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Arsenic removal by layered double hydroxides (LDH): A mini review

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ABSTRACT

The World Health Organization (WHO) has classified arsenic as the most toxic chemical with carcinogenic effects and WHO has established a value of 10 μg/L arsenic as the maximum contaminant level for arsenic in potable water. High selectivity and high removal capacity are one of the most important factors when choosing an adsorbent. Hydrotalcite and hydrotalcite-like compounds have received much attention for the remediation of As-contaminated waters. The most interesting properties of layered double hydroxides (LDHs) include a large surface area and high anion exchange capacity. In this study, the importance of double-layered hydroxides, which has gained importance with the increasing environmental pollution recently, in removing oxyanion pollutants from water has been emphasized. This review article focuses on LDH synthesis and characterization techniques and arsenic removal potential using LDHs, as well as the safe consumability of water obtained after removal. In this review, various factors such as pH, dosage, and temperature that affect oxyanion adsorption on LDHs are discussed. The reusability of LDHs has been discussed. In addition, it has been investigated whether double-layer hydroxides (FeHT) could be used successfully in the removal of arsenic from polluted waters. It was also discussed whether the adsorbent used was safe for health.

Key words: Fe-hydrotalcite, layered double hydroxides (LDHs), maximum adsorption capacity, safe drinking water

HIGHLIGHTS

- Many methods have been developed so far to remove toxic arsenic from water.
- Adsorption is the most preferred method. It is essential to choose the most effective and economical adsorbent for adsorption.
- Efficiency is very important, but while we want to remove toxic material from the waters, other harmful substances should not harm the waters and people.

1. INTRODUCTION

Groundwater is the main source of water for most places in the world. The release of toxic chemicals into the soil and the contamination of underground water resources threaten both human life and the environment (Türk 2016). There are high levels of oxyanions (arsenite, arsenate, selenite, selenate, chromate, phosphate, borate, nitrate, fluorite, chloride, etc.) in our environment that may be harmful to human health. Besides Turkey, countries such as Argentina, Bangladesh, Chile, China, Hungary, India, Mexico, Taiwan and Vietnam are among the main countries with arsenic problems in groundwater. For drinking and using water, without any harm to human and animal health, it must be purified from harmful substances at certain rates. For this reason, some norms have been developed by the World Health Organization (WHO) for water to be healthy. The European Union (EU) and the WHO have reduced the limit value for arsenic in drinking water from 50 to 10 μg/L. Countries such as Japan, Germany, Australia, and the USA have reduced the arsenic concentration in drinking water to 10 μg/L. In countries such as Bangladesh, Egypt, Indonesia, Saudi Arabia, India, and Chile, the limit value for arsenic is 50 μg/L. There is an excessive amount of arsenic in the groundwater resources of these countries. The limit level in drinking water in Turkey (TS 266 1984) was 50 μg/L, but the highest amount of arsenic in waters used for humanitarian purposes was reduced to 10 μg/L by the Ministry of Health. Table 1 shows the maximum permissible arsenic levels in drinking water (Rahman 2002).

To remove these impurities, precipitation (Clara & Magalhães 2002; Türk & Alp 2010; Türk *et al.* 2010, 2020a, 2020b; Türk 2017), coagulation (Wickramasinghe *et al.* 2004), reverse osmosis (Mondal *et al.* 2006; Chan &

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Table 1 | Maximum permissible arsenic levels in drinking water (Rahman 2002)

Country	μg/L
WHO	10
USEPA	10
Canada	25
Japan	10
European Union	10
Germany	10
Turkey	10
Bangladesh	50
Egypt	50
Indonesia	50
India	50

Dudeney 2008), ion exchange (Korngold *et al.* 2001; Anirudhan *et al.* 2007), and adsorption (Mohan & Pittman 2007) methods are used. Among these techniques, adsorption is the most widely used method with its simplicity and regeneration potential (Zhang *et al.* 2004). Adsorption is an effective technology if the adsorbent is cheap and easy to use.

Since environmental pollution has been an important issue recently, anionic clays known as layered double hydroxides (LDHs) have attracted great attention. LDH can occur naturally or can be synthesized simply and economically. The structure of an LDH is balanced by layers of positively charged brucite and anions in the inner region. LDH has very weak inner layer bonds. The most important feature of LDH is its high surface area and high anion exchange capacity (Ulibarri *et al.* 2001; Bruna *et al.* 2006). LDHs have uses such as catalyst, photochemistry, electrochemistry, polymerization, magnetization, medical science, and environmental applications (Li & Duan 2005; Centi & Perathoner 2008).

Various studies have been conducted on the arsenic adsorption of LDH (Nishida *et al.* 2004; Chang *et al.* 2007). For arsenic removal, LDH containing Mg, Fe, Al, and Co cations are mostly used due to their abundance. MgAl LDHs have been used to remove arsenic from water. Recently, there have been studies on the use of sjögrenite structure, produced by replacing Al^{+3} in the hydrotalcite (HT) structure with Fe^{+3}, in the removal of arsenite (As(III)) and arsenate (As(V)) (Türk & Alp 2010, 2012; Türk *et al.* 2020a, 2020b; Boyraz *et al.* 2024). Studies show that Fe and Fe minerals are effective adsorbents in removing arsenic due to the high affinity of iron toward arsenic (Boyraz *et al.* 2024). It is important that the pH is between 11.5 and 12 during MgAl LDH synthesis. If pH > 12, Al^{3+} ions dissolve. Moreover, for As(V) adsorption studies using HTs as adsorbents, the chemical dissolution of structural components in HTs such as aluminum (Al) and magnesium (Mg) has been investigated in only very few studies. But now, the presence of Al in human organisms is shown to be one of the causes of Alzheimer's disease (Dias & Fontes 2020). Therefore, in this review, MgFe LDH (FeHT), obtained by modifying Fe metal instead of Al metal in HTs, was examined as an LDH. Iron-based HTs have high selectivity against arsenic and are not harmful to human health. This review article focuses on LDH synthesis and characterization techniques and arsenic removal potential using LDHs. In addition, various factors affecting oxyanion adsorption in LDHs, such as pH, dosage, and temperature, are discussed in this review.

1.1. Arsenic species and sources

Arsenic occurs naturally in rocks, soil, air, water, plants, and animals. They are spread to the environment by natural activities such as volcanic activities, erosion, and forest fires or anthropogenically. Arsenic is also released into the environment because of the combustion of fossil fuels, paper production, cement production, and mining activities.

The earth's crust is a natural source of arsenic. There are more than 200 arsenic minerals, the most common of which is arsenopyrite. About one-third of the arsenic found on earth is of natural origin. Volcanic activities are the most important natural resources. Organic arsenic compounds containing carbon are found in sea creatures. Arsenopyrite, orpiment, loellingite, and realgar minerals are natural sources of arsenic. The physical properties of minerals, which are important sources of arsenic, are shown in Table 2.

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Table 2 | Physical properties of important arsenic-bearing minerals (Habashi 1995)

Mineral	Formula	Mohs hardness	Density (g/cm^3)	Crystal system	Arsenic content (%)
Arsenopyrite	FeAsS	5.5–6	5.9–6.2	Orthorhombic	46
Loellingite	$FeAs_2$	5–5.5	7.1–7.3	Orthorhombic	73
Orpiment	As_2S_3	1.5–2	3.4–3.5	Monoclinal	61
Realgar	AsS	1.5–2	3.4–3.6	Monoclinal	70
Native arsenic	As	3–4	5.6–5.8	Trigonal	90–100

Anthropogenic sources of arsenic are mainly pesticides, plant desiccants, poultry feed additives and pesticides, coal, petroleum, mine smelters, and detergents. Organic compounds such as arsenic trioxide (white arsenic), sodium arsenite together with monosodium methanearsonate, disodium methanearsonate, alkali salts such as potassium, and lead arsenite are frequently used as herbicides. Copper acetoarsenite (Paris green) and lead arsenate are strong insecticides (insecticide). Arsenic compounds are also used in the structure of some detergents, paint pigments, leather and paper industry (potassium acid arsenate), ceramics, glass, and rubber manufacturing (Bilgin & Baklaya 2003).

In addition, ash, mines, hydrocarbons, and metallurgical processes that are formed as a result of combustion in the facilities can also be shown as arsenic sources. Mine tailings and wastewater are potential sources of pollution that contain high concentrations of arsenic. Arsenic in mine wastes is found in various forms such as arsenopyrite (FeAsS) and arsenic-rich pyrite (FeS_2). Arsenic-containing minerals are found as co-minerals in many mineralization processes. Considering the mining activities in our country, some gold, silver, copper deposits, and boron deposits are important in terms of arsenic pollution. In addition to smelting and base metal refinery facilities, thermal power generation stations are the main sources of anthropogenic arsenic. Arsenic is also released during coal operation and combustion. Mining activities result in high concentrations of arsenic from the oxidized sulfide minerals. This results in high concentrations of arsenic in surface and groundwater (Duker *et al.* 2005). The cycle of arsenic in nature is shown schematically in Figure 1 (Shaid *et al.* 2018).

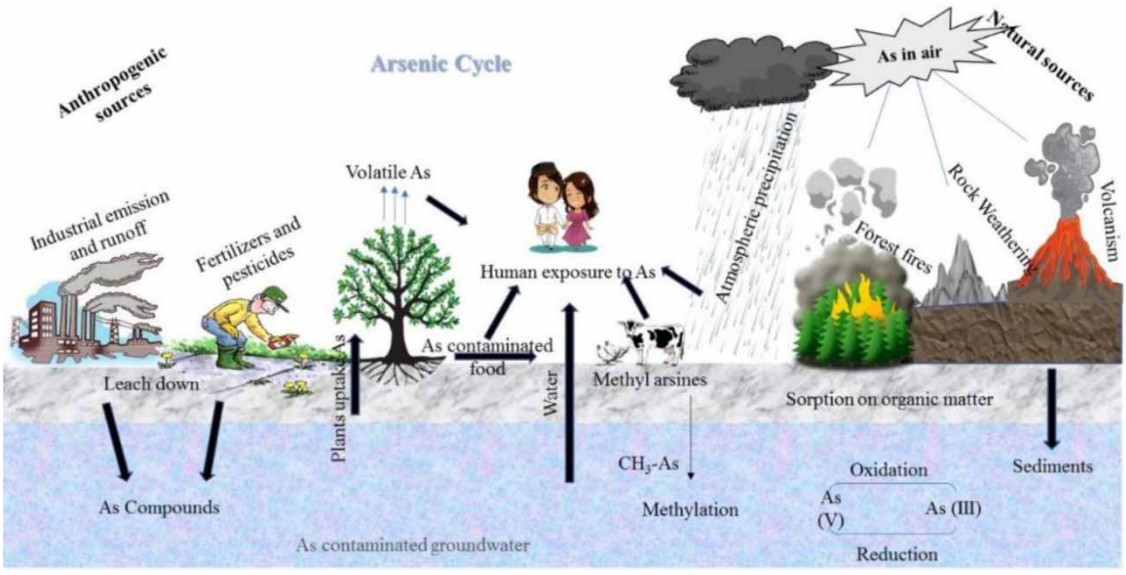


Figure 1 | Arsenic cycle in the environment as a schematic diagram (Shaid *et al.* 2018).

Eh and pH are important factors determining the dominant species in solution. In Figure 2, the Eh–pH diagram for arsenic is shown. Arsenic exists in the environment in some oxidation states (+3, −3, 0, +5). In natural waters, arsenic is mostly found in inorganic form with (+3) or (+5) valence states. Arsenate has four types such as H_3AsO_4, $H_2AsO_4^-$, $HAsO_4^{2-}$, and AsO_4^{3-}, and arsenite has four types such as H_3AsO_3, $H_2AsO_3^-$, $HAsO_3^{2-}$, and AsO_3^{3-}. In surface waters under oxidizing conditions, As(V) is dominant. Under reducing conditions, As(III) is

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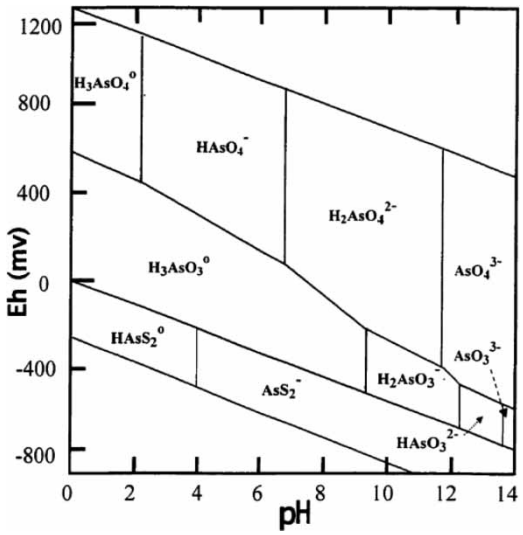


Figure 2 | Species diagram of arsenic according to Eh–pH (25 °C) (Fujimoto 2001).

stable. At neutral pH, $H_2AsO_4^-$ and $HAsO_4^{2-}$ for arsenate and uncharged H_3AsO_3 for arsenite are the dominant species (Figure 3) (Fujimoto 2001).

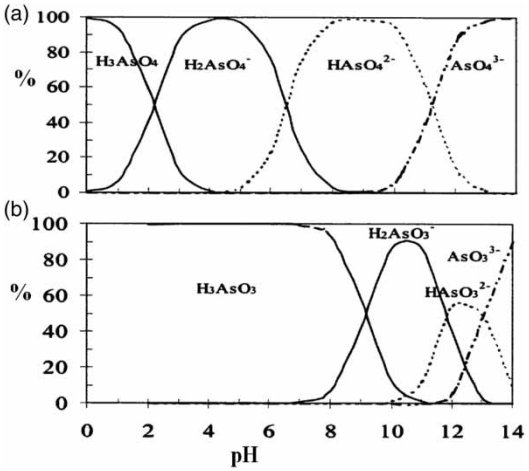


Figure 3 | Change of arsenate (a) and arsenite (b) species depending on pH (Bang 2003).

Organic species of arsenic are methyl arsenic acid, dimethyl arsenic acid, trimethyl arsine oxide, methyl arsine, dimethyl arsine, trimethyl arsine, arsanilic acid, triparsamide, alkylchlorarsine, and dialkylchloroarsine (WHO 1981). Inorganic and organic arsenic species are shown in Table 3.

1.2. Arsenic toxicity and effect on human health

The toxicity of arsenic depends on its chemical form. Inorganic arsenic combines with sulfhydryl groups inside proteins and accumulates in the human body. Arsenite is more toxic, as arsenite is more likely to interact with proteins than arsenate. The toxicity of inorganic arsenic can be reduced by methylation. In the methylation process, inorganic arsenic is converted to the less toxic methyl arsenic and excreted in the urine (Fujimoto 2001). Aqueous ferrous oxide can be used as an antidote in arsenic poisoning (Reddy 2004). Biological monitoring of arsenic is essential for the identification of acute or chronic arsenic poisoning. Arsenic is excreted mainly in the urine. Total arsenic concentration in urine is usually an indicator of recent arsenic poisoning. The half-life of inorganic arsenic in humans is 4 days. Since the half-life of absorbed organic and inorganic arsenic in blood is very short, blood is not a suitable biological material for chemical analysis in oral arsenic poisoning. Hair and nails are the areas with the highest arsenic concentration when compared to other tissues of the

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Table 3 | Some inorganic and organic arsenic compounds (WHO 1981)

Name	Formula
Inorganic arsenic, As(III)	
As(+3) oxide	As_2O_3
Arsenic acid	$HAsO_2$
Arsenic acid salts	$H_2AsO_3^-$ $H_2AsO_3^{2-}$ AsO_3^{3-}
As(+3) chloride	$AsCl_3$
As(+3) sulfur	As_2S_3
Inorganic arsenic, As(V)	
As(+5) oxide	As_2O_5
Arsenate acid	H_3AsO_4
Salts of arsenic acid	$H_2AsO_4^-$ $HAsO_4^{2-}$ AsO_4^{3-}
Organic arsenic compounds	
Methyl arsenic acid	$CH_3AsO(OH)_2$
Diethyl arsenic acid	$(C_2H_5)_2AsO(OH)$
Trimethyl arsine oxide	$(CH_3)_3AsO$
Methylarsine	CH_3AsH_2
Trimethylarsine	$(CH_3)_3As$
Dimethylarsine	$(CH_3)_2AsH$

body. Hair is mostly used to measure inorganic arsenic poisoning (Yağmur & Hancı 2002). Arsenic is toxic and carcinogenic. The effects of arsenic exposure on the human body are shown in Figure 4. Long-term use of drinking water with high concentrations of arsenic can lead to many health problems. The most common of these are pigmentation changes and skin and lung cancers. The presence of arsenic, which is found in nature in

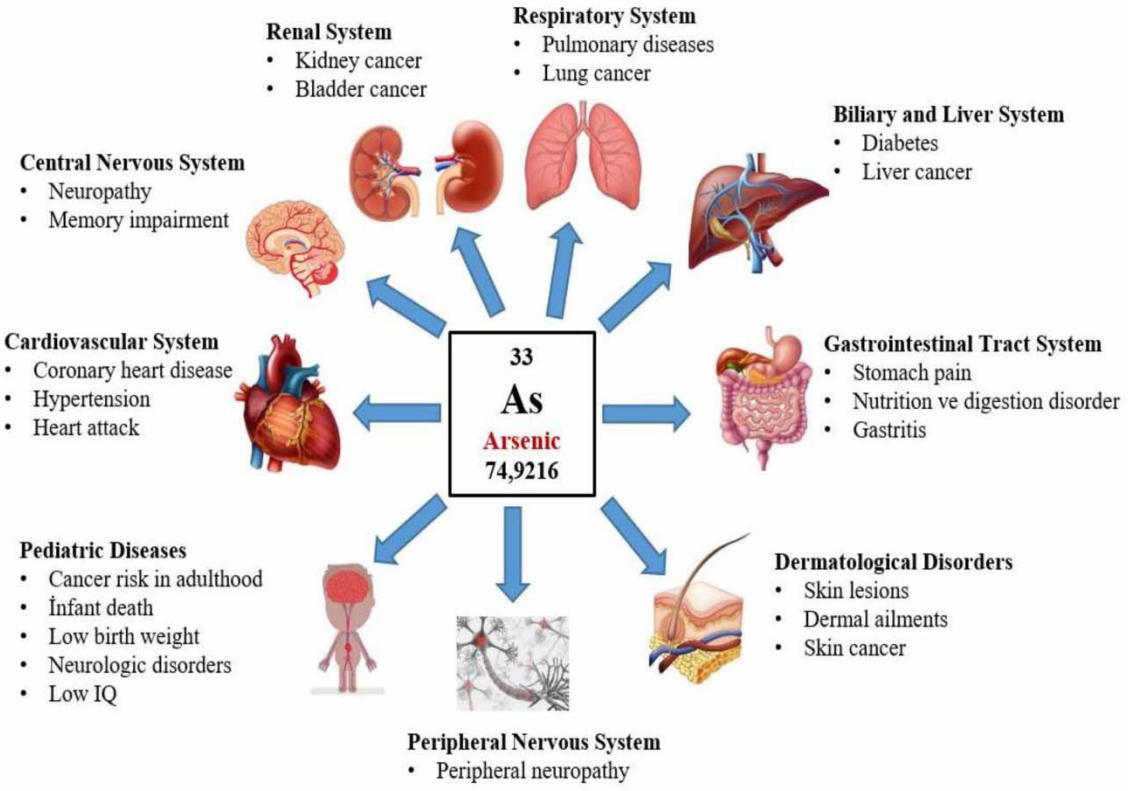


Figure 4 | Effects of arsenic exposure on the human body.

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combination with oxygen, sulfur, and chlorine, in foods and water damages the nervous system, gastrointestinal system, and skin tissues. Inhalation of large amounts causes wounds in the lungs and respiratory tract. It can reach lung cancer. Exposure to low levels of arsenic may cause nausea, vomiting and diarrhea, and tingling in the hands and feet, while warts may appear on the skin over a longer period. Ingestion through food can lead to bladder, kidney, liver, and lung cancer. Arsenic can also be distributed to soft tissues such as kidney, liver, heart, and brain. Chronic arsenic exposure can cause specific dermal effects (Duker *et al.* 2005). The health effects of arsenic are thought to depend on its dosage and exposure time. Non-specific gastrointestinal effects such as diarrhea and cramping; Exposure to high dosages of arsenic (0.04 mg/kg/day) after weeks or months may occur when chronic arsenic exposure may cause specific dermal effects. Diffuse or blotchy hyperpigmentation with ingestion of arsenic at high dosages (0.04 mg/kg/day or 40 μg/kg/day) for 6 months to 3 years or low doses (0.01 mg/kg/day) for 5–15 years seen has been reported. When inorganic arsenic is taken orally in high doses (>60 mg/L), it can result in death. Most affected organs are the digestive system, circulatory system, liver, kidney, heart, nervous system, and other sensitive tissues (Duker *et al.* 2005). Long-term exposure to inorganic arsenic compounds may cause discomfort in the upper respiratory tract, such as perforation of the nasal chambers, laryngitis, pharyngitis, and bronchitis. These ailments are common among workers in the smelter industry. Generally, this type of upper respiratory tract disease occurs when airborne inorganic-based trivalent arsenic is several hundred $\mu g/m^3$ (WHO 1981). The carcinogenic potential of inorganic arsenic compounds that can be found in the smelter industry has been proven by epidemiological studies. In one study, it was stated that there is a linear relationship between the cumulative amount of arsenic exposed and the risk of lung cancer. This may occur when several grams of arsenic are ingested over a 10-year period in drinking water and ingestion of medication (WHO 1981). An important issue is the increased risk of lung cancer with inhalation of inorganic arsenic. This effect is especially severe for workers exposed to arsenic in mines and chemical factories. However, this effect is also seen in those living near mines and chemical factories. Inorganic arsenic compounds can cause chronic effects in the human nervous system. As a result of these effects, sensory loss and brain abnormalities may occur. Moderate sensory loss has been reported in children living in the vicinity of coal-burning power plants, where significant amounts of arsenic are emitted. Since inorganic As(III) compounds are effective on the blood system, they have been used in the treatment of various types of leukemia for the last 20 or 30 years. The daily dose used for this is a few milligrams. Treatment with organic arsenic compounds such as triparsamide has side effects, mainly in the central nervous system, such as weakening of brain and vision functions (WHO 1981) (Figure 4).

1.3. Boron ore and arsenic contamination

The average boron concentration in the earth's crust is 10 mg/L. Boron is found in natural and human sources in water resources. High boron levels in surface and groundwater cause environmental problems. Boron is not carcinogenic (WHO 1993). There are high amounts of boron minerals in some areas of Turkey. The highest boron concentrations are found in sedimentary rocks (Çöl & Çöl 2003). Boron is found in the environment in the form of various compounds. It is generally found in some volcanic spring waters as orthoboric acid, forming borate minerals such as borax ($Na_2B_4O_7.10H_2O$), colemanite ($Ca_2B_8O_{11}.5H_2O$) and ulexite ($NaCaB_5O_9.8H_2O$). Boron minerals contain boron oxide (B_2O_3) in different proportions. Boron minerals that are commonly found in Turkey are tincal, colemanite, and ulexite. Important boron deposits in the world are Turkey, USA, and Russia. Turkey ranks first in the world's total boron reserves with a share of 72%. Almost all of the world's colemanite reserves are in Turkey. The colemanite enterprise with the highest reserve and production capacity in Turkey is located in the Emet-Hisarcık region of Kütahya. It has been stated that the colemanites of the region, which have a total reserve of 105 million tons, contain arsenic in the form of orpiment and realgar, unlike other boron deposits (Aydin *et al.* 2003). Extremely high arsenic pollution in groundwater has been observed in hydrothermal studies carried out in the area around the Kütahya-Hisarcık colemanite mine. Concentrations are between 0.474 and 7.754 mg/L and exceed the WHO (1993) drinking water standards (Çolak *et al.* 2003). It is thought that the As pollution in water resources in the district center, which has very important boron deposits in Turkey, may be related to arsenic minerals such as realgar (As_2S_2) and orpiment (As_2S_3) found in the surrounding colemanite (Boron) deposits. The ore itself, solid and liquid wastes resulting from ore enrichment, and solid and liquid wastes generated during the production of products such as boric acid can be listed as potential causes of pollution (Doğan & Doğan 2007). Drinking water in the region is obtained from groundwater. These waters can be displaced between the rock layers thousands of meters underground, and a small earthquake

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can affect the direction of the waters. Clean sources may contact layers containing harmful substances after ground movements. The contamination of groundwater with arsenic in the region is due to the dissolution of arsenic-loaded minerals such as realgar and orpiment (Çolak *et al.* 2003). Environmental arsenic exposure in the Kütahya province causes various arsenic-related skin diseases (Figure 5) (Doğan & Doğan 2007).



Figure 5 | Arsenic-related skin lesions on soles and palms.

2. LAYERED DOUBLE HYDROXIDE

2.1. Composition and structure of LDH

LDHs are naturally occurring anionic clays with the general formula $[M^{2+}_{1-x}\ M^{3+}_{x}(OH)_2]^{x+}\ A^{m-}_{x/m}.nH_2O$. M^{2+} and M^{3+} are two and three valent cations, respectively. The X value is equal to the $M^{3+}/(M^{2+}+M^{3+})$ molar ratio and takes values between $0.2 < x < 0.33$ (Yang *et al.* 2007). A is the m-valent anion in the inner layer region (Lazaridis *et al.* 2003; Ferreira *et al.* 2006; Tamura *et al.* 2006). M^{2+} (Mg^{2+}, Fe^{2+}, etc.) and M^{3+} (Al^{3+}, Fe^{3+}, etc.) ions have similar ionic diameters (Seron & Delorme 2008). The anion exchange capacity and charge density of LDH can be controlled by the ratio M^{2+}/M^{3+} (Ferreira *et al.* 2004). The structure of the LDH is shown in Figure 6. The basal cavity in Figure 6 is the total thickness of the brucite layer and the inner layer region. The octahedral units of M^{2+} or M^{3+} are dispersed at the corners to form unlimited layers. These layers are connected to each other by hydrogen bonds (Goh *et al.* 2008).

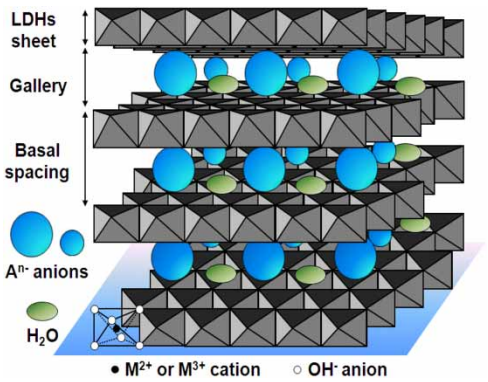


Figure 6 | Schematic representation of the structure of LDHs (Bi *et al.* 2014).

When $M^{2+} = Mg^{2+}$ and $M^{3+} = Al^{3+}$, HT with the general formula $[Mg_6Al^{3+}_2(CO_3)(OH)_{16}.4H_2O]$ is produced (Seida & Nakano 2000). It can be easily synthesized by co-precipitating a dilute aqueous solution of magnesium and aluminum. The crystal structure of the HT mineral is shown in Figure 7. HT contains layers of magnesium and aluminum hydroxide and is positively charged. The presence of anions between the layers is required for charge balance. These anions are commonly carbonates. Anions in the inner layer region between the brucite layer compensate for the net positive charge (Nishida *et al.* 2004). It can be modified by adding different ions to its structure (Palmer *et al.* 2008). For example, if synthesis is made with Fe^{3+} ions instead of Al^{3+} ions in the structure of the mineral, sjögrenite $[Mg_6Fe^{3+}_2(CO_3)(OH)_{16}.4H_2O]$ mineral is obtained (Frost & Erickson 2004; Choudhary *et al.* 2005). It is accepted that the presence of iron in drinking water does not harm human health. More precisely, this metal is not harmful to health at typical concentrations seen in water sources. It has been reported that in people who are hereditarily prone to hemochromatosis, excessive amounts of iron

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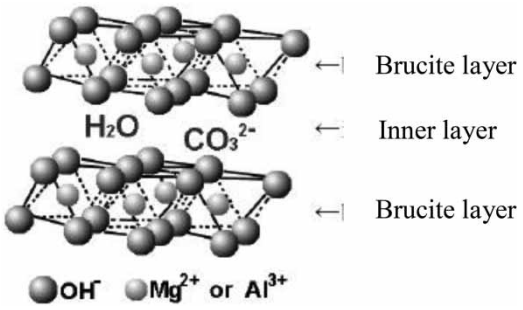


Figure 7 | $[Mg(II)_{1-x}Al(III)_x(OH)_2]^{x+}$ $[(CO_3^{2-})_{x/2}.yH_2O]^{x-}$ crystal structure of hydrotalcite (Nishida *et al.* 2004).

entering the body may accumulate in the body, and taking high doses of iron for a very long time may lead to liver, pancreas, and heart disorders. It is reported that approximately 1 in 200 people have such a risk, but this occurs much less frequently (AWWA 1999).

2.2. Synthesis methods of LDH

LDH can be easily synthesized in the laboratory. Some methods have been developed to prepare LDH.

2.2.1. Co-precipitation

The **simplest** and most common method is precipitation (Kang *et al.* 2005; Yang *et al.* 2005; Gillman 2006; Li & Zhou 2006). To form LDH with this method, the interaction of anion-containing M^{2+} and M^{3+} aqueous solutions are required. Co-precipitation at low saturation occurs by the slow addition of solution mixtures of two or three valent metal salts, desired inner layer anions, in the desired ratio, into a mixer containing an aqueous solution. The second basic solution is then added into the simultaneous mixer to achieve the desired pH (Figure 8). In high saturation co-precipitation, a mixture of salt solutions is added into the basic solution containing the inner layer anion. High saturation precipitation generally produces less crystalline material. The pH being between 11.5 and 12 during the synthesis process is important for HT production. If pH $>$ 12, Al^{3+} ions dissolve. If the pH is $<$11.5, the precipitation process cannot always be completed (Wright 2002).

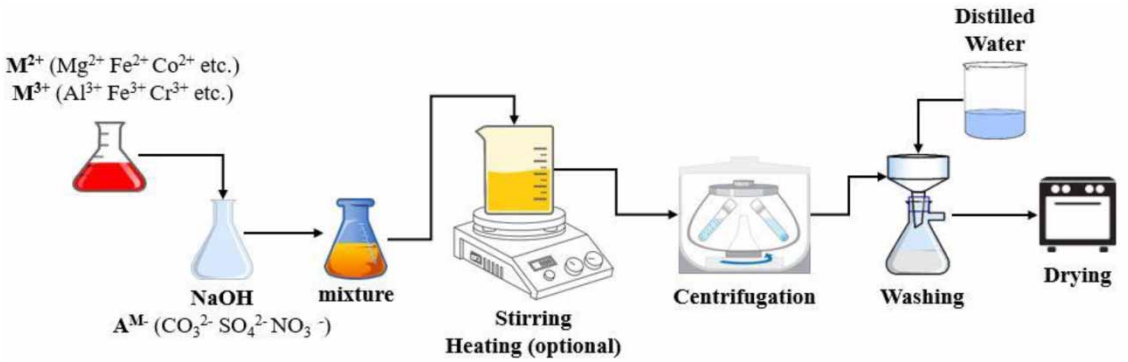


Figure 8 | Synthesis of hydrotalcites by the co-precipitation method.

2.2.2. Urea hydrolysis method

The urea hydrolysis method is also used in the synthesis of LDH. Urea ($CO(NH_2)_2$) has high water solubility and weak base properties. In this method, urea is used instead of NaOH used for precipitation. The advantage of the urea hydrolysis method is that it offers high saturation during the precipitation process. This is because the urea hydrolysis process takes place slowly (Liu *et al.* 2014). Also, prolonged hydrolysis results in the formation of carbonate anions that act as interlayer anions in basic medium (Costantino *et al.* 1998; Ogawa & Kaiho 2002; Oh *et al.* 2002; Pagano *et al.* 2003; Iyi *et al.* 2004).

In the synthesis process (Figure 9), primarily magnesium nitrate, aluminum nitrate, and urea are dissolved in deionized water. The molar ratio of Mg/Al is set to 2. The molar ratio of $[CO(NH_2)_2]/([Mg] + [Al])$ is set to 4. The

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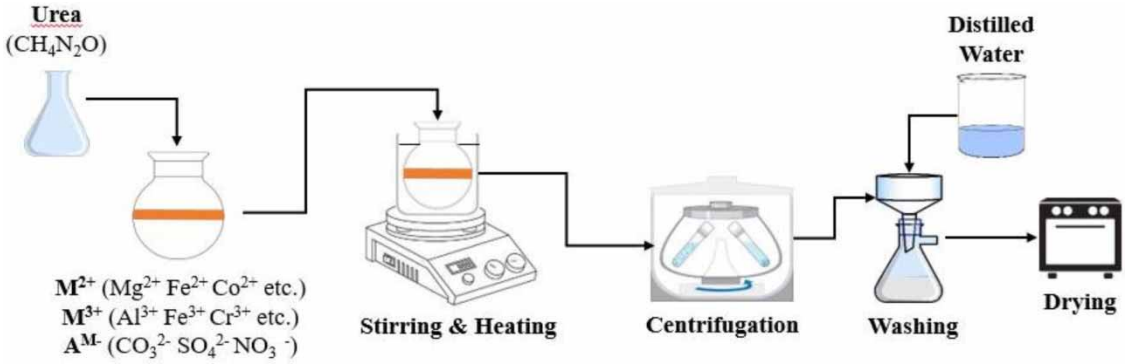


Figure 9 | Synthesis of hydrotalcites by the urea hydrolysis method.

solution containing metal ions and urea is mixed and heated. When the target temperature (90–120 °C) is reached, the temperature is fixed and mixed for 24–96 h. After mixing, the containers are allowed to cool at room temperature. Then, repeated washing with water is performed. After the washing process, the synthesis is completed by air drying (Hibino & Ohya 2009).

The urea method has been applied to prepare LDHs with relatively narrow particle size distribution compared to co-precipitation method (Pagano *et al.* 2003).

Costantino *et al.* carried out HT synthesis through urea hydrolysis. In this method, solid urea was added to aqueous solutions of magnesium chloride and aluminum chloride and clear homogeneous solutions were obtained. In addition, ZnAl-LDH and NiAl-LDH were successfully produced with this urea method (Costantino *et al.* 1998).

In a study, HT particles were prepared by two methods: urea hydrolysis and hydrothermal synthesis, and their particle sizes were compared. While particle sizes were larger at low temperatures, particle sizes were smaller at high temperatures. Urea hydrolysis is basically a form of the hydrothermal method in which the precipitant remains as urea. The advantage of using urea versus NaOH is that urea hydrolysis proceeds slowly, leading to a low degree of supersaturation during precipitation. In addition, the key advantage of using this method is that, unlike co-precipitated samples, it does not require repeated washings to get rid of alkali metal ions (Rao *et al.* 2005).

2.2.3. Sol–gel method

The sol–gel synthesis method is a low-cost and high reproducibility method. The homogeneity and purity of the obtained compounds are high. As a result of the synthesis, a nanoscale product with a high surface area is obtained. However, in the sol–gel method, lower crystallinity is observed (Dylan *et al.* 2021).

This technique consists of dissolving the desired metal sources, namely, M^{2+} and M^{3+} cations, in water at room temperature (Figure 10). The concentration of these cations can be adjusted. The solution is finally aged for several hours or days by gently heating at room temperature or up to 100 °C.

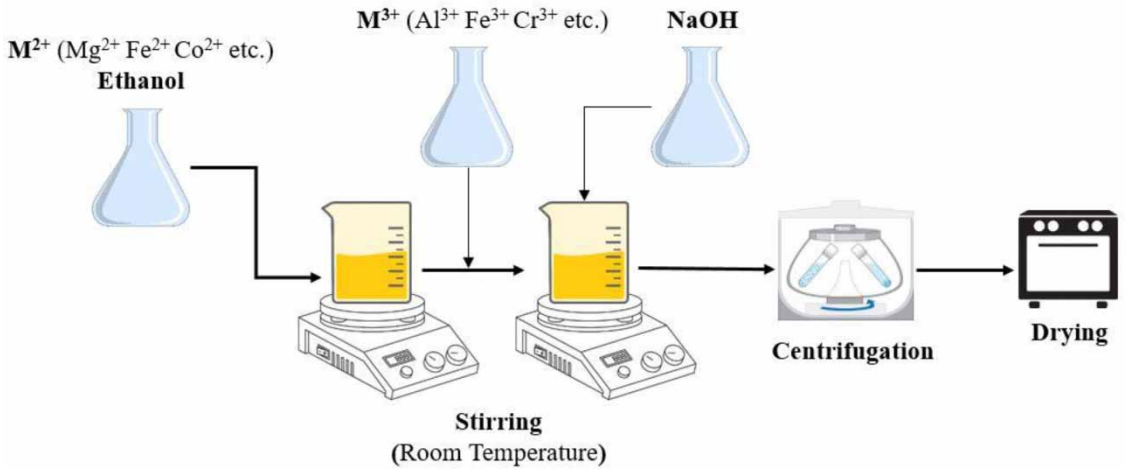


Figure 10 | Synthesis of hydrotalcites by the sol–gel method.

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Othman *et al.* (2006) compared the effect of hydrothermal treatment on HTs prepared by co-precipitation and sol–gel methods. They showed that in both cases the specific surface area and pore sizes increased significantly, with a stronger effect on sol–gel synthesized samples. In most cases, the sol–gel approach is preferred due to its simplicity and the high quality of the resulting materials (Othman *et al.* 2006; Smalenskaite *et al.* 2017).

2.2.4. Hydrothermal method

It is generally used when low-affinity organic anion species for LDH need to enter the inner layers and when sol–gel and precipitation methods cannot be applied (Bravo-Suárez *et al.* 2004; Dupin *et al.* 2004; Kovanda *et al.* 2005). This method is effective because only the desired organic anions can be formed on the substrate surface under hydrothermal conditions, since insoluble magnesium and aluminum hydroxides are used as the mother solution (Figure 11). It is possible to control the particle size and distribution with the hydrothermal synthesis method. Temperature is effective in the crystal structure of the material. The longer the hydrothermal treatment, the lower the water content. The hydrothermal process is applied at 120 °C for regular crystal structures and 100 °C for irregular crystal structures (Labajos *et al.* 1992). Higher crystallinity, crystal sizes, and purity of the samples are the advantages of this synthesis (Sharma *et al.* 2007). However, the energy required to heat the solution and the long synthesis time are disadvantages for the industrial application of hydrothermal treatment.

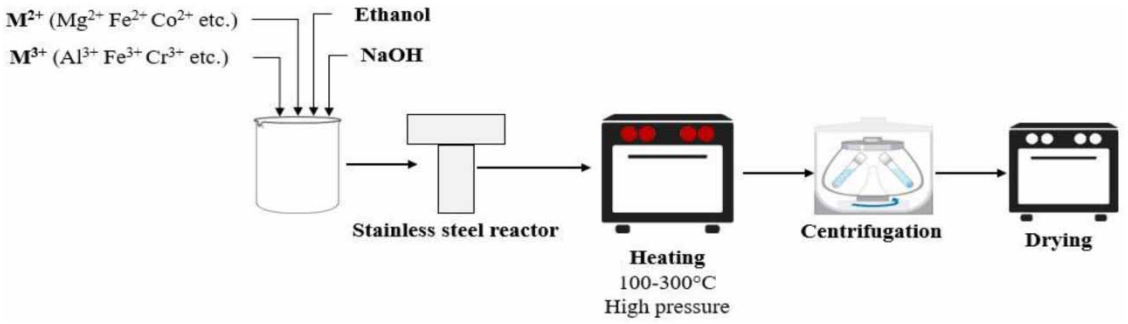


Figure 11 | Synthesis of hydrotalcites by hydrothermal method.

2.3. LDH characterization

There are many analytical techniques used to characterize LDH. X-ray diffractometry (XRD) and Fourier transform infrared (FTIR) spectroscopy are frequently used in routine analysis (Allada 1999; Nakahira *et al.* 2007). Other routine analyses are performed with thermogravimetry (TG), differential thermal analysis (DTA), scanning electron microscope (SEM), and transmitting electron microscope (TEM) (Tao *et al.* 2006; Rivera *et al.* 2008; Wang *et al.* 2008a, 2008b, 2008c). X-ray samples examined by XRD are obtained using powder samples. A typical XRD example of LDH is shown in Figure 12. Sharp basal reflections (001) at low 2θ values are consistent with cascading basal spaces. In addition, rather weak basal reflections were observed at higher 2θ values (Zhang *et al.* 2004; Carja *et al.* 2005).

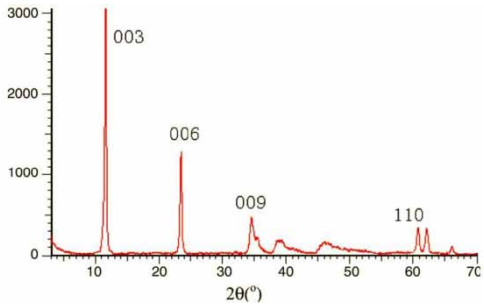


Figure 12 | MgAl-CO_3 LDH XRD view (Türk 2009).

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FTIR spectroscopy is another useful method for characterizing LDH. This method is used to determine the presence of charge balancing anions in the inner layer, the type of bonds formed with these anions, and how they are located. The most common inorganic anions, peaks in the IR spectra (Figure 13) 1,350–1,380 (ν_3), 850–880 (ν_2), and 670–690 (ν_4), indicate CO_3^{2-} (Kustrowski *et al.* 2004; Panda *et al.* 2008). While the morphology of LDH is observed by SEM and TEM, its thermal properties are commonly determined using TG and DTA. The SEM image is shown in Figure 14. LDH is shaped in a flat, smooth, hexagonal form and the crystals are overlapped. Other physicochemical methods include elemental analysis, surface area, and porosity analysis (Kang *et al.* 2005).

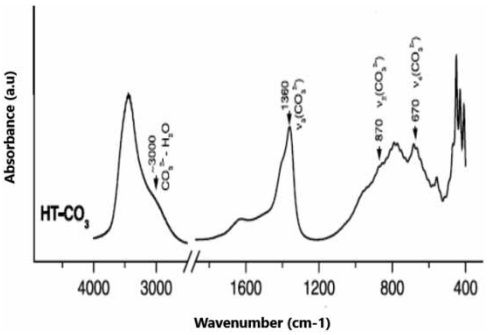


Figure 13 | Example of FTIR Spectra of LDH (Türk 2009).

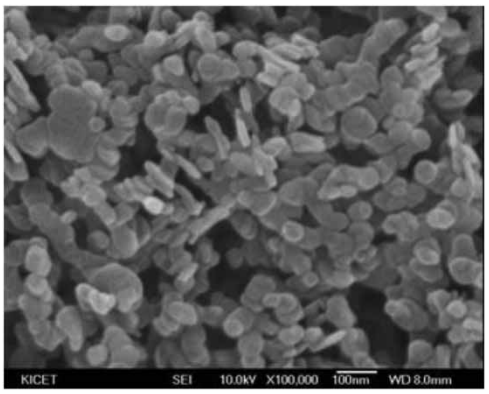


Figure 14 | SEM image of LDHs (Türk 2009).

2.4. Adsorption of arsenic oxyanions on LDH

LDH has a high surface area and high anion exchange capacity, as well as a flexible inner layer region that can be reached by various anion species. In general, there are two different mechanisms to removal of pollutants from the aqueous medium by LDH: surface adsorption and inner layer anion exchange (Mohan & Pittman 2007).

Various studies have been conducted on the oxyanion adsorption of LDH (Bhaumik *et al.* 2005). Arsenite (Manju & Anirudhan 2000; Ye *et al.* 2005; Yang *et al.* 2006), which has caused environmental pollution in recent years, arsenate (Lazaridis *et al.* 2002; Dousovai *et al.* 2003; Kiso *et al.* 2005; Liu *et al.* 2006; Chang *et al.* 2008), selenite (Das *et al.* 2002), selenate (Shahrivari 2005), chromate (Lazaridis *et al.* 2004), phosphate (Das *et al.* 2006; Frost *et al.* 2007), borate (Ay *et al.* 2007) and nitrate (Frost & Musumeci 2006; Hosni & Srasra 2007) can be removed from water with LDH, an anionic clay.

In recent years, intensive studies have been carried out on the removal of arsenic from solutions. These studies are basically the rehabilitation of wastewater (Singh *et al.* 1996; Navarro & Alguacil 2002; Solozhenkin *et al.* 2003) and the purification of drinking water (Kuriakose *et al.* 2004; Singh & Pant 2004; Lenoble *et al.* 2005; Guo *et al.* 2007; Zhang *et al.* 2008).

In research studies, generally in arsenic removal, flocculation-microfiltration (Han *et al.* 2002), precipitation (Clara & Magalhães 2002), membrane process (Shih 2005; Mondal *et al.* 2006; Chan & Dudeney 2008), ion

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exchange (Korngold *et al.* 2001; Anirudhan & Unnithan 2007), biological processes (Katsoyiannis & Zouboulis 2004), and adsorption (Mohan & Pittman 2007) are used. At the center of these studies is LDH, which has a very good anion exchange capacity (Gillman 2006; Chang *et al.* 2007). Various studies have shown that this LDH is effective in removing anions such as boron, chromate, selenium, and phosphate (Houri *et al.* 1999; Yang *et al.* 2005; Ferreira *et al.* 2006; Kuzawa *et al.* 2006; Ay *et al.* 2007). In arsenic adsorption on LDH, the effects of grain size (Yang *et al.* 2006), exchangeable anion type (Toraishi *et al.* 2002; Carja *et al.* 2008), and roasting temperature (Goh *et al.* 2008) on As adsorption are studied.

Some studies on arsenic adsorption on LDH in aqueous systems are summarized. Adsorption experiments were carried out at 25 °C, pH 3–12, 150–200 rpm mixing speeds, 0.04–10 g/L adsorbent dosages, 10–7,200 min adsorption times, and initial arsenic concentrations of 20–10,000 μg/L. The arsenic removal efficiency ranges from 60.6 to 99.02% for As(V) and 6.3 to 99% for As(III). Manju & Anirudhan (2000) used Mg-Al-CO_3 to remove As(III) from water. The effects of contact time, mixing speed, As(III) concentration, pH, and temperature were tested. They achived70.2% removal at pH 8.5 for an initial concentration of 150 mg/L. Chang *et al.* (2007) succeeded in reducing the arsenate concentration from 588 to 20.06 μg/L in 270 min using Mg-Fe-NO_3-HT. Nishida *et al.* (2004), using Mg-Fe-CO_3-HT ($Mg^{2+}/Fe^{3+} = 2$) in their study, the solution with an initial arsenic concentration of 10 mg/L was converted to 0.13 mg/L within 1 h and 0.098mg/L within 18 h. They managed to reduce it to mg/L. In the case where $Mg^{2+}/Fe^{3+} = 4$, the initial 10 mg/L solution was reduced to 2.48 mg/L within 1 h and to 0.95 mg/L within 18 h. From this, it is understood how important the Mg^{2+}/Fe^{3+} ratio is. As this ratio increases, Fe content and removal efficiency increase. In FeHT synthesis, the Mg/Fe ratio is generally adjusted to be 2–3 or 4. As this ratio decreases, the Fe content increases. Due to the high affinity of iron for arsenic, the arsenic removal efficiency increases. The anion exchange capacity and charge density of bilayer hydroxides are combined with the ratio M^{2+}/M^{3+}.

2.5. Factors affecting adsorption of arsenic oxyanions on LDH

Factors such as pH, adsorbent dosage, temperature, calcination, and particle size are the factors that affect LDH and oxyanion adsorption.

2.5.1. pH

The pH effect on adsorption with LDH depends on the type of oxyanions and LDH. The point of zero charge (pzc) is a very important value for adsorption measurements and surface characterization. The pzc plays an important role in understanding sorption of protons and hydroxyl groups, which depends on the acid–base properties of the surface. When $pH > pH_{pzc}$, the surface of LDH is negatively charged. Therefore, at higher pH values, oxyanions will be repelled by the LDH surface. In the case of $pH < pH_{pzc}$, the LDH surface is positively charged, and adsorption of oxyanions is desired in this case. At very low pH ($pH \leq pH_{pzc}$), the stability of the structures of LDHs is disrupted and adsorption is negatively affected. The base metals of LDH can go into solution at low pH. At higher pH ($pH \geq pH_{pzc}$), adsorption is negatively affected due to the competitive nature of OH^- ions (Szekeres *et al.* 2005; Türk *et al.* 2009). As the pH increases from 3 to 12 at an initial arsenate dosage of 300 μg/L, the As(V) concentration increases from 9 to 12.6 μg/L. The addition of FeHT to water at different pH values creates a buffering effect. This buffering effect can be explained by the amphoteric nature of FeHT. In a low pH environment, the pH of the solution increases in the presence of FeHT, while the dissolution of FeHT in a high pH environment causes the pH of the solution to decrease (Zhang *et al.* 2004).

2.5.2. Adsorbent dosage

Determining the appropriate adsorbent dosage for adsorbate removal in batch adsorption systems is important for cost-effectiveness of the application. In a study, 2.5 g/L MgAl LDH is required for a 92% yield at an initial concentration of 18 mg/L boron. When MgFe LDH is used as an adsorbent, boron removal increases linearly with increasing adsorbent dosage, but the maximum removal capacity is only 33% (Ferreira *et al.* 2006). In another study, it was observed that arsenate removal efficiency increased as the calcined-FeHT (cFeHT) dosage increased. Experiments were carried out with different adsorbent dosages to reduce the initial arsenate concentration of the sample (300 μg/L) to 10 μg/L, which is the limit value determined by the WHO. While the arsenate removal efficiency was 92% at the lowest adsorbent dosage value chosen in this study, it was 97.2% at the highest adsorbent dosage (Türk *et al.* 2020a, 2020b). This situation is observed similarly in other adsorbents.

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2.5.3. Temperature

When the temperature increases, the adsorption process is negatively affected. Pavan *et al.* (1999) showed that as the temperature decreases, the adsorption increase increases. This directional operating feature can be explained by the increase in the kinetic energy of the adsorbed species at temperature. The increase in temperature indicates the stability of the adsorbents. In simple adsorption processes, adsorbate removal decreases as exothermicity increases (Yang *et al.* 2005). In general, since simple adsorption reactions are exothermic, the removal of adsorbate decreases with increasing temperature. As(V) adsorption on calcined and non-calcined LDH is an exothermic process (Yang *et al.* 2005). The negative value of ΔH^{o} confirms the exothermic nature of adsorption (Das *et al.* 2002).

2.5.4. Calcination

Another major factor affecting the oxyanion adsorption with LDH is calcination during the synthesis of LDH (Chibwe & Jones 1989; Hibino & Tsunashima 1998). While LDH is thermally decomposed at 200 °C and below, it loses only surface water, while it loses carbon dioxide and water between 250 and 450 °C (Hibino *et al.* 1999). LDH calcined at 450–500 °C loses its layer structure, and active metal composites with large surface area, small crystal size, and high thermal stability are formed. This calcined product can be reproduced by rehydration. At temperatures above 900 °C, $MgAl_2O_4$ is formed and the lattice structure of LDH is completely degraded and cannot be reproduced (Palmer *et al.* 2008). In one study, the crystal extents of FeHT and calcined-FeHT (450 °C) were determined by XRD. The (003) and (006) peaks of synthesized FeHT on the XRD pattern are shown in Figure 15(a). Peak values (003) and (006) disappeared after calcination. In this case, it is understood that the FeHT structure is broken. The peaks seen in Figure 15(b) belong to Mg-Fe oxide (Türk *et al.* 2020a, 2020b). Oxyanion adsorption with calcined LDH is higher than with uncalcined LDH. One of the behavioral differences between these two products is the surface area of the uncalcined product. The other is that calcined LDH has less carbonate anions in its inner layer than uncalcined (Yang *et al.* 2005). In addition, high basicity is required if calcined LDH is to be used (Goh *et al.* 2008). Türk *et al.* (2020a, 2020b) analyzed the thermal behaviors of FeHT using TG-DSC in their study. As shown in Figure 16, a 39.5% mass loss of FeHT was observed in the range of 25–600 °C, where three endothermic peaks were detected. In recent studies on LDH, the effect of removing water, hydroxyl groups, and interlayer anions by calcination on the adsorption properties is

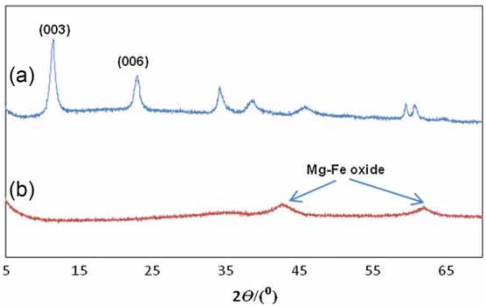


Figure 15 | XRD patterns of FeHT (a), CFeHT (b) (Türk *et al.* 2020a, 2020b).

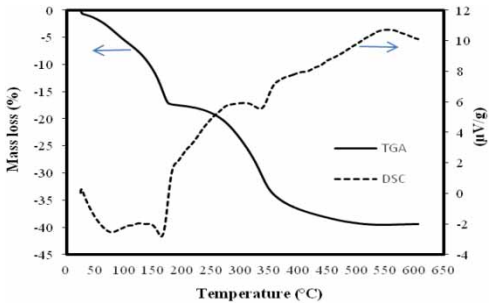


Figure 16 | TG-DSC curves for FeHT (Türk *et al.* 2020a, 2020b).

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investigated (Goh *et al.* 2008). Kovanda *et al.* (1999) tried to remove arsenate with calcined synthetic HT at 350–550 °C in their study. Experiments were carried out at an initial arsenate concentration of 0.002 mol/L, 20 °C, and natural pH. The maximum adsorption capacity was obtained with HT calcined at 450 °C. The adsorption of arsenic on LDH is highly affected by the calcination process during the synthesis of LDH. For example, when LDH is calcined, As(V) adsorption increases from 71.5 to 87.5% (LDH dosage 0.25 g/L) (Yang *et al.* 2005). Oxyanion adsorption with calcined LDH is higher than with uncalcined LDH. The most crucial difference between these two products is the surface area of the uncalcined product (Yang *et al.* 2005). The surface area of LDH ranges between 1.5 and 47 m^2/g, while the surface areas of calcined LDH vary between 26–198 m^2/g. Türk *et al.* (2020a, 2020b) studied As(V) removal from water with CFeHT. They reduced the initial As(V) concentration from 1,000 to 10 μg/L using 0.5 g/L adsorbent. As can be seen from these results, calcination positively affects the efficiency of arsenic removal.

2.5.5. Partial size

In most of the adsorption studies with LDH, particle sizes were not investigated. Particle characteristics are an important parameter in diffusion controlled models. Studies generally reported that the decrease in the particle size of LDH had a significant effect on the oxyanion adsorption rate. However, it is ineffective on oxyanion capacity (Yang *et al.* 2006). Experiments by Yang *et al.* (2006) showed that the adsorption capacity of As(V) is independent of LDH particle size. Smaller particle size provides higher adsorption rate and equilibrium is reached in a short time.

2.6. Different Ion effects

Na^+, Al^{3+}, Fe^{3+}, Mg^{2+}, NO_2^-, PO_4^{3-}, Cl^-, NO_3^-, and SO_4^{2-} can be found in water. The presence of Cl^- and NO_3^- in the solution during adsorption processes is not effective in removing arsenic (EPA 815 2000). While the presence of SO_4^{2-} in the solution does not affect arsenate removal between pH 9 and 11, it reduces arsenite removal at pH <7 (Wickramasinghe *et al.* 2004). The presence of PO_4^{3-} in the solution has little effect on arsenic adsorption (Guo *et al.* 2007). The presence of sodium in some underground spring waters may be due to ulexite ($NaCaB_5O_9.8H_2O$). Ulexite is a Na-Ca borate and causes an increase in Na content in groundwater (Gemici *et al.* 2008). Ca sources are colemanite ($Ca_2B_8O_{11}.5H_2O$), calcite ($CaCO_3$), and aragonite $(Ca,Sr,Pb,Zn)CO_3$. Mg concentration is found in Mg hydroboracite ($CaMgB_6O_{11}.6H_2O$). The maximum limit value of aluminum in water is 0.2 mg/L (EPA 816 2002). As another element, the drinking water standard for Fe is 200 μg/L (EPA 816 2002).

Türk & Alp (2012) also investigated the removal of As by FeHT at different boron concentrations, and in parallel with the literature findings, it was observed that the removal of boron was quite low. As a result of the tests, it is seen that As(V) and As(III) concentrations do not change as the boron concentration increases. With FeHT, the boron concentrations of 100 and 3,000 μg/L and the initial arsenate concentration of 300 μg/L were reduced to 4.1 and 3.2 μg/L, respectively. Considering all these evaluations, in this study, it has been determined that arsenic can be successfully purified from boron-containing waters by using FeHT as an adsorbent.

2.7. Desorption and regeneration

In plant-based applications, remediation systems, regeneration, and reuse of LDHs for arsenic removal are desirable. Recovering the initial properties of the sorbent in the desorption/regeneration process is essential for success. Surface properties of the adsorbent, ion concentration and desorption solution, pH, and duration of contact of arsenate with desorption are the parameters that have an impact on the desorption efficiency of arsenic (Türk *et al.* 2021). Schematic representation of arsenic sorption–desorption–regeneration is given in Figure 17. Alkaline and salt solutions or mixtures of solutions are used for the desorption of oxyanion-loaded LDH (Toraishi *et al.* 2002). Desorption of As(V) depends on the anion species and their concentration (Goh *et al.* 2008). The reproducibility of oxyanion-loaded LDH with carbonate and chloride solutions has been demonstrated by earlier studies. Lazaridis *et al.* (2004) showed that Cr(VI)-loaded non-calcined LDH was completely regenerated with Na_2CO_3 in 20 min. As(V)-loaded MgAl can be regenerated with LDH carbonate solutions. Türk *et al.* (2021) were able to maintain the regeneration efficiency of As(V) at 98.5% over four regeneration cycles using 0.5 M NaOH. They successfully regenerated and reused MgFeHT with an aqueous NaOH solution with a small loss of sorption efficiency. Adsorption is often used to remove arsenic in small-scale drinking water systems. Adsorbent change in adsorption technology accounts for approximately 80% of the total operating cost. If the

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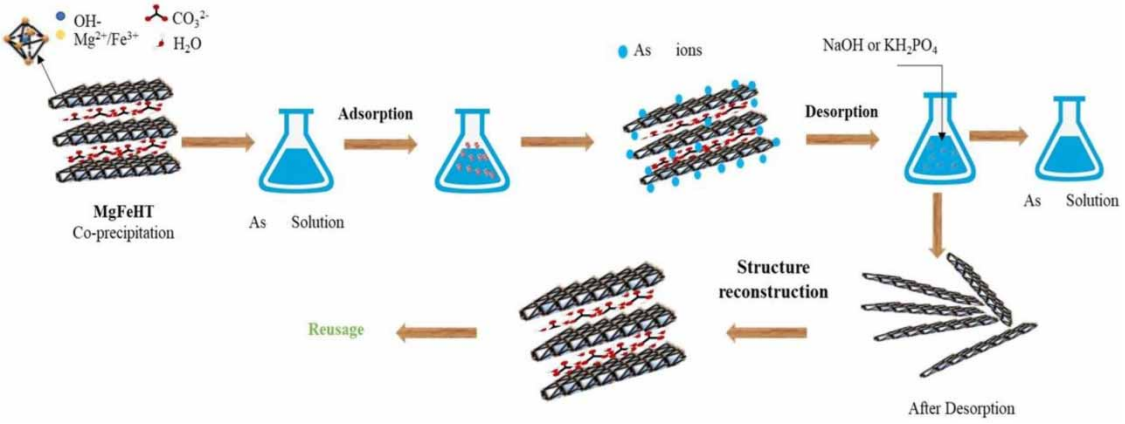


Figure 17 | Schematic representation of the arsenic sorption–desorption–regeneration.

Table 4 | Advantages and disadvantages of different synthesis methods on LDHs

Method	Advantages	Disadvantages
Co-precipitation	• Low cost • Facile procedure	• Large particles • Low surface area
Urea hydrolysis	• Large thin platelets	• Carbonate anions as interlayer anions
Sol–gel	• Low cost • High reproducibility and homogeneity • Purity of compounds • Small particle size • High surface area	• Lower crystallinity • Additional steps are required (sonication, microwave irradiation or hydrothermal treatment)
Hydrothermal	• Higher LDH crystallinity • Crystal sizes • Purity of samples • Possible to control the particle size and distribution	• High energy requirement • Long synthesis time

adsorbent can be regenerated and reused without loss, regeneration is a potential method to reduce the cost of the adsorptive medium (Türk *et al.* 2021).

3. CONCLUSION

Arsenic is very toxic. This review addresses the ability to remove toxic arsenic from water. While we do this, the adsorbents we use must be safe. There are a limited number of studies on this subject. LDHs show promise as adsorbents for the removal of arsenic species from waters. LDHs are common and abundant anionic clays on earth. LDH can occur naturally or can be synthesized simply and economically. The structure of LDH is based on positively charged brucite layers, and the positive charges are balanced by anions in the inner region. LDH has very weak inner layer bonds. The most important feature of LDH is its high surface area and high anion exchange capacity. Thanks to these properties, LDH is successfully used to remove oxyanions such as arsenite and arsenate from water. LDH can be regenerated in a short time for reuse. However, large-scale applications of LDH are limited. The base metals of LDH can go into the solution at a low pH, and high basicity is required if calcined LDH is to be used. In addition, factors such as pH, adsorbent dosage, temperature, calcination, and particle size are factors that affect LDH and oxyanion adsorption. Within the scope of the study, the removal of arsenic from waters with FeHT, known as iron-based LDH, was examined. Obtained data suggest

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that FeHT can be used effectively for arsenic removal from waters, and the arsenic concentration can be reduced below the maximum drinking water pollution level (10 μg/L) determined by the WHO; it is safe as well. If pH > 12, Fe^{3+} ions dissolve. However, this probability is very low. Iron does not have any harmful effects on human health, even if it is slightly dissolved. Therefore, in bringing hydrotalcite to potable standards for the removal of arsenic from contaminated water, it should be checked whether the adsorbent is safe for health.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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