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Desorption in heavy metal adsorption: A review

Beyhan KOCADAGISTAN
Atatürk University, Environmental Engineering Department, 25240 Erzurum, Türkiye
beyhank@atauni.edu.tr

Abstract:
Heavy metal pollution is an important environmental pollution problem that is attracting more and more attention today. When treatment methods such as chemical precipitation, micro, ultra and nanofiltration, reverse osmosis, electrocoagulation or adsorption, which are frequently applied for heavy metal removal from water and wastewater, are insufficient, heavy metal pollution in rivers, lakes and similar water sources has increased. In all of these methods, more economical and more applicable methods are preferred, and adsorption has been one of the most used methods. However, it is an important point to make the final disposal of the pollutant as a result of this process, which works according to the principle of removing the pollutants from the water by trapping them in the adsorbent. In this way, the adsorbent, which has retained the heavy metal, is prevented from being reintroduced to the nature, and the adsorption process is more usable and more economical. In this review article, the method used for desorption (regeneration), environmental factors affecting it and the amount of recovery depending on the desorption efficiency were also examined and summarized.

***Key Word**: Heavy metal; adsorption; desorption; water treatment.*

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I. Introduction

The availability of clean drinking water, which is a basic requirement for humans and wildlife, is a primary condition for maintaining a healthy life. However, the increasing demand for industrialization due to population growth also causes a gradual decrease in the amount of accessible clean water. What is really frightening here is the abundance of research that indicates that this difficulty in accessing clean water due to global warming will increasingly change in the coming years. It has been reported that diseases increase and millions of people die every year due to simple diseases that are actually preventable, especially in developing countries, when the conditions of access to clean water are difficult [1–5].

With rapid population growth and accelerated industrialization, heavy metal contents have begun to reach concentration limits in clean water sources that can harm human health. Among the heavy metals harmful to human health, Pb, Hg, Cd, As, Cu, Zn and Cr draw more attention. As and Cd can cause cancer, Hg can cause mutations and genetic damage, while Cu, Pb and Hg can cause brain and bone damage. Mostly, metals and their compounds such as Cu, Zn, Hg, Cd, Pb, Sn, Mn, As, Cr, Co, Ni, Ag and Al with atomic density greater than 4 ± 1 g/cm^3 cause heavy metal pollution. They are generally considered to be the most important toxic mineral pollutants in water and soil systems [6].

Chemical precipitation, electrochemical processes, ion exchange, adsorption and membrane processes have an effective place among the treatment methods that are frequently used in the removal of heavy metals from water, because these pollutants cannot be removed at desired levels in the primary, secondary or tertiary treatment stages of water. Among these processes, adsorption is widely used in the industrial field due to its low cost, simplicity and less sensitivity to hazardous pollutants [7, 8].

There are many studies in the literature on the removal of heavy metals from water and wastewater by the adsorption method. In these studies, different types and structures of adsorbent materials, such as activated carbon supported nanoscale zero-valent iron [9], magnetite nanoparticles [10], chitosan/epichlorohydrin composite [11], Pectin-guar gum [12], alginate and magadiite [13], *Chrysanthemum indicum* [14], kaolinite [15, 16], iron blast furnace slag [17], tea plant waste [18], Gelatin–Siloxane Hybrid Monoliths [19], pectin-based biosorbents [20], chitosan/polyethyleneimine magnetic hydrogels [21], modified coal fly ash [22], walnut green peel [23], lignocellulosic waste-derived biosorbents [24] were used.

Adsorption is a widely used technology to remove heavy metals from water and wastewater, but the regeneration capacity of adsorbents is an important limiting factor for them in practice [24]. It is important that heavy metal ions bound on the adsorbent material by adsorption are not released back to the nature after the process. Already, since the metal binding mechanism is realized by chemisorption, the adsorbed metal can be desorbed back into the solution under suitable conditions [25]. In this respect, desorption method is used to recover the retained substances after adsorption in an effective, economical and environmentally friendly manner. Moreover, desorption is an environmentally friendly technique with low energy consumption, and due to the reversible nature of most adsorption processes, adsorbents can be regenerated by simple desorption methods [26].

In this study, heavy metal adsorption studies encountered in the literature in recent years were examined in detail. This review article, in which the methods and agents used for desorption and the regeneration yields obtained are considered to be an effective resource for further research.

II. Desorption of Heavy Metals

Different adsorption methods and different desorption agents have been used in studies for the adsorption of heavy metals. In these studies, the following equations were generally used for the adsorption and desorption;

$$C_r = C_s - \frac{C_{des}}{m} V \qquad (1)$$

where C_r and Cs are the amounts (mg/g) retained and adsorbed metal concentrations, respectively; C_{des} is the equilibrium concentration (mg/L) in the desorption process; V is the volume (L) of solution used for the desorption process; and m is the mass (g) of adsorbent [27].

$$\%D = \frac{C_{des}}{C_o} x100 \qquad (2)$$

where $\%D$ is the desorption efficiency, C_o is the concentration of metal ions (mg/L) in solution before and after desorption, respectively [28].

Importance of pH

pH is one of the most important physicochemical parameters that affect the electronic balance on non-covalent bonds and destabilize the electronic configuration in the environment. In low pH solutions, high concentrations of hydronium cations are present and compete for a significant amount of functional groups interacting with light metals or other metal cations [29]. As the pH increases, more negatively charged ligands (carboxyl, hydroxyl groups, among others) are depleted, then metallic cations are attracted and binding to the cell surface occurs [30].

Finding the optimum adsorption pH is very important, as pH affects the removal of metal ions from aqueous solutions by influencing the chemistry of metals and changing the surface charge of the particles [31]. At the same time, since the amount of heavy metal recovered as a result of the desorption process also depends on the amount of heavy metal retained by the adsorption process. pH is also an important parameter in desorption, although not as much as in adsorption, because overall desorption is controlled by both metal desorption and re-adsorption reactions. For example, in a desorption process, pH has been found to have little effect on total Cu(II) desorption [32]. In studies where adsorption and desorption are carried out simultaneously, the desorption pH value is generally carried out with the optimum pH value used for adsorption. In the study in which the Ni adsorption properties of peat, compost, brown algae, sawdust and wood ash were compared, adsorption and desorption experiments were carried out together at pH 7 [33]. However, the importance of pH in the desorption process depends on the method applied for desorption. When methods such as thermal processes [34–36] or sonification [37] are used for regeneration, pH is no longer an important monitoring parameter. On the contrary, when acid, base or different chemical substances are used as regeneration agents, the pH value of the environment becomes more important [11, 38–41]. Accordingly, many researchers have tried to reveal the effect of pH on the desorption process. Many researchers have tried to reveal the effect of pH on the desorption process. In a study studying the adsorption of cadmium on thiol‐ modified bentonite grafted with cysteamine hydrochloride, it was seen that as the pH increased from 1.5 to 5.5, desorption rates decreased as some of the H^+ of the adsorbed cadmium interacts with the Cd^{2+} adsorbed on the adsorbent [42]. In a study investigating heavy metal adsorption with EICP-Treated Plastic Fines, it was shown that the rate of cadmium desorption decreased with decreasing pH [43]. Desorption of T1 from magnetite adsorbent was found to be ineffective at pH greater than 4.0, but highly effective at pH less than 3.0 (97 ± 3%). In fact, almost complete desorption (101% ± 9%) was achieved at pH 2.0 [44]. In the study performed with different concentrations of NaOH solutions (0.05-0.50 M), it became clear that Cr(VI) desorption starts at pH ≥ 8, and higher NaOH concentration is required to increase the Cr(VI) desorption efficiency [45].

Desorption and reusability

Desorption is the opposite of adsorption and is a physical process in which an adsorbed substance is released from a surface when it gains enough energy to overcome the activation barrier of the limiting energy that binds it to the surface. At the same time, the mechanism of Desorption is similar to that of adsorption. It may involve ion exchange or complexation, in which metals are separated from the adsorbent with a suitable solution to produce a small, concentrated volume of metal-containing solution [46].

The desorption process is an extremely important aspect of the adsorption-absorption process in terms of revealing the mobility and state of the chemicals in the environment. Also, the desorption property of the adsorbent is very important as it can significantly reduce the overall cost of the process.

An important part of the studies on desorption is in the direction of reducing soil pollution. Heavy metals, which are both the soil's own component and mixed with the soil as a result of pollution, are generally desorbed into the waters after the pH change [47]. Therefore, soil contaminated with heavy metals must be treated effectively. A wide variety of soil remediation approaches such as chemical treatment, soil washing, electrokinetic method, bioremediation and phytoremediation technique, thermal treatment have been applied to contaminated soils [39, 48, 49].

In the desorption process, adsorbed chemicals are not always easily desorbed, as some sorption reactions are partially irreversible. In such cases, the desorption process may require drastic destruction of the adsorbate, destruction of the adsorbent, or both [50]. Desorption may occur either by thermal treatment or through suitable desorbing agents. However, chemical compounds are widely used as regeneration agents in the desorption process. Due to the reactions of heavy metals in chemical reactions, solution pH value is one of the most important parameters. For this reason, the most common method used during desorption is to change the pH value of the solution by various methods. Sulfuric acid [51], hydrochloric acid [52, 53], nitric acid [31, 54, 55] and EDTA [56–58] are the most prominent. In addition, sodium hydroxide [59–61], various salt solutions and different chemical substances [56, 62, 63] are also used for this purpose. There are many studies showing that protons of mineral acids such as HCl, H_2SO_4 and HNO_3 can remove metals from sorbent binding sites [64]. In addition, EDTA, a powerful chelating agent, has also been shown to be an effective desorbing agent [65].

Two important parameters to consider in the desorption process are the solid/liquid ratio and the final concentration after adsorption equilibrium, which should be as high as possible because only a small volume of eluant is required to displace all the deposited metal. However, since metal adsorption is a reversible process, the high metal concentration released into the solution may reduce the desorption efficiency as it may still leave some metal residue at equilibrium [46].

In the adsorption and desorption researches, two processes are carried out simultaneously or sequentially. In a study in which adsorption and desorption were carried out in a sequential process, the metal solution was sent to the adsorption system for 150 minutes to allow adsorption, and then treated with electrolyte solution for another 150 minutes to induce desorption [66]. Different solutions (EDTA, NaOH, urea, Na_2CO_3 and NaCl) were used for the desorption of humic acid coated iron oxide nanoparticles (HINP), 3.7 mg nanosorbent was mixed with 50 mg/L Cr^{+6} in the centrifuge tube and incubated for 30 minutes for adsorption to occur. The chromium-loaded nanoparticles were separated by centrifugation and resuspended in the eluents (1 mL) referred for desorption, while the Urea-free mixtures were heated at 60 °C for 1 h and then the desorbed HINP was reused for the second chromium adsorption cycle [67]. Similarly, in a study comparing different regeneration solutions, desorption was applied in five cycles, and maximum regeneration efficiencies of 93.44% for H_2SO_4 and Cu and 92.0% for Ni were reached [68]. In the study using NaOH as the desorption agent, the percentages of desorbed Hg^{2+} were found to be 37.5 %, 41.6% and 68.3% at 10, 100 and 1000 mmol/L NaOH concentrations, respectively [69]. In another study, when the adsorption process was over, 20 mL of 0.05 M $NaNO_3$ without Cd^{2+} and Cu^{2+} were added sequentially and the mixtures were mixed at 200 rpm in an orbital shaker at 25.0 °C for 8 hours. The desorption process was carried out in duplicate. The amount of metal retained was calculated from the difference between the amount adsorbed and the amount desorbed [27]. Various concentrations (0.25-3.0 mol/L), volumes (4-8 mL) and flow rates (1-6 mL/min) of diluted HNO_3 and HCl were investigated as desorption eluents and according to the results obtained, higher recovery values with HNO_3 has been obtained. In addition, quantitative recovery values were obtained for metals even at lower tested concentrations (0.50 mol/L) when using HNO_3 as the eluent [55]. In a study examining the desorption of Pb^{2+}, both water and $NaNO_3$ solution were used. In the desorption procedure, 20 mL of ultrapure water was added to the tubes after adsorption, the procedure was repeated twice, two desorption steps were created, and the Pb^{2+} concentration in the supernatant was determined within 24 hours using atomic absorption spectrometry [70]. In the study, in which desorption was carried out in the same steps as adsorption, 20 mL of $NaNO_3$ solution was added to each of the residues after the adsorption process, then the mixture solution was shaken for 24 hours. To analyse the desorption ability of HAC, the concentration of V passed into the solution was determined [71]. In the study in which both Pb2+ and Zn2+ adsorption were investigated with nanostructured zeolite, much more desorption was observed with hydrochloric acid, and it was also observed that Pb^{2+} ions were more desorbed. It has been stated that this may be a result of the formation of a $PbCl_2$ precipitate, which is sparingly soluble in water, as opposed to the formation of the $ZnCl_2$ salt, which is well soluble in aqueous media [72]. The choice of the agent used during desorption can sometimes vary according to the reactions of the adsorbent to the chemicals. In the study, in which HCl, H_2SO_4 and HNO_3 with 0.1 M concentration were used to investigate the desorption ability, desorption experiments were carried out in triplicate, at pH 2 and 298 K for 3 hours with mixing. According to the results obtained, HCl solutions had the highest desorption value as well as the highest adsorbent degradation. However, since both the desorption value and adsorbent degradation for HNO_3 are less than HCl and H_2SO_4, HNO_3 was chosen as the optimum agent for the desorption experiments. The obtained desorption efficiencies were 60% and above [31]. The desorption agents used in the adsorption-desorption studies in the literature and the yields obtained are given in Table 1.

Table 1: Various desorption agents and regeneration efficiencies.

Adsorbent	Heavy metal	Regeneration rate (%)	Desorption agent	Ref.
Grafted cross-linked chitosan beads	Pb^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, Cd^{2+}	95.98-98.87	HCl	[11]
Natural allophane	Cd^{2+}, Cu^{2+}	83.5-95.0	$NaNO_3$	[27]
κ-carrageenan and N-doped carbon dots	Pb^{2+}, Cu^{2+}, Hg^{2+}, Cd^{2+}	> 90	HNO_3, EDTA	[28]
$MnFe_2O_4$ and $CoFe_2O_4$	Zn^{2+}	> 70	HNO_3	[31]
Poly(N-isopropylacrylamide)	Cr^{3+}	--	Heating	[34]
A-MIL-121	Cu^{2+}	> 90	Heating	[35]
Soil	As^{3+}, Zn^{2+}, Cu^{2+}, Cd^{2+}	> 90	Heat, NaOH, $NaNO_3$	[36]
Graphene oxide	Pb^{2+}	--	NaCl, $CaCl_2$	[37]
Polyamide-based microfibers	As^{3+}, Zn^{2+}, Cu^{2+}, Cd^{2+}, Cr^{3+}, Pb^{2+}, Ti^{2+}, Al^{3+}	90–95	Nitric Acid	[38]
Soil	Cr^{3+}, Mn^{2+}, Ni^{2+}	39.9- 77	Citric acid	[39]
Natural V, Ti-bearing magnetite	Pb^{2+}	99.3 wt	HNO_3	[40]
L-Lysine Modified Montmorillonite	Pb^{2+}	95	H_2SO_4, HNO_3	[41]
Magnetite	Tl^{1+}	99.97	HCl, H_2SO_4, HNO_3	[44]
Aminophosphonate-based sorbents	Pb^{2+}	> 95	HCl	[52]
Green marine macro alga	Zn^{2+}	90.34	HCl	[53]
MgO/WO3 nanoadsorbent	Cu^{2+}, Fe^{2+}, Cr^{6+}	> 90	HNO_3	[54]
Green nano sorbent	Cd^{2+}, Fe^{3+}, Co^{2+}, Cr^{3+}, Cu^{2+}, Mn^{2+}, Pb^{2+}, Ni^{2+}	≥ 93	HNO_3, HCl	[55]
Clays and clay minerals	Ni^{2+}, Pb^{2+}, Zn^{2+}	> 90	$CaCl_2$ EDTA	[56]
Natural High Buffering Soil	Pb^{2+}, Zn^{2+}, Ni^{2+}	66-93	EDTA	[57]
Soil	Pb^{2+}	79	EDTA	[58]
Cobalt ferrite nanoparticles	Pb^{2+}	95	HCl, HNO_3, NaOH, KOH	[59]
Nanocomposite fiber membrane	Pb^{2+}	> 70	NaOH	[60]
Soil	Cd^{2+}	> 90	$NaNO_3$	[61]
Tire wear particles	Cd^{2+}, Pb^{2+}	65.4-75.73	Pepsin A + NaCl	[63]
Goethite	Ni^{2+}, Co^{2+}, Cr^{3+}	> 90	NaNO3	[66]
Biochar	Pb^{2+}	59	$NaNO_3$	[70]
Nanostructured zeolites	Pb^{2+}, Zn^{2+}	73.3-80.7	NaCl	[72]
Water treatment residual nanoparticles	Cr and Hg		KNO3	[73]
Alginate beads	Cu^{2+}, Cd^{2+}	96.5-97.1	HCl	[74]
Graphene nanoplatelets	Hg^{2+}, Pb^{2+}, Cu^{2+}	> 90	HNO_3	[75]
Magnetic graphene oxide/lignin	Pb^{2+}, Ni^{2+}	> 90	HCl	[76]
Activated carbon/magnetite nanoparticles	Cu^{2+}	> 90	CaCl2	[77]
Chemically Modified Biochars	Pb^{2+}	98.31	HNO_3	[78]
Phaseolus vulgaris L.	Cd^{2+}, Pb^{2+}	98	HNO_3	[79]
Silty Clay	Cu^{2+}	100	NaNO3	[80]
Magnetic Bentonite	Pb^{2+}	95.12	NaNO3	[81]
Fax fibres	Zn^{2+}, Cu^{2+}, Pb^{2+}	80-100	HCl, HNO_3	[82]
Polyacrylonitrile-based Hydrogel	Cr^{3+}, Ni^{2+}	51.6-98.3	HCl + electric current	[83]
Hydrous Ce_1–xZrxO2	Cr^{6+}	97	NaOH	[84]
TiO2 nanofibers	Pb^{2+}, Cu^{2+}, Ni^{2+}	98.76	HCl, HNO_3, NaOH	[85]
Micro and Nano-sized Biogenic CaCO3	Cd^{2+}, Pb^{2+}	20	$NaNO_3$	[86]
Magnetic hydrogel nanocomposite	Cu^{2+}, Cd^{2+}, Pb^{2+}	92	HCl	[87]

Desorption kinetics

To investigate the control mechanism of adsorption processes such as mass transfer and chemical reaction, a suitable kinetic model is needed to analyse the data [88]. Pseudo-first order, pseudo-second order, Elovich and Intraparticle diffusion kinetic models are generally used to reveal the reaction rate in adsorption-desorption processes and the linearised form of these models are given below [89].

Pseudo-first order model : $log(q_e - q_t) = log q_e - \frac{k_1}{2.303}t$ (3)

Pseudo-second order model: $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t$ (4)

Elovich model : $q_e = \left(\frac{1}{\beta}\right)\ln(\alpha\beta) + \left(\frac{1}{\beta}\right)\ln t$ (5)

where q_e is the adsorbed amount of adsorbate (mg/g) at equilibrium, q_t is the adsorbed amount of adsorbate (mg/g) at t time, k_1 (L/min) and k_2 (g/mg.min) are the rate constants of pseudo-first and pseudo-second order models, respectively, α is rate constant of adsorption (mg/(g.min)) and β is constant of desorption (g/mg).
The results obtained in the kinetic studies carried out in the adsorption-desorption studies are given in Table 2.

Table 2: Kinetic findings of adsorption-desorption studies.

Kinetic Model	Parameters	Ref.
Pseudo-second order	q_e=0.017-0.111, k_2=1.38-41.1, R^2=0.93-0.99	[33]
Elovich	α= 0.028-0.732, β= 56.9-316, R^2=0.87-0.94	[33]
Pseudo-first order	q_e=0.445-4.857, k_1=0.0437-0.2084, R^2=0.954-0.983	[42]
Pseudo-second order	q_e=0.493-5.096, k_2=0.0219-0.3282, R^2=0.992-0.998	[42]
Elovich	α= 0.211-1.964, β= 2.078-12.61, R^2=0.917-0.982	[42]
Pseudo-first order	q_e=259.82-610.51, k_1=0.0025-0.0226, R^2=0.521-0.964	[57]
Pseudo-second order	q_e=370.37-1986.95, k_2=0.00012-0.00075, R^2=0.956-0.999	[57]
Elovich	α= 168.81-22577, β=0.006-0.0021, R^2=0.837-0.987	[57]
Pseudo-first order	q_e= 4.87, k_1=0.3169, R^2=0.999	[71]
Pseudo-second order	q_e= 5.39, k_2=0.0744, R^2=0.985	[71]
Pseudo-first order	q_e= 7.752, k_1=0.028, R^2=0.673	[81]
Pseudo-second order	q_e= 18.182, k_2=0.033, R^2=0.992	[81]
Elovich	α=1543, β= 0.107, R^2=0.921	[81]
Elovich	α= 929-1183, β= 0.0075-0.0094, R^2= 0.945-0.989	[90]

III. Conclusions

In this review, we focused on the desorption processes carried out in adsorption processes to remove various heavy metals from water, both to recover the heavy metal and prevent it from being released back into the environment, and to obtain a more economical process by reusing the adsorbent. In the studies examined, it was observed that the desorption generally depended on the pH value, the type of adsorbent and especially the desorption agent used. It has been found in many studies that the heavy metal retained in adsorption can be recovered in large proportions and the adsorbent can be reused by creating suitable conditions and using a suitable desorbing agent. It has been seen that for the desorption process, acid or base solutions, which are mostly used to change the ambient pH value, have a more common use. Due to the high dependence of heavy metals on the pH value in chemical reactions, the method of changing the pH of the environment to release the retained metal may explain the fact that it is the most applied method. Apart from this, the use of EDTA as a desorption agent is also a very common method due to its chelating feature. Numerous studies have been found in which EDTA has been successfully used as a desorption agent. Another method used for desorption is thermal processes, and it has been seen that they are used in few numbers due to cost effect. Some studies focusing on desorption kinetics were found in the reviewed studies, but it was seen that there are not enough studies on desorption kinetics in the literature and it would be beneficial to focus more on the subject. One of the most important findings of the literature review is that although a large number of articles investigating adsorbents and their use have been published in recent years, the desorption process, which is one of the most important problems of the current century, which directly affects the economy of the environment and treatment plant, has not received enough attention. As the interest in the findings of adsorption efficiency or kinetics alone is decreasing, it may be a useful way for new researchers to focus on studies on the safe removal/reuse of heavy metals or other pollutants retained in adsorption by desorbing. In this review, it was concluded that there is some deficiency in the literature on this subject as well. Also, more focus should be placed on systems in which adsorption and desorption are carried out simultaneously for a more economical process.

IV. Conflict of interest

There are no conflicts to declare.

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