



Elaboration of magneto-thermally recyclable nanosorbents for remote removal of toluene in contaminated water using magnetic hyperthermia



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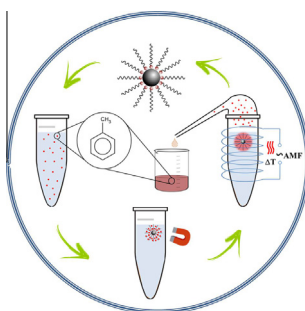
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HIGHLIGHTS

- Magnetic nanoadsorbents with high affinity for toluene (BTEX).
- Nanoadsorbents are magnetically separable and reusable.
- Remote recycling of nanosorbent by alternating magnetic field.
- Early pioneer in use of magnetic hyperthermia for environmental remediation.

GRAPHICAL ABSTRACT



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ABSTRACT

Magnetic nanosorbents are novel materials that have been impacting various applications in environmental fields, including the detection of pollutants and remediation of polluted environments. In this work, the thermal heating effect caused by the interaction of nanoparticles' magnetic moments with an alternating magnetic field (AMF) – magnetic hyperthermia – is proposed as proof of a concept to illustrate the remote controlled recycling of magnetic nanosorbents during the treatment of water containing toluene, a potentially toxic and volatile contaminant. Nanosorbents were synthesized by functionalization of ~15 nm coprecipitated cobalt/manganese mixed ferrite with polydimethylsiloxane. Structural, chemical, and magnetic characteristics of the nanosized adsorbent were investigated by elemental analysis (ICP-OES), X-ray diffractometry (XRD), high resolution transmission electron microscopy (HRTEM), Fourier transform infrared spectroscopy (FTIR) and vibrating sample magnetometry (VSM). Batch experiments indicated that nanoadsorbents, which can be magnetically recovered, exhibit high adsorption affinity for toluene in aqueous solution (~325 mg/g) and that adsorption follows the Langmuir model. Moreover, application of AMF resulted in uniform heating within the nanosorbents (after being magnetically separated from the contaminated water) and triggered toluene desorption/evaporation, leading to accelerated release of pollutants from the nanosorbent, improving its reusability; even after seven cycles the removal efficiency was quite high (83%).

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1. Introduction

More effective and low-cost treatment options are being pursued to remediate water contaminated by hazardous organic compounds, such as benzene, toluene, ethylbenzene, and xylenes (BTEX), commonly present in large amounts in industrial regions or originating from leakages of storage tanks and fuel pipelines [1]. The World Health Organization (WHO) guidelines state that the maximum acceptable concentrations of benzene, toluene, ethylbenzene and xylene in drinking water are 0.01, 0.7, 0.3 and 0.5 mg/L, respectively. Thus, the treatment of wastewater containing BTEX is a key issue [2]. It has become clear that physical separation accompanied by forced evaporation (vapor extraction and multi-phase extraction) can remove a large quantity of BTEX from spills [3]. Chemical extraction using dispersants, oxidation and photo-catalysis has been also reported for these purposes [4]. Also, treatments based on assisted bioremediation processes are very promising, since they cause less harm to the contaminated ecosystem than the conventional chemical and physical methods [5]. Unfortunately, most of these cited remediation methods demand a long operation time and require expensive apparatus and/or the introduction of non-recyclable (toxic) chemicals. However, adsorption on solid supports is an alternative because it presents a lower initial cost, simplicity of design, ease of operation, low quantity of residues generated, significant recovery of pollutant and the possibility of reusing the adsorbent [6]. In particular, due to their high surface area and capability of modifying the surface, adsorbents are more effective when the size of solid supports is reduced to nanometric scale [7]. The high dispersibility and difficult recovery of these nanosorbents – known to be constraints in remediation techniques – can be minimized by associating a responsive material, such as a magnet, to the adsorbent to form a magnetic sorbent that can be remotely controlled by an external magnetic field, improving its carrying, separation and recovery in remediation technologies [8]. Indeed, the ability to recover nanosorbents represents an improvement in the systematic use of nanoscale materials in the field of environmental remediation, since dispersed nanoparticles have been also considered to be emerging contaminants [9].

Elaboration of these magnetic nanosorbents normally involves coating/dispersing magnetic nanoparticles with/into adsorbent materials which present selective compatibility with the contaminant [10]. In the case of BTEX, and particularly toluene, which was investigated as the adsorbate in this work, the adsorbing substrate must present hydrophobic chains capable of interacting with the hydrocarbon contaminant. Thus, polydimethylsiloxane has been chosen to functionalize our magnetic nanoparticles – this is a commonly used adsorbent that, in addition to being easy to prepare and inexpensive, easily adsorbs/desorbs BTEX molecules [11] and, moreover, presents high biocompatibility [12]. In the literature, there are several examples of magnetic nanoparticles functionalized with polydimethylsiloxane, but with applications that are not directed toward environmental remediation [13–15].

Commonly, after pollutant removal, a magnetic sorbent can be magnetically recovered from the contaminated sample and reused in new cycles of decontamination. For recycling, desorption of a contaminant from the solid support can be achieved by changing the physical and chemical conditions of a solution containing nanosorbents, such as pH [16] and temperature [17], or by solvent extraction [18].

To the best of our knowledge, this is the first report describing the use of the thermal heating effect caused by the interaction of the nanoparticles' magnetic moments with an alternating magnetic field (AMF) known as magnetic hyperthermia [19] and widely used in biomedical applications, such as cancer treatment and

remote controlled drug release. The results shown here serve as proof of a concept to illustrate the remote controlled recycling of magnetic nanosorbents in the remediation of water containing a volatile contaminant.

In summary, magnetic nanoparticles (MNPs) of a mixed ferrite were functionalized with polydimethylsiloxane and introduced in a sample of water contaminated with toluene. After adsorption of the pollutant, an *in situ* magnetic hyperthermia procedure was employed, which increased the temperature of the magnetic nanosorbents. The local heating induced desorption/evaporation of the volatile contaminant, and as a consequence the release of the pollutant. The magnetic nanosorbents were then available for re-use in new decontamination cycles by magnetic recovering.

2. Materials and methods

2.1. Magnetic nanosorbent elaboration

All chemical reagents were of high purity grade and thus used without any further purification. The synthesis procedure for $\text{Co}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles was adapted from previous works [20–21], as follows: adequate aliquots of 1.0 mol/L metal stock solutions were diluted with distilled water to form a 60 mL precursor solution containing 10.0 mmol of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 2.5 mmol of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 2.5 mmol of $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$. Then, 400 mL of a 2 mol/L NaOH solution was quickly poured into the stock solution under vigorous stirring at 100 °C for 2 h. After cooling to room temperature, the precipitate was separated by magnetic decantation, and washed three times with H_2O and once with 1.0 mol/L HNO_3 solution. To ensure the thermodynamic stability of the particles, the precipitate was boiled with a 0.5 mol/L ferric nitrate solution. Finally, after washing the precipitate three times with acetone, the particles were conveniently peptized in 10.0 mL of water, resulting in a stable solution.

The carboxyl-terminated polydimethylsiloxane (PDMS-COOH), used for particle surface modification, purchased from Polymer Source Inc. (Canada), presents molecular weight $\sim 10^4$ g/mol and polydispersity index of 1.09, as provided by the manufacturer. A ligand exchange reaction was used to complex the PDMS-COOH ligand on MNPs. Specifically, the pH of MNP suspension was adjusted to approximately 4, and 500 mg of PDMS-COOH were dissolved in 10 mL of hexane. These two solutions were mixed, sonicated for 2 h and moved to an overhead shaker with vertical rotation for 12 h. After this process, MNPs were transferred from the aqueous to the organic phase. The precipitate was then washed with ethanol and dried under vacuum.

2.2. Characterization

The chemical determination of metals was carried out by slurry nebulization using an Optima 8000 inductively coupled plasma optical emission spectrometer (ICP-OES) from Perkin Elmer [22]. The size and morphology of the materials were examined by high-resolution transmission electron microscopy (HRTEM) using a JEOL 1100 apparatus. X-ray diffraction analysis was performed on powder samples using a Rigaku – Miniflex 600 diffractometer with radiation of 1.541 Å (40 kV and 30 mA – $\text{CuK}\alpha$). The room-temperature magnetization curves were obtained using an ADE vibrating sample magnetometer model EV7. Hysteresis loops were performed under applied magnetic fields varying from –18 to 18 kOe at 300 K. FTIR spectra were recorded with crystalline KBr in the range of 3700–400 cm^{-1} and resolution of 2 cm^{-1} , on a Shimadzu IR-Prestige 21 system.

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