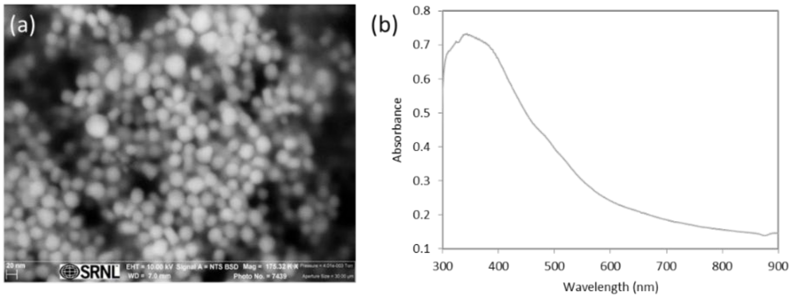

Figure 1. (a) Scanning electron microscopy (SEM) and UV-Vis spectra of iron oxide nanoparticles.

3.2. Nanoparticle's surface engineering:

Functional and bifunctional moieties can be used to generate rational assemblies of nano-architectures at the nanoparticle's surface. Surface bound surfactants, ligands and linkers can transform nanoparticle's charge, functionality, stability, and reactivity. For example, it can increase/decrease its chemical stability, passivation, and/or serve as a membrane-type of network available for selective manipulation and capture of analytes. Through partitioning or confining of small molecules at the solid-liquid interface one could generate effective molecular-scale nanoparticle platforms for sensing and catalytic reactions.[24],[25]

We developed here a straightforward approach for engineering the surface of iron oxide nanoparticles with cetyltrimethylammonium bromide (CTAB) for pertechnetate (TcO4-) contaminant's capture and removal from aqueous environments. CTAB is often used in the preparation of anisotropic metallic nanoparticle, i.e. rods, cubes, triangles, due to its ability to serve as a structure-directing agent leading to exquisite control over nanostructure's shape and aspect ratio.[22,26] Previous studies have shown that CTAB serves as a good stabilizing agent to prevent nanoparticle's agglomeration. Its ability to easily bind to different moieties and surfaces promotes generation of nanoparticles with increased dispersibility and stability.[27] CTAB is an fascinating surfactant as it presents both: a polar trimethylammonium group at one of its end making soluble to aqueous systems and a 16-carbon cetyl tail that has a hydrophobic character. The unique intertwining assembling feature of the CTAB exposes the cationic head groups to the environment which imparts a positive surface charge of the nanoparticles. Additionally, the hydrophobic tail of CTAB interdigitates creating a "zipper-type" bilayer on the nanoparticle's surface. The hydrophobic layer can be further manipulated for reactions by partitioning of water-insoluble moieties from the aqueous environments. [21]

In order to confirm the surface functionalization of iron oxide nanoparticles, we monitored their surface charge before and after the surface engineering experiments. We collected the zeta potential measurements with phase analysis light-scattering system. The effective surface charge of the Fe_2O_3 nanoparticles in water at neutral pH before incubation in CTAB was negative with a value of −11 mV. The negative surface charge is attributed to the electric potential formed at the solid support-liquid interface due to the presence of hydroxide ions adsorbed on the surface, specifically the deprotonated −Fe-OH species.[28] The positively charged CTAB can interact with the negatively charged surface of the Fe_2O_3 in an alkaline pH through an electrostatic interaction, thus engineering the surface of the nanoparticles and preventing aggregation. Once incubated in CTAB, the Fe_2O_3 nanoparticles' surface charge significantly changes, becoming highly positive (+19 mV) and stabilizing of the magnetite nanoparticles.

The nanoparticle's surface charge, however, varies with the solution's pH. Therefore, zeta potential measurements of bare Fe_2O_3 and CTAB-engineered Fe_2O_3 were measured in a pH range between 4 to 10 (Figure 2). The zeta potentials fluctuate from positive to negative as the pH changed from acidic to basic. Figure 2 showed that the point of zero charge (PZC), which describes the pH value at which the surface density of positive charges (cations) equals that of negative charges (anions) of bare Fe_2O_3 was around 6.2, which was similar as those acquired for bare Fe_3O_4 (pHpzc 6.1).[19] On the other hand, the pHpzc of CTAB-engineered Fe_2O_3 was around 7.2. The increase in the pHpzc might be due to the presence of the cationic CTAB bound to the Fe_2O_3 surface, which suggest the successful functionalization of the Fe_2O_3 with CTAB.

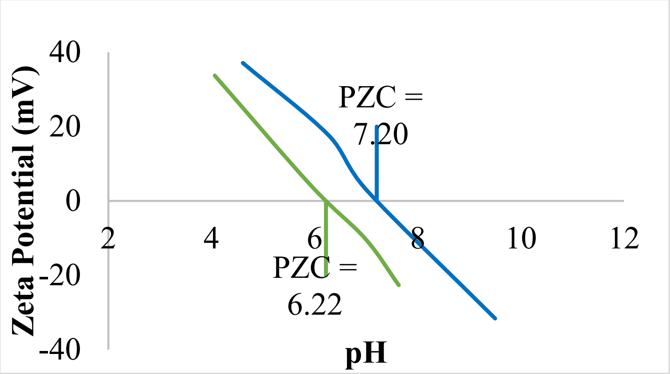

Figure 2. Zeta potentials of bare-Fe2O3s (green) and CTAB-engineered Fe2O3 (blue) at different pH. The point of zero charge of each material is also shown.

SEM images of the CTAB-engineered Fe_2O_3 were also recorded. As expected, it was observed that the CTAB-functionalized Fe_2O_3 retain their spherical structure and the diameter remain constant to bare nanoparticles.

Dynamic light scattering was also used to determine the hydrodynamic radius of the nanoparticles in aqueous solution (**Table 1).** The hydrodynamic diameter decreases when the surface of the nanoparticles is functionalized with CTAB. This is due to repulsive and steric forces between the CTAB molecules in agreement with previous studies. These forces help stabilize the bare nanoparticles by preventing their aggregation. [29]

Table 1. Dynamic Light Scattering Analysis on bare-Fe_2O_3 and CTAB-Fe_2O_3 nanoparticles.

DLS Analysis	
Material	Hydrodynamic Diameter (µm)
Bare-Fe_2O_3	2.4
CTAB-Fe_2O_3	0.38

3.3. Sorption Studies:

ReO4- has similar aqueous chemistry properties to those of TcO4-, such as oxidation states, ionic radius and energy of hydration, and was used as a non-radioactive chemical analog for TcO4- in our studies. To demonstrate the perrhenate sorption capabilities of the CTAB functionalized iron oxide nanoparticles, the material was exposed to a NaReO4 solution. After 24 hour of incubation time, the iron oxide nanoparticles were washed and separated from the analyte solution and compositional analysis was performed. The Energy dispersive X-ray (EDS) analysis shows that, before exposure to the analyte, the only elements present in the material were iron and oxygen, from the iron oxide crystalline structure, and carbon from the aliphatic chain of the surfactant (Figure 3). After being exposed to the analyte solution the EDS analysis shows the presence of rhenium in the material. This indicates the adsorption of Re by the CTAB-functionalized nanoparticles. An in-depth analysis of the EDS results showed that the Re to Fe mass ratio is around 0.68 ± 0.06, which may demonstrate a high adsorption capacity of the material.

Taken together, the zeta potential and the sorption data suggest that the Re in aqueous solution at pH between 5-6 is adsorbed as a negatively charged rhenium oxide species (ReO4-).

The adsorption occurs between the negatively charged Re and the positively charged amine functional groups of the CTAB-functionalized iron oxide. A schematic illustration of the proposed adsorption mechanism of Re onto the CTAB-functionalized Fe_2O_3 nanoparticles shown in Figure 4. A similar adsorption mechanism was reported by others when using CTAB modified magnetic nanoparticles for other contaminants.[18,19, 30]

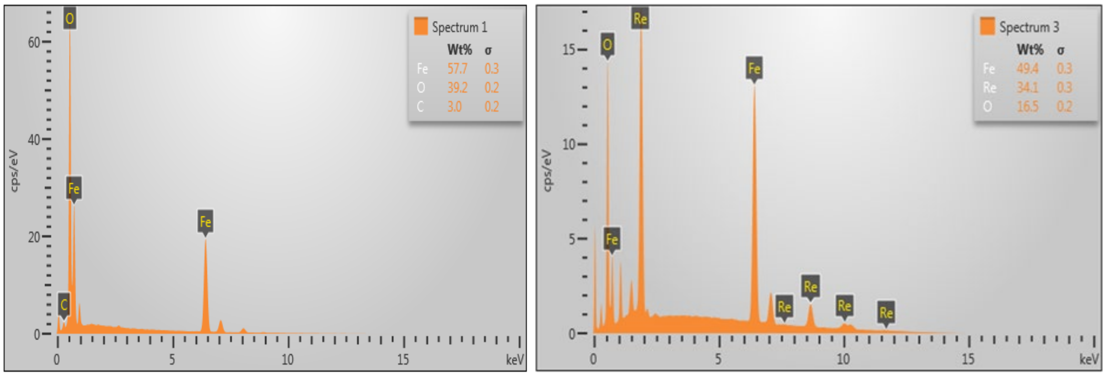

Figure 3: EDS analysis of CTAB-Fe_2O_3 nanoparticles before (left) and after (right) perrhenate adsorption. Re/Fe mass ratio after adsorption study.

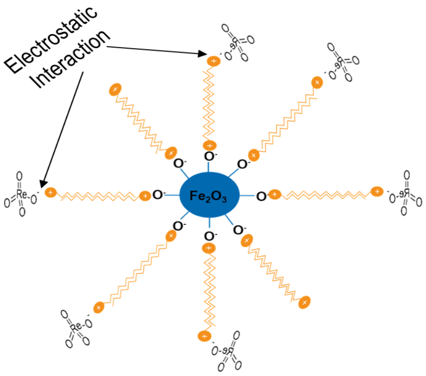

Figure 4: Interaction mechanism between ReO_4^- molecule and CTAB functional groups.

4. Conclusions:

Magnetic iron oxide nanoparticles were successfully functionalized with a micelle directing surfactant, namely cetyltrimethylammonium bromide (CTAB). The functionalization of the iron oxide nanoparticles affects their surface charge and their hydrodynamic diameter. The addition of CTAB decreased the hydrodynamic diameter of the nanoparticles due to repulsive and steric forces. Scoping experiments shows the successful adsorption of perrhenate ions (ReO4-) onto CTAB-functionalized Fe_2O_3 nanoparticles.

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