Chapter 9
A Perspective on Environmental and Disposal Assessment of Magnetic Sorbents

Nishesh Kumar Gupta, Herlys Viltres, and Carolina Leyva

Abstract While adsorption is an age-old technique for the treatment of contaminated water, the involvement of magnetic phase separation is relatively new. Magnetism in adsorbents (or magnetic adsorbents) has eased the recovery of micro/nano-adsorbents post-adsorption. Magnetic adsorbents could lower the operation time/cost and improve the overall efficacy of the adsorption process. In this book, most of the chapters have highlighted the use of magnetic adsorbents in water remediation applications. This chapter is focused on the regeneration strategies and disposal assessment of magnetic adsorbents and eluents post-exhaustion. Moreover, the chapter has been updated with some accounts of repurposing strategies for exhausted magnetic adsorbents.

Keywords Green strategies · Magnetic adsorbents · Regeneration · Repurposing

9.1 Introduction

In general, adsorbents are judged based on their robustness and efficacy for pollutant uptake [1, 32]. Apart from the efficacy, ease of recyclability and the extent to which an adsorbent could be recycled are equally important, which are often ignored

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in the published works [26]. In wastewater treatment facilities, the cost of sludge processing and disposal is significantly higher than the cost of the adsorbent. Thus, the commercialization of magnetic adsorbents for wastewater treatment relies on the regeneration process and management of fully used adsorbents. Another feature that the studies lack is the environmental significance of the magnetic adsorbents-based treatment processes. The use of toxic chemicals or high energy input in the regeneration process needs to be criticized in the interest of achieving sustainability. At the same time, the disposal of toxic spent adsorbents in the landfills should be strictly monitored and guided by environmental norms. Reuse and disposal are two different aspects of waste management. Though regeneration of adsorbents is always an energy-intensive process, the choice of the regeneration process and the economic value of pollutants may balance the equation. Disposal is the process of discarding spent adsorbents either by burning in incinerations (applicable for organic adsorbent-pollutant systems) or in landfills. Since magnetic adsorbents cannot be incinerated, a landfill is considered the only viable option. In the subsequent sections, we have discussed different approaches for regeneration and disposal (Fig. 9.1).

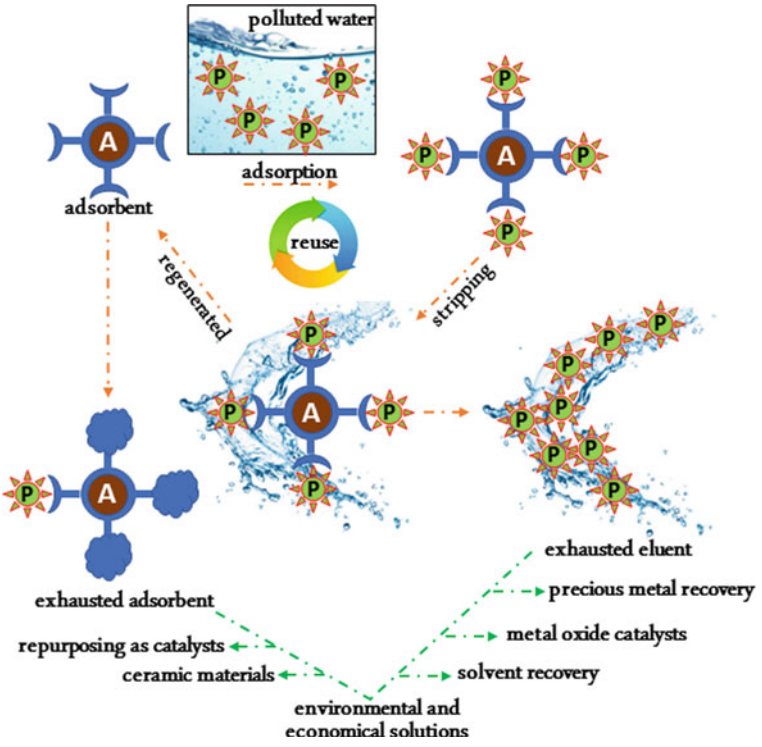

Fig. 9.1 A schematic representation of the adsorption process as an economical and greener initiative

9.2 Regeneration and Reuse of Spent Adsorbents

The regeneration of a spent magnetic adsorbent is a crucial process to cover the environmental and monetary cost of the adsorbent. An ideal regeneration process should revert the adsorption process and restore the physicochemical properties of the adsorbent. In the regeneration process, adsorbed pollutants are stripped out of the adsorbent and a limited number of adsorption sites are made available for the next cycle. The choice of the regeneration process is based on the physicochemical properties of pollutants, stability of adsorbent, and economic constraints. For heavy metal-loaded magnetic adsorbents, stripping agents (eluents) are recommendable. The eluents are either acid/base solutions or solutions of complexing agents, which could extract heavy metals into the solution phase [26]. The strength of eluents depends on the nature of adsorbate–adsorbent interactions, contact time, and temperature. For extracting organic pollutants from the spent adsorbent, the use of polar solvents like ethanol and methanol is recommendable [35]. In some studies, regeneration methods like thermal regeneration and ultrasonication have been reported, which are less tedious and suitable for stable adsorbents. The reusability of magnetic adsorbents for the removal of heavy metals and organic pollutants has been listed in Table 9.1. In general, the choice of using acid/base solutions for the desorption process is largely based on adsorbent–adsorbate interactions as a function of pH. In the pH-dependent adsorption profiles, the pH at which the pollutant uptake is the least is considered a suitable pH to desorb it. The adsorbed cations of heavy metals are stripped using strong acidic solutions due to the competitive nature of protons for the adsorption sites [39, 43, 44]. The use of strong acids for desorption lowers the structural stability of the adsorbent, which is due to the dissolution of magnetic nanoparticles. In some cases, where heavy metals are adsorbed via an ion-exchange mechanism, the use of electrolytic solution (KCl/NaCl) is preferred. The choice of using electrolyte solution for desorption of Tl(I) from magnetic Prussian blue derivative adsorbent was reported by Yeisy et al. [29]. The study reported the use of HNO_3, NaOH, and KCl as three elution agents, where pH swing does not affect Tl(I) desorption (Fig. 2a). Though HNO_3 could desorb 26% of the adsorbed Tl(I) through proton exchange, KCl was the best eluent with a 97% desorption efficacy (Fig. 2b). The oxyanions (arsenate and chromate) are usually desorbed with strong basic solutions (NaOH) [6, 25]. Chen et al. reported the use of 0.5 mol L^{-1} NaOH solution for the elution of chromate from spent polyethyleneimine-functionalized Fe_3O_4 [6]. The pH-dependent adsorption profile showed very low chromate uptake at pH 12, which made 0.5 mol L^{-1} NaOH solution, an ideal choice for the desorption studies. For this reason, the adsorbent was used for 20 cycles with a 13% loss in the uptake capacity after the 20th cycle.

The method of pH swing is equally applicable to the stripping of organic pollutants from spent adsorbents. Since organic charged dyes are adsorbed by electrostatic interaction, pH variation using an acid/base could give a better desorption efficacy [42, 50].

Table 9.1 Reusability of magnetic adsorbents for heavy metals and organic pollutants removal

Adsorbent	Pollutant	Eluent	%Ads (1st cycle)	%Ads (*n*th cycle)
Fe_3O_4 decorated with β-cyclodextrins-functionalized graphene oxide [25]	As(V)	NaOH	99	61 (5)
Diethyl-4-(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)phenyl phosphonate-capped biogenic Fe_3O_4 [43]	Cd(II)	HCl	91	77 (5)
EDTA-cross-linked magnetic chitosan DTPA-cross-linked magnetic chitosan [49]	Cd(II)	HNO_3	99 99	87 (10) 82 (10)
Fe_3O_4/MnO_2 [24]	Cd(II)	HNO_3	95	83 (5)
Polyethyleneimine-functionalized Fe_3O_4 [6]	Cr(VI)	NaOH	100	87 (20)
Magnetic iron oxide@$CaCO_3$ [21]	Cr(VI)	HCl	100	95 (5)
Fe_3O_4–walnut shell [39]	Pb(II)	HCl	79	60 (10)
Fe_3O_4@diaminophenol and formaldehyde-based polymer [44]	Pb(II)	HNO_3	92	81 (5)
3,4-Dihydroxyphenethylcarbamodithioate-capped Fe_3O_4 [45]	Hg(II)	HNO_3	93	83 (5)
Magnetic Prussian blue derivative [29]	Tl(I)	KCl	100	95 (6)
Magnetic@chitosan/halloysite [42]	Congo red	NaOH	100	90 (6)
Tryptophan-functionalized Fe_3O_4 [40]	Congo red	Ethanol	98	87 (5)
Magnetic bacterial cellulose nanofiber/graphene oxide polymer aerogel [2]	Malachite green	Acetic acid/methanol	93	63 (7)
Fe_3O_4@graphene oxide nanohybrids [13]	Rhodamine B Methylene blue	Ethanol	100 100	86 (8) 66 (5)
$CoFe_2O_4$/graphene oxide [15]	Methylene blue	400 °C	96	79 (4)
Ce–MOF@Fe_3O_4@activated carbon composite [35]	Methylene blue Indigo carmine	Methanol	100 100	96 (4) 87 (4)
Magnetic hollow carbon microspheres [30]	Rhodamine B	NaOH + 400 °C	98	92 (6)
Carbon dot-modified magnetic carbon nanotubes [9]	Carbamazepine	Acetonitrile	77	75 (7)
Magnetic carbon microspheres [8]	Sulfonamide	pH 10 + ultrasonication	94	75 (5)
Hypercrosslinked magnetic resin Q100 [50]	Tetracycline	NaOH	100	95 (10)

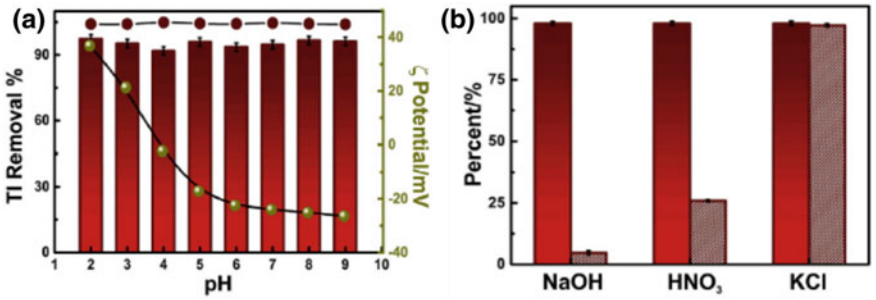

Fig. 9.2 **a** Tl(I) removal versus pH (*right axis:* ζ potential); **b** Tl(I) recovery percent versus Eluting solution for magnetic Prussian blue derivative adsorbent. Reproduced with permission from Elsevier (2021) [29]

Organic solvents like ethanol and methanol are also recommended as eluents either in the form of pure solvents [2] or acid/basic solvents [13] due to their high solubility in polar organic solvents. As compared to solvent stripping, thermal regeneration is a simplistic approach to eliminating organic pollutants from spent adsorbents. For a thermally stable adsorbent, heating at an elevated temperature could volatilize the adsorbed organic pollutants. Gupta et al. demonstrated the use of thermal regeneration of methylene blue-adsorbed $CoFe_2O_4$/graphene oxide by heating at 400 °C for 1 h in an inert atmosphere [15]. The nanocomposite adsorbent retained 80% of its dye uptake capacity even after the 4th cycle. In another study, the combined effect of pH variation and thermal regeneration was used to desorb rhodamine B from magnetic hollow carbon microspheres. The spent adsorbent was treated with 1.0 mol L^{-1} NaOH solution and heated at 400 °C for 1 h in an inert atmosphere. The method successfully regenerated the adsorbent with a 6% loss in the performance after the 6th cycle, which made it suitable for more adsorption–desorption cycles [30]. Thus, the development of regeneration strategies is largely dependent on the chemical/thermal stability of the adsorbent, the strength of interactions, the chemical state of pollutants, and so on. More information on the regeneration of adsorbents and recovery could be found in the published specialized reviews [14, 26, 34].

9.3 Disposal of Spent Stripping Solutions

The regeneration of spent adsorbents by solvent stripping methods generates large volumes of toxic organic/inorganic solutions, which need to be managed as per the rules set by the environmental protection agencies. So far, research works are the least concerned with the management of spent eluents. The most judicious way is the use of the same eluent for multiple cycles to produce solutions with a high concentration of heavy metals, which are like ore leachates. Heavy metals like copper, platinum, palladium, and nickel have a high monetary value due to their widespread use. The

heavy metals from the spent solutions could be extracted by different metallurgical technologies like flotation with pyrometallurgy [38], solvometallurgy, and electrolysis [3]. Even multi-elemental solutions could be processed by varying the pH to extract precious metals [47]. Moreover, heavy metals are used as catalysts and co-catalysts in different industrial processes like catalytic cracking, syngas production, and Fischer–Tropsch process [4, 27]. These spent solutions can be converted to metal oxide catalysts by precipitation and thermal treatment, which will be an alternative way to convert waste into value-added products [10]. Many of the heavy metals (e.g., arsenate, chromate, and mercury) are costlier to recover than their market value but are highly toxic to living beings. Spent solutions, rich in such heavy metals are concentrated, encapsulated, and disposed of in well-maintained landfill sites [33]. Organic solvents used as dye-stripping agents like acetone, methanol, and ethanol have a low boiling point of 56, 65, and 78 °C, respectively. The dye-rich organic solvents could be regenerated by evaporation to recover the stripping solvents and dyes. Though the evaporation process is energy-intensive, the use of acetone as the stripping agent in combination with the concentrated solar radiation as the heating source could be a cost-cutting solution for the management of dye-rich spent solvents. Most of the suggested methods are yet to be reported in the literature in detail. The research work dealing with wastewater treatment is largely focused on answering whether the fabricated adsorbent can adsorb. If yes, then how much? Whether it could be reused and if yes, then for how many cycles? But it is expected that in the future studies, researchers will be more interested in exploring disposal strategies for exhausted eluents.

9.4 Disposal of Exhausted Adsorbents

After numerous cycles of adsorption-regeneration, the pollutant uptake capacity of an adsorbent reaches a point where it is no more economically feasible to reuse it. The spent adsorbent is replaced by a fresh one and the spent adsorbent is treated as a highly toxic solid waste. Dumping of these toxic exhausted adsorbents in open spaces poses severe risks to human health and the environment, especially in countries where waste segregation and disposal strategies are limited [5]. Even improper disposal of exhausted adsorbents in landfills or poor maintenance after disposal could generate the issue of secondary pollution of groundwater or soil [51]. Thus, it is critical to construct suitable waste-disposal mechanisms from the environmental perspective. However, the concept of finding an alternative application for a waste product is more valuable than finding a new path for disposal. Gupta et al. reported an alternative application of exhausted CuO after the H_2S desulfurization process. The study reported better methylene blue photodegradation efficiency for exhausted materials compared to the fresh CuO (Fig. 9.3) [16]. A mesoporous silica (MCM-41) was developed for the removal of chromate from waste solutions. The Cr-loaded MCM-41 was calcined in air at 550 °C for 6 h to produce the Cr/MCM-41 catalyst, which was used for the catalytic degradation of methyl mercaptan (a volatile organosulfur

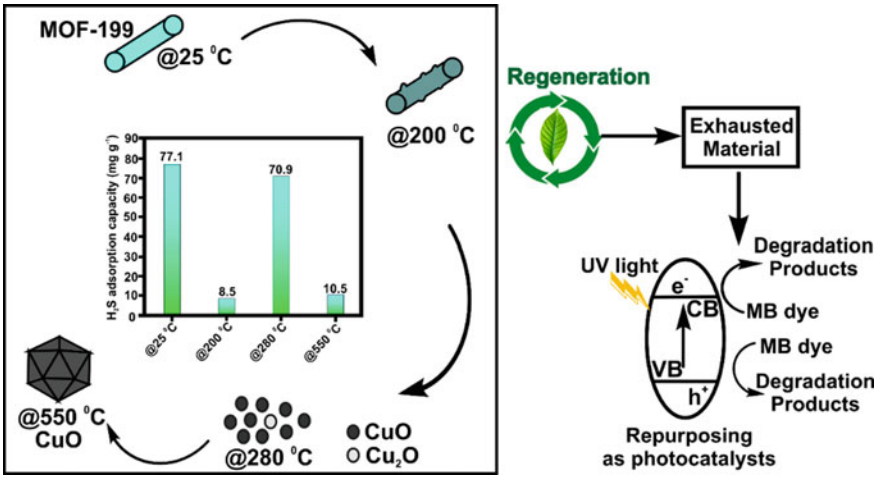

Fig. 9.3 The exhausted CuO was repurposed for dye photodegradation as an environmentally benign route to provide value to the waste. Reproduced with permission from the American Chemical Society (2021) [16]

compound with an obnoxious odor) [19]. The waste-to-wealth concept has been used for the conversion of spent Pb-loaded hydrochar to a carbonaceous anode for lithium-ion batteries [48].

On a similar note, reports on the waste-to-wealth concept are available for spent magnetic adsorbents as well. A magnetic mesoporous silica/poly(m-aminothiophenol) nanocomposite was fabricated for Hg(II) removal [11]. The adsorbent showed a remarkable sorption capacity of 243 mg g^{-1} within 10 min of agitation. The adsorbent was reused for 5 cycles with a retention of 76% of its initial adsorption capacity. The spent adsorbent was repurposed as a catalyst for the conversion of phenylacetylene to acetophenone with a 97% yield. Further modifications were made to the material to develop a magnetic network polymer composite by introducing poly(m-aminothiophenol) and chitosan onto the magnetic nanoparticle using tannic acid as a cross-linking agent. The material possessed a Hg(II) removal capacity of 245.5 mg g^{-1} with excellent reusability and selectivity. Moreover, the exhausted adsorbent catalyzed the transformation of different phenylacetylene derivatives into corresponding acetophenone with ~ 98–99% yield [12]. In another study, the fabricated magnetic mesoporous silica/chitosan composite was studied for Hg(II) removal [20]. The adsorbent has an uptake capacity of 437.8 mg g^{-1} with the retention of 87% of its initial adsorption capacity after the 6th adsorption-regeneration cycle. The spent adsorbent was used as a thermal catalyst for the conversion of phenylacetylene to acetophenone with a yield of 98.3%.

Cruz et al. have reported a novel concept of turning waste into wealth on two fronts [7]. The natural organic matter (NOM)-rich water from a waterfall was used for the synthesis of $CoFe_2O_4$/NOM as an adsorbent for the removal of chromate. The adsorbent was reusable, where the adsorption performance was 82% in the 5th cycle

as compared to 96% in the 1st cycle. The spent adsorbent was used as a catalyst for the reduction of 4-nitrophenol in the presence of $NaBH_4$ as the reducing agent, where a conversion efficiency of 99.9% was recorded within 3 s. The catalyst was studied for two additional cycles, where the conversion efficiency was still 99.9% within 30 s. Apart from excellent reusability, the material showed insignificant leaching of chromium and iron in the solution phase. On the same concept, an adsorbent and subsequently a catalyst was developed using waste biomass [28]. Fir sawdust was used as a bio-sorbent for the adsorption of Cu(II) and Fe(III) ions. This metal-loaded biomass was pyrolyzed to yield Cu–Fe_3O_4–carbon material, which found application as a reusable catalyst for the reduction of 4-nitrophenol.

A low-cost Fe_3O_4/Carbon composite, developed for the adsorptive capture of chromate from wastewater, was repurposed as an anode material for potassium-ion batteries after chromate adsorption. The redox reaction between Cr(VI) and Cr(III) was responsible for the K-storage mechanism. Thus, more adsorption on chromate was favorable for improving the K-storage capacity of the material [31]. Zn–Al–La layered double hydroxide is an inorganic synthetic clay, which showed a high uptake behavior for amaranth (azo dye). After dye adsorption, the spent material was calcined (to form a carbonaceous composite material) and repurposed as a photocatalyst for the degradation of ibuprofen [41]. On the same line, spent magnetic adsorbents could be used as suitable photocatalysts for the degradation of organic pollutants. Specifically, transition heavy metals along with iron oxide in these adsorbents are excellent UV–visible responsive photocatalysts [17, 18]. Thus, spent magnetic adsorbents could be used for alternative applications of catalysis that comply with the principles of industrial symbiosis with extraordinary environmental benefits.

Spent adsorbents rich in heavy metals could be repurposed to produce ceramic materials, which works as an alternative method for stabilizing toxic metals and at the same time serves the purpose of construction material. One such example was the use of lime activated fly ash for adsorptive removal of heavy metals (Pb, Zn, and As) and subsequent valorization of exhausted adsorbent as an additive in construction material [23]. Rathore and Mondal have reported the stabilization of arsenic-loaded adsorbents namely, thermally treated laterite, acid–base treated laterite, and aluminum oxide/hydroxide nanoparticles as clay bricks. Brick properties like density, water absorption, shrinkage, compressive strength, and efflorescence complied with the Indian standards set for construction materials. Moreover, the maximum amount of arsenic in the brick leachate was well below the United State Environmental Protection Agency permissible limit of 5.0 mg L^{-1} [36, 37]. Though stabilization of spent magnetic adsorbent as clay bricks has not been studied, Verbinnen et al. developed a novel method to stabilize heavy metals and oxyanions forming elements (Cr, Ni, Cu, Zn, As, Cd, and Pb) by adsorbing it on zeolite- or perlite-supported magnetite. This was followed by mixing the spent adsorbent with industrial sludge (sludge/adsorbent ratio of 97/3) at 1100 °C for 0.5 h. Based on the study, the leaching of toxic metals from the ceramic was below the regulatory limits [46]. The production of lime and cement used for the solidification/stabilization of spent adsorbents

before being dumped in landfills has a high carbon footprint due to CO_2 release [22]. Stabilization of spent adsorbents as bricks and ceramics saves raw clay materials and lowers the environmental impact and cost of the landfill process.

9.5 Conclusion

Repurposing of spent adsorbents is a new concept, which needs to be explored more often to make the overall adsorption process, environmentally benign and affordable. In the literature, only a handful of research works have been dedicated to the cause. Nevertheless, the idea that has cropped up in these studies is worth reporting in the context of the environmental sustainability of the process. While the researchers are dedicated to exploring regeneration techniques, finding alternative applications for the exhausted adsorbents is highly lucrative and novel. With the enrichment of the literature with such studies in the future, it will be easier to judge the environmental applicability of the adsorption processes.

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