

Review

# Use of Surfactant-Modified Zeolites and Clays for the Removal of Heavy Metals from Water

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**Abstract:** The presence of heavy metals in water for human use or consumption represents a major risk to human health. It is therefore important to find materials to remove or minimise the concentration of these pollutants. The adsorption process for the removal of heavy metals is favoured by the use of low-cost materials that exhibit a porous structure and a high cation exchange capacity, such as zeolites and clays. On the other hand, chemical treatments, e.g., using acids and bases, can modify the properties of these materials, but more recently the application of surfactants has also shown to be successful for broadening their metal affinity and allowing the removal of diverse organic and inorganic pollutants from water. This paper reviews the application of modified zeolites and clays for the removal of heavy metals from water.

**Keywords:** heavy metals; water treatment; surfactant-modified surfaces; zeolite; clay

## 1. Introduction

Proper multi-criteria of the ecological, environmental, and socio-economic impacts of contaminated sites are necessary for the decision support of sustainable strategies. Environmental impact assessment for economic development, for example, requires the analysis of qualitative and quantitative factors more than subjective decisions. Diverse simulation tools, such as the eutrophication modelling of coastal zones [1]; the Soil and Water Assessment Tool (SWAT) applied in the evaluation of ungauged watersheds [2] and the impact of agriculture on water quality [3]; and fuzzy methods [2] have been used for water quality evaluation and represent helpful options to monitor the fluid flow, water quality and interactions of water treatments in real time in order to support sustainable land and water management [4,5].

Amongst the factors that affect the water quality, the discharge of heavy metals has exposed millions of people to their harmful effects, and it is one of the biggest environmental challenges faced by the world [6–9]. Heavy metals are persistent, bioaccumulative, and able to disrupt the metabolic functions and vital organs in humans and animals [10]. The biological function of heavy metals in trace concentrations is considered essential for plants and animals, but the chemical or physical transformation to which these metals are subjected will determine their toxicity and environmental behaviour [11]. Chromium (Cr) causes severe respiratory, cardiovascular, gastrointestinal, hematological, hepatic, renal, and neurological damage and has been suggested to be carcinogenic [12,13]. Arsenic (As) is a carcinogen present in water for human use that also produces harm to the cardiovascular, dermatologic, nervous, hepatobiliary, renal, gastrointestinal, and respiratory systems [14]. Mercury (Hg) is a widespread genotoxic environmental pollutant that induces strong alterations in the body tissues, damages the pulmonary and kidney functions, and affects the immune and cardiovascular systems. Hg also hampers the development of the nervous system in fetuses and children [15]. Cadmium (Cd) is considered a human carcinogen with an impact

on the respiratory system; its other effects include damage to the liver as well as the cardiovascular, immune, and reproductive systems [12,16]. Lead (Pb) causes poor development of the brain and the nervous system, and it is particularly dangerous to fetuses and young children. Exposure to Pb damages the kidneys and the reproductive and nervous systems, and it accumulates in the teeth and bones [17]. Nickel's serious health effects may include chronic bronchitis, reduced lung function, and cancer of the lungs, and it also causes allergic reactions in humans [12,18]. Zinc (Zn) is an essential trace element that is considered to be relatively nontoxic to humans, but an overexposure can cause stomach cramps and irritation, vomiting, nausea, anemia, or even death [19].

Heavy metals are removed from aqueous solutions applying treatments such as osmosis [20,21], ion exchange [22], coagulation and precipitation [23]. Adsorption is extensively applied due to the flexibility of the process and, accordingly, many adsorbents with high adsorption capacity, including zeolites and clays, have been investigated. On the other hand, some zeolites and clays have some limited metal adsorption capacity and may require a chemical treatment before use in water or wastewater treatments. In recent years, the chemical modification of zeolites and clays has been explored with the purpose of increasing the heavy metal binding ability of these materials. This review seeks to show the results of the application of surfactant-modified zeolites and clays on the removal of heavy metals from water. The reader is encouraged to consult the references for detailed information about the modification processes

## 2. Surfactants

Surfactants are long-chain molecules that contain both hydrophilic and hydrophobic moieties; they are found in food [24] as well as in cosmetic [25] and industrial formulations [26]. Depending on their origin, surfactants are either biosurfactants or synthetic, and they can be classified as ionic and nonionic according to their hydrophilic moiety [27].

### 2.1. Biosurfactants

Biosurfactants are synthesised by microorganisms, plants, or animals, with a critical micelle concentration (CMC) ranging from 1 to 200 mg/L and a molecular mass ranging from 500 to 1500 Da [28]. They are classified as glycolipids, lipopeptides, phospholipids, fatty acids, neutral lipids, and polymeric and particulate compounds [29]. Their hydrophilic moiety comprises carbohydrates, amino acids, cyclic peptides, phosphates, carboxylic acids, or alcohols, with the hydrophobic moiety made up of  $\alpha$ -,  $\beta$ -, and long-chain fatty acids [30,31]. Biosurfactants have been applied for the removal of copper, zinc, cadmium, and lead from soil and sediments [28,32].

### 2.2. Synthetic Surfactants

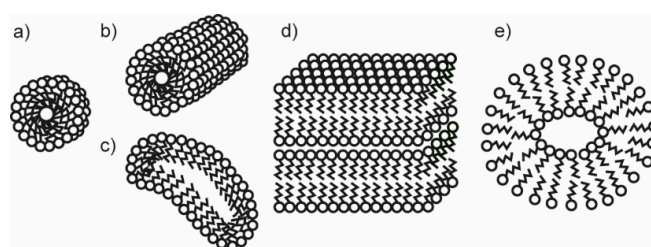
Synthetic surfactants can be either nonionic or ionic molecules. Nonionic surfactants cannot be ionised in aqueous solution; their hydrophilic moiety contains polyoxyethylene, polyoxypropylene, or polyol groups, and their hydrophobic moiety is comprised of fatty alcohols and saturated or unsaturated fatty acids. Compared to ionic surfactants, nonionic surfactants' lower CMC, higher degree of reduction in surface tension, and relatively constant properties in the presence of salts renders them more suitable to the remediation of soils, although the presence of clays and organic matter may influence their adsorption rate [30].

Ionic surfactants comprise the anionic, cationic, and zwitterionic or amphoteric surfactants [27]. Anionic surfactants usually have a sulfate, a sulfonate, or a carboxylate group as their hydrophilic moiety. They are found in pharmaceutical applications and detergent formulations and are used in soil remediation. Cationic surfactants have a hydrophilic part made up of quaternary ammonium groups. When cationic surfactants are used in soil remediation, they adsorb at negatively charged surfaces, such as silica [33] and clays [34], but they can also adsorb at cell membranes [30]. Dimeric (twin or gemini) surfactants contain more than one hydrophobic tail and hydrophilic head. These molecules usually have hydrophobic moieties comprised of paraffins, olefins, alkylbenzenes, alkylphenols, or

alcohols [30]. They have low CMC values, a hard-water tolerance, superior wetting times, and low Krafft points, and they are much more surface active than conventional surfactants [35]. A special type of dimeric surfactant that contains both anionic and cationic surfactants and can adsorb onto both negatively and positively charged surfaces is called a zwitterionic surfactant. These behave either as cationic or anionic surfactants depending on the pH of the solution [30].

### 2.3. Critical Micelle Concentration (CMC)

In an aqueous solution at a certain concentration, surfactants form aggregates (the CMC). A micelle is a structure containing a hydrophobic interior and a hydrophilic exterior that behaves as large molecule [8]. In nonpolar media, the structure of the micelle is similar but reversed, with the hydrophilic heads comprising the interior region surrounded by an outer region containing the hydrophobic groups and nonpolar solvent [36]. At concentrations above the CMC, surfactants can form other structures (Figure 1). These structures are continuously forming and disintegrating [36].



**Figure 1.** Surfactant aggregates: (a) spherical micelle; (b) cylindrical micelle; (c) worm-like micelle; (d) lamellar formation; and (e) vesicle.

## 3. Treatments for Metal Removal

Conventional processes include chemical precipitation [37,38], coagulation/flocculation [39–41], and ion exchange [22,42,43]. Adsorptive and membrane processes are widely used for the immobilisation and separation of metal species.

### 3.1. Adsorption

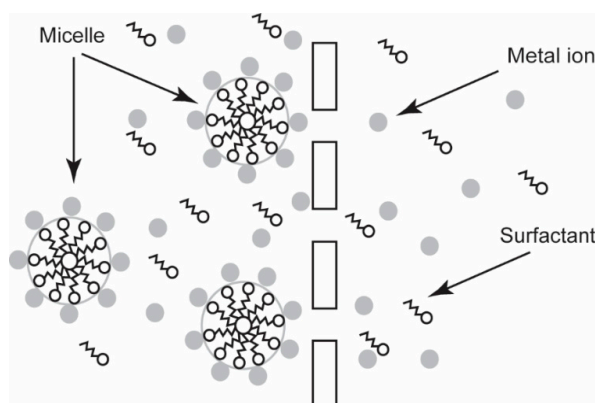
Adsorption has long been considered as a highly efficient water treatment method due to its low cost and ease of operation [12]. It involves the uptake and immobilising of contaminants on an adsorbent and the related mechanisms, such as surface adsorption, partition, surface precipitation, and structural adsorption [7,44–46]. Low-cost adsorbents such as lignocellulosic materials [47–49], biomasses [50–52], agrowastes [44,53–55], and inorganic materials [41,56,57] are frequently used.

### 3.2. Membrane Processes

Owing to their efficiency, easy operation, and space-saving properties, membrane processes, including microfiltration, reverse osmosis, nanofiltration, and ultrafiltration, are promising technologies for the removal of metals from water [58–60]. Microfiltration removes contaminants using membranes with pore sizes in the range of 0.1 to 10  $\mu\text{m}$  and at pressures of 0.5 to 2 bar. In reverse osmosis, there is a semi-permeable membrane that moves ions from a region of high solute concentration to a region of low concentration by applying an operating pressure ranging from 20 to 100 bar. Nanofiltration can retain ions and low-molecular-weight organics of less than 1 nm at lower pressure (typically 7 to 30 bar). Ultrafiltration separates heavy metals in solution on the basis of the pore size of the membrane (5 to 20 nm) and the molecular weight of the separating compounds, operating with a pressure of up to 10 bar [7,12].

The application of surfactants to a metal-laden water below the CMC promotes the formation of insoluble surfactant-metal complexes [7]. At concentrations above the CMC, the metal ions bind

into negatively charged micelles [61]. Both the metal-surfactant complexes and the metal-micelle aggregates can be further removed using micellar enhanced microfiltration (MEMF) or micellar enhanced ultrafiltration (MEUF; Figure 2). MEMF can remove multivalent metal ions from aqueous solutions using membranes manufactured from clays [62]. In MEUF, the surfactants are added at a concentration above their CMC to form large metal-surfactant micelles that are subsequently retained in an ultrafiltration membrane [31,63]. The MEUF increases the removal efficiency of metal ions using surfactants with an electric charge opposite to that of the targeted ions, but it has been suggested that the selectivity of this technique could be improved by using surfactants with the same charge as the metal ions [8,63].



**Figure 2.** Representation of a surfactant-based separation process (MEUF) for the removal of metal ions [64].

#### 4. Surfactant-Modified Zeolites and Clays

A variety of zeolites and clays play an important role in the environment and are frequently used as adsorbent materials for the removal of heavy metals. The sorption capacity of these materials can be enhanced when treated using acids and bases [65,66]. Owing to their amphiphilic nature, surfactants adsorb at diverse surfaces and interfaces in an oriented mode which will determine the hydrophilicity or hydrophobicity of the surface. Applied on zeolites and clays, surfactants have been used in the removal of diverse soil pollutants [11,67], remediation of non-aqueous phase liquids [68], pump-and-treat processes [69], and, more recently, to remove heavy metals from water.

The mechanisms for surfactant adsorption onto solid substrates are [36]: ion exchange, which involves the replacement of counter-ions adsorbed onto the substrate from the solution by similarly charged surfactant ions; ion pairing, which occurs via adsorbing surfactant ions from a solution onto the oppositely charged sites of the adsorbent that is unoccupied by counter-ions; hydrophobic bonding, which is based on the attraction between a hydrophobic group of an adsorbed molecule and a molecule present in the solution; adsorption by polarisation of  $\pi$  electrons, which occurs when there is attraction between the electron-rich aromatic nuclei of a surfactant and the strongly positive sites of an adsorbent; and adsorption by dispersion forces, which is based on the London–van der Waals force between the surfactant and the adsorbent.

##### 4.1. Zeolites

Zeolites are potential adsorbents used for water remediation because of their high ion exchange, adsorption molecular sieving capacity, and low cost. Natural and synthetic zeolites are aluminum silicates characterised by three-dimensional networks of tetrahedral units of silica and alumina, linked by the sharing of all oxygen atoms, with channels and/or interconnected voids. Zeolites display excellent an affinity for metal cations and adsorption and catalytic properties. In addition, cations of sodium, calcium, and potassium that are typically present in the channels of the zeolite structure

can be replaced by other metal cations, including lead, cadmium, zinc, copper, nickel, iron, and manganese [7,70,71].

By the addition of surfactants, the zeolite surface can be modified to enhance the anion exchange capacity and, to an extent, the potential to remove cations, anions, and organic compounds [70,72–75]. Two control factors for surfactant sorption on zeolites are the initial concentration of the surfactant and the external cation exchange capacity (ECEC) of the zeolite. The surfactant concentration determines the behaviour of the surfactant in solution, while the ECEC characterises the exchange capacity of the mineral surface.

#### 4.2. Clays

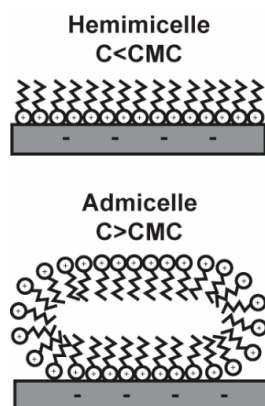
Clays are hydrous aluminum silicates characterised by a structure composed of a tetrahedral sheet of silicates linked to an octahedral sheet of aluminates. They can adsorb cations, anions, and neutral metal species from water bodies [9,75,76].

The metal adsorption in clays is driven by either selective or non-selective mechanisms. In selective sorption, clays adsorb ions through exchangeable cations and electrostatically bound counter-ions in the diffusion layer, which is formed due to the surface charge [77]. In non-selective or exchangeable sorption, the amount of cations sorbed is proportional to their relative concentration in the solution [78], and this occurs in a large range of pH values around the zero point of charge (ZPC) of the minerals whenever  $\text{pH} > \text{ZPC}$ ,  $\text{pH} = \text{ZPC}$  or  $\text{pH} < \text{ZPC}$  [77]. When clays are used in acid media, the metal adsorption rate increases as the pH increases. At near-neutral to alkaline conditions, the metal ions can be removed from the aqueous phase, presumably as hydroxyl species [12].

The application of cationic surfactants increases both the anionic adsorptive capacity and the cation affinity of clays, although the modification is limited to their external surfaces [76,79,80]. Surfactant-modified clays can be applied for the removal of heavy metal from water through ion exchange, precipitation-dissolution, and counter-ion binding mechanisms [8,81]. They can also be used to sorb metal cations and form complexes when their surfaces have negatively charged sites or there is a replacement of weakly held counter-ions in the solution [9].

### 5. Use of Surfactant-Modified Zeolites and Clays

A general model of the modification of a solid surface via the sorption of an ionic surfactant is shown in Figure 3. When low surfactant concentrations are applied onto the zeolite surface, surfactant molecules exist as monomers that are exchanged with the exchangeable cations of the zeolite until a hemimicelle (or monolayer) of surfactant molecules is established at its external surface. At concentrations above the CMC, the surfactant molecules will form an admicelle (or bilayer) attached to the external surface of the zeolite [82,83].



**Figure 3.** Surfactant modification of a solid surface forming a positive monolayer (hemimicelle) and a positive bilayer (admicelle) on a solid surface [84].

The application to zeolites and clays of cationic surfactants with long alkyl chains, quaternary ammonium groups, and a counter-anion changes their external charge from negative to positive, mainly by cationic exchange and hydrophobic interactions. Chutia et al. [85] indicated that the new positive surface can be balanced by anionic counter-ions, which increase the affinity of zeolites to sorb anionic contaminants [72,80,83,86]. Li et al. [87] suggested that the use of cationic surfactants with chloride ( $\text{Cl}^-$ ) and bromide ( $\text{Br}^-$ ) counter-ions in the treatment of a zeolite originates complete bilayer formations, whereas surfactants containing the hydrogen sulfate ion ( $\text{HSO}_4$ ) cause less than full bilayer formations.

### 5.1. Surfactant-Modified Zeolites

Zeolites have been widely used for their adsorptive properties and low cost. However, zeolites possess a net negative structural charge resulting from the isomorphic substitution of cations in the crystal lattice that gives zeolites little or no affinity for anions. The modification of clinoptilolite using hexadecyl trimethyl ammonium bromide (HDTMA-Br) and octadecyl trimethyl ammonium bromide (ODTMA-Br) surfactants increased its anionic sorption capacity, allowing for the removal of chromates and iodide from aqueous solutions [88]. Both surfactants altered the zeolite surface, although the ODTMA-clinoptilolite exhibited greater chromate and iodide sorption capacity (2.27 and 4.02 mg/g, respectively) than the HDTMA-clinoptilolite (2.18 and 3.37 mg/g, respectively). Leyva-Ramos et al. [83] indicated that HDTMA-clinoptilolite can sorb up to 22 times more Cr(VI) than an untreated zeolite. Other contaminants removed using HDTMA-Br clinoptilolite are selenates and sulfates [84].

Mordenite showed a considerable affinity for Cr(VI) species after treatment with HDTMA- $\text{HSO}_4$  and ethyl hexadecyl dimethyl ammonium (EHDDMA). The unmodified zeolite had no affinity for such species, whereas removal of up to 67% and 93% was achieved using HDTMA- $\text{HSO}_4$  and EHDDMA mordenites, respectively [89]. The modification of mordenite and clinoptilolite using HDTMA-Br notably increases their As(V) removal efficiency from 52% and 20% up to 92% and 86%, respectively [85]. Natural clinoptilolite and chabazite, which have no affinity for Cr(VI), removed up to 1.8 and 2.0 mg/g, respectively, after modification with hexadecyl pyridinium bromide (HDPB). The highest adsorption capacity for modified clinoptilolite occurs at a pH range of 3.0 to 5.0; for modified chabazite, it occurs at a pH range of 3.0 to 11.0 [90]. The use of HDTMA-Br also increases the Cr(VI) adsorption capacity of the zeolites, especially for clinoptilolite in a pH range of 3.0 to 11.0 [91]. Cationic surfactants can also enhance the metal affinity of synthesised zeolites; for instance, HDTMA-Br laumontite was successfully tested for the removal of chromate, molybdate, and tungstate [72]. Yusof and Malek [92] reported the adsorption of Cr(VI) and As(V) using a zeolite Y synthesised from rice husk ash that had been modified using HDTMA-Br. The unmodified zeolite Y has little or no affinity for these metal species.

Comparing the application of the cationic surfactant Uniquat QAC-50 and the amphoteric surfactant Miranol-C2MSF for the removal of heavy metals from mine waters, it was found that the amphoteric-treated zeolite adsorbed up to 85% of the initial metal concentration, with a preference for chromates. However, such efficiency was lower than that observed using the cationic surfactant, especially for copper (Cu) and iron (Fe) [93]. Cr(VI) has been successfully removed using natrolite modified with the gemini surfactant *N,N,N,N',N',N'*-hexamethyl-1,9-nonanediammonium dibromide (HMNA- $\text{Br}_2$ ), with an efficiency ranging from 70% to 90%, depending on the pH of the solution [94].

### 5.2. Surfactant-Modified Clays

The application of a cationic surfactant on clays causes the change of the clay surface from hydrophilic to hydrophobic and from negatively to positively charged, due to the sorption of the molecule onto its external surface and into the interlayer spacing. Therefore, the surfactant-modified clay exhibits an anion exchange capacity [95]. Quaternary ammonium cations are the most frequently used surfactants for preparing clays, with an excellent hydrophobicity and great efficiency for metals and organic contaminants recovery, although the mechanisms for surfactant adsorption on the clay

surface are not clear. Krishna et al. [96] indicated that the application of HDTMA-Br on montmorillonite constricts the pore channels of the clay due to the attachment of the surfactant moieties to its surface. More recently, Li and Gallus [80] indicated that when a cationic surfactant is applied on kaolinite, the surfactant sorption is limited to the external surfaces, although the surfactant molecules sorb on the surfaces as well as in interlayer spaces when using smectites. The metal speciation also determines the removal mechanisms acting on a surfactant-modified clay. In the case of the sorption of arsenate and arsenite on HDTMA-kaolinite, anion exchange is considered the dominant mechanism for arsenate sorption, whereas surface complexation could also be accounted for arsenite sorption [97].

The modification of kaolinite using cationic surfactants changes the negative basal surface charge of the clay and enhances the adsorptive capacity for contaminants; for example, HDTMA-Cl kaolinites are able to adsorb up to 10 times more Cr(VI) than the natural form by significantly increasing both the specific surface area and the CEC of the clay [79]. Sodium dodecyl sulfate (SDS) montmorillonites can remove copper, mercury, and zinc from water [9,81]. HDTMA-Br montmorillonites achieved a high Cr(VI) affinity at a low pH [96], and a cetyl pyridinium bromide (CPB) montmorillonite removed Cr(VI) over a pH range from 3.0 to 6.0 with an approximated efficiency of 98% [98]. Palygorskite and sepiolite modified with HDTMA-Br showed a Cr(VI) sorption capacity of 42 and 34 mmol/kg for HDTMA-palygorskite and HDTMA-sepiolite, respectively. Using dodecyl trimethyl ammonium bromide (DDTMA-Br) in the treatment of palygorskite resulted in a chromate sorption capacity of 17 mmol/kg [99]. Cetyl pyridinium bromide (CPB)-modified bentonite adsorbed up to 0.7 and 1.4 mmol/g of Cr(VI) and Mo(VI), respectively, from water [100].

Surfactant-modified clays also allow for the simultaneous removal of organic contaminants and heavy metals. Montmorillonite modified with carboxy decyl triethyl ammonium bromide (CDTEA-Br) showed promise for the adsorption of both Pb(II) and chlorobenzene [101]. Kaolin modified with cetyl trimethyl ammonium (CTAB) removes both Cu(II) and organic pollutants, such as *o*-xylene and phenol [95]. The modification notably increases the adsorption capacity of the natural kaolin (19.2 and 5.1 mg/g for Cu(II) and phenol, respectively) up to 38.5 mg/g for Cu(II), 91.5 mg/g for phenol, and 51.8 mg/g for *o*-xylene. Using the zwitterionic surfactant hexadecyldimethyl (3-sulphonatopropyl) ammonium on montmorillonite allows the removal of Cu(II) and phenol; however, the comparison with HDTMA-Br montmorillonite suggests that the adsorption capacity of the clay, both for organics and metals, is slightly higher using a cationic surfactant [81]. However, the application of quaternary ammonium cations on montmorillonites decreased the metal adsorption capacity of the clay since the interlayer quaternary ammonium cations are not readily exchangeable or the hydrophobic interlayer environment of the montmorillonite restrains the adsorption toward the hydrated metal cation [81].

Mixtures of surfactants have a more complex behaviour than a single surfactant. They have unique properties with industrial applications, but these properties will change depending on the surfactants used and the concentration in which they are present [102]. The mixtures can be unstable and precipitate, although several studies indicate that combination is possible. Mixed surfactants applied on zeolites and clay surfaces could be considered as pseudo-zwitterionic molecules, but this area requires more research for diverse contaminants [103–108].

## 6. Conclusions

Adsorption is still an important treatment for water contaminated with heavy metals, and therefore the research into new materials with better adsorption properties is relevant for increasing the efficiency of the process. Zeolites and clays are low-cost materials broadly applied in adsorption processes, hence the research on the development of these materials requires continued investigation and testing, for example regarding the application of surfactants to promote the sorption of metal anions and organic pollutants while retaining the sorption capacity for metal cations. Moreover, the consideration of such promising adsorbents for water and wastewater treatments, combined with the monitoring and modelling of water quality, offers an integral solution for the sustainable management of water resources in order to determine the level of contaminants and the decisions required. Further

research on these topics is needed to better understand the mechanisms for metal removal using modified materials and the impact on the environment.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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