

ORIGINAL RESEARCH PAPER

Remediation of artificially Arsenic Contaminated clay Soil (Sepiolite) by extraction and its effect on soil properties

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ABSTRACT

Soil properties such as particle size distribution, plasticity, compressibility, or strength of the soil always affect the geotechnical design in construction. The knowledge of contaminants and their extraction on natural adsorbents (clay soils) with various treatments, and extractants is essential for geo-environmental engineering applications. To address these challenges, sepiolite as a widely used environmental natural resource (which is a native and non-expansive clay soil with high cation exchange capacity), was subjected to a study of the effect of arsenic (As) contamination and extraction using the different solutions on its characteristics in two incubations period (7 and 28 days curing). After incubation, various physical and mechanical tests were performed on As contaminated and extracted clay soil to investigate the effect of anionic contaminant on the clay soil properties and to evaluate how the properties of the contaminated soil change after extraction, as well. The optimal extractant for arsenic extraction from sepiolite was determined by hydrochloric acid and nitric acid, respectively. The results revealed improvements in some soil characteristics. Arsenic extraction by acid after contamination caused changes in the properties of clay soil specimens, including a decrease in pH, plastic index, and fine particles content which reduced the permeability coefficient in the soil. The overall results showed that the environmental and engineering properties of clay mineral are suitable for use as clay liners in pounds containing anionic leachates with improving workability.

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1. Background

Heavy metal removal has become one of the most important concerns because of the increasing release of heavy metals into the environment (Ajami and fataei, 2015; Babaei et al., 2017; Rezaei-Aghdamet al., 2022). Arsenic contamination of soil is a significant global human health risk (Gerdelidani et al., 2021) and cause skin, kidney, liver, bladder, and lung cancer (Khalili Arjaghi et al., 2020; Aprila et al., 2023). Arsenic contamination of terrestrial environments has occurred globally as a result of anthropogenic activities. Remediation of heavy metal contaminated soil is an expensive process, and a clear understanding of the factors controlling the levels of risk is needed to improve risk assessments and inform remediation efforts (Abbasi et al., 2021). Arsenic is usually associated with gold, copper and lead ores. Extraction and smelting of these mines can enhance arsenic and metals contamination (Lee et al., 2011; Li et al., 2007; Cheng et al., 2021, Gholamin and Khayatnezhad, 2011). Therefore, before arsenic can reach body water or groundwater, arsenic contamination of soil must be treated. One of the factors affecting the toxicity of arsenic is soil texture; sandy soils are five times more toxic than clay soils. Natural mineralization and activities of microorganisms stabilize arsenic; but human interference causes arsenic contamination (Chen and Costa, 2021; Gerdelidani et al., 2021; Ghomi Avili and Makaremi, 2020; Lake et al., 2021; Li et al., 2021; Loukola-Ruskeeniemi et al., 2022).

Sadr et al. & Mousavi Moghanjooghi, et al., (2021) concluded that all methods that are commonly used for heavy metals removal suffer from one or more drawbacks, limitations and scope of application and advantages of each method is depend on some conditions such as pH, adsorption time, solvent, heavy metal type and etc (Sadr et al., 2021; Mousavi Moghanjooghi, et al., 2022; Ahmadi et al, 2019; Fazeli et al., 2019; Gazijahani et al., 2017). Different treatment techniques can be used for remediation of polluted soil include electrokinetic remediation, phytoremediation, solidification, soil replacement, thermal treatment, microbial remediation stabilization, soil washing technology, etc. Soil washing has been introduced as a rapid, time-economical, simple operation, and high efficient method and was widely used for soil remediation (Abbaspour et al., 2013; Jalilzadeh yengejeh et al., 2014; Feng et al., 2020; Wang et al., 2020; Xu et al., 2022).

Soil washing efficiency is influenced by washing agents (Hajjabbari and fataei, 2016; Jalilzadeh Yengejeh et al., 2017; Feng et al., 2020). Organic acids, inorganic acids, inorganic salts, chelating agents, and surfactants can be used as chemical agent for soil washing in order to heavy metals remediation (Huang et al., 2022; Farsani et al., 2022). During soil washing process, physical and chemical properties of polluted soil can be changed. Physical separation process resulted in soil texture change and chemical washing solution that extract metals from solid to aqueous phase can affect chemical properties of

soil (Yi and Sung, 2015; Masoumi and Yengejeh, 2020).

Any change in the soil layer engineering specifications results in reduced load-bearing capacity and increased overall and relative subsidence of the structures. Therefore, structures can become structurally disrupted or unusable. Accordingly, to obtain a good understanding of the behavioral pattern of contaminated soils, various investigations such as triaxial, direct shear, permeability and consolidation experiments have been conducted by researchers to clarify the effect of different contaminants on the geotechnical parameters of soils and more or less similar or different results have been presented (Maleki and Barkhordari, 2014; Jafari et al., 2017; Marrani et al., 2019). Ouhadi and Rafiee (2009) investigated the effect of lead and zinc on kaolinite clay; subsequently, physical experiments and microstructural studies evaluated the change in soil behavior. The results of Atterberg limits, as a physical index of soils, show that the liquid limit decreases with increasing pollutant densities (Ouhadi and Rafiee, 2009).

There are many methods to extract and analyze total arsenic (Hudson-Edwards et al., 2004). Several types of extracts can be used to extract heavy metals from the soil such as mineral salts, potassium phosphate, potassium chloride, potassium nitrate, and inorganic acids such as sulfuric acid, nitric acid, hydrochloric acid, etc. (Jang et al., 2005). The sequential extraction method is, in fact, an experimental and analytical method in which the different geochemical phases of metals in sediment samples are synthesized during 5 operational phases using chemical solvents (Opara et al., 2022; Rodgers et al., 2015). Desogus et al. (2013) conducted Batch leaching experiments using acid solutions as extracting agents. During leaching tests, concentrations of Zn, Pb, Cd, Cu, Mn and Al were monitored. The results indicate that the best leaching solution varies for all analytes and soil samples examined (Desogus et al., 2013). Soil ecological properties cannot be fully improved after the removal of metals due to adverse physical and chemical changes (Yi and Sung, 2015). Dos Anjos et al. (2014) also used inorganic acids such as hydrochloric acid and nitric acid to remove heavy metals like arsenic (dos Anjos et al., 2014). In other soils, with high arsenic contamination rates, acid washing was used to extract arsenic to reduce its environmental and health risks (Fazle Bari et al., 2022; Guo et al., 2020; Tokunaga and Hakuta, 2002).

Therefore, given the importance of arsenic contaminant and its prevalence in the environment, besides the world wide applications of sepiolite clay soil especially as a liner in environmental geotechnical structures, the present study has been conducted with the aim of (1) studying the properties of sepiolite clay as a soil material (2) the effect of contamination on physical properties of soil (3) investigation of the forms of arsenic adsorption in the studied soils (4) the effect of arsenic extraction treatment on the physical and mechanical properties of the sepiolite soils.

2. Materials and Methods

1.2. Soil and materials

Sepiolite clay soil was selected for the study due to its ability to adsorb heavy metals and to be used as a material in geo-environmental projects. The clay was sampled from a mine at Khorasan Razavi province (Iran) and soil was passed through sieve #10 for experiments.

2.2. SCharacterization of clay soil

The physical properties of sepiolite clay soil were first studied to obtain a good understanding of the soil characteristics as a material; for this purpose, experiments such as particle size distribution (D7928), Atterberg limits (D4318), specific gravity (D854), modified compaction, unconfined compressive strength of modified compacted soil samples (D1557), falling head permeability coefficient (D5084) were performed in accordance with ASTM standard. Standard methods were used for the determination of soil calcium carbonate equivalent (CCE) and gypsum (Nelson and Sommers, 1983), soil pH (0.01 M CaCl₂ suspension), cation exchange capacity (CEC) (Chapman, 1965). The specific surface area (SSA) obtained by the Brunauer-Emmett-Teller (BET) approach (Brunauer et al., 1938).

3.2. Preparation of artificially polluted clay soil

Sepiolite (S0) samples were contaminated with sodium meta arsenate and sodium hydrogen arsenate hepta hydrate at 1000 ppm concentration of AsIII and AsV solutions; As solutions mixed with soil at optimum soil moisture content and kept for 7 (S1) and 28 (S2) days. The optimum moisture content of sepiolite was maintained by distilled water over time. After the end of periods, the contaminated samples were exposed to air and collected after drying for the experiment. Arsenic was extracted from the sample in 28 days soil treatment due to the effect of time on the distribution of different forms of arsenic (ES2).

4.2. Extraction of Arsenic

4.2.1. Extraction of Arsenic

One of the most important methods to quantify water-sediment interactions is 4-step sequential extraction and distribution coefficient calculation method; pH or ionic strength of water also affects these processes. The following extraction experiments were performed:

Soluble and exchangeable fraction: 8 ml of 1 M magnesium chloride at pH of 7 for 1 hour at 25 °C with one gram of soil.

Carbonate bonding fraction: Soil residue from the exchangeable phase with 8 ml of 1 M sodium acetate with pH of 5 adjusted with acetic acid for 5 h at 25 °C.

Iron and manganese oxides binding fraction: Soil residue from carbonate phase with 20 ml of 0.04 molar hydroxylamine hydrochloride salt in 25% acetic acid at pH of 2 for 6 h at 96 °C.

Organic component: Soil residue from the bonding of iron and manganese oxides with 3 ml of 0.02 M nitric

acid to 5 ml of 30% added aqueous water at 85 °C for 2 hours (Tessier et al., 1979).

Finally, the concentration of the arsenic element in different soil phases was measured using the Perkin Elmer-800 graphite furnace atomic absorption apparatus.

3.2.2. Total extraction

Total As amounts were extracted from soil samples by 1 M di-potassium phosphate (K₂HPO₄) (Georgiadis et al., 2006), 10% sulfuric acid, 10% hydrochloric acid, 10% acetic acid, 10% nitric acid (Alam and Tokunaga, 2006) and 1 M potassium dichromate at the ratio of soil to solution 1: 25 (1 g soil -25 g solution); As was extracted at room temperature. Arsenic adsorption capacity and removal percentage were calculated using equation 1 and 2, respectively (Hashemi nasab et al., 2016).

$$Q_e = ((C_0 - C_e)/M) * V \quad (1)$$

Q_e: mg of arsenic adsorbed in the gram of adsorbent

C₀: Initial concentration of adsorbate (mg/L)

C_e: Secondary concentration of adsorbate (mg/L)

M: Adsorbent mass in (kg)

V: Volume of solution in (L)

$$\text{Adsorption}\% = ((C_i - C_f) / C_i) * 100 \quad (2)$$

C_i: Initial contaminant concentration (mg/L)

C₀: Initial concentration of adsorbate (mg/L)

C_f: Final contaminant concentration (mg/L)

5.2. Instrumental analysis of Arsenic polluted soil and washing soil

To examine the type of soil minerals, soil samples were tested using the XRD-D6792 X-ray diffraction apparatus manufactured by PHILIPS with a Glass Cu Anode tube with the maximum power of 3 kW. Scanning electron microscopy (SEM) studies of the clay samples were performed using TE-SCAN electron microscopy imaging with nm resolution and semi-quantitative analysis by EDS (Energy Dispersive x-ray Spectrometry) analyzer. Elemental analysis of the clay in this study was performed by the X-ray fluorescence spectroscopy apparatus made by the PHILIPS Company (PW1410).

3. Results and Discussion

1.3. Soil Properties

Sepiolite minerals are alkaline minerals that are mostly formed in saline-gypsum media. The studied clay soil has a cation exchange capacity of 15 meq/100 and contains 15% carbonate minerals, 10% quartz, and 5% sulfate minerals as impurities. The specific surface area of this clay soil is 119 m²/g, the higher the specific surface area, the higher the clay's ability to adsorb heavy metals. Also, according to the elemental analysis results using XRF (Table 1), sepiolite as a fibrous silicate minerals is associated with silicon and magnesium oxides with the highest content.

2.3. As fractionation

The distribution of different forms of arsenic in samples

S1 and S2 has been given in Table 2. Percentage of arsenic chemical species in S1 sample were respectively: Fe and Mg oxides bonding > carbonate > exchange > organic fraction and the highest fraction in S2 sample is related to iron and manganese and then the carbonate phase; but the third percent is related to the organic phase and finally the lowest one is associated with exchange phase. Iron oxides and carbonate compounds play the

most role in the adsorption of contaminants. Liao et al. (2016) investigated As washing from different particles size, their results showed Fe–Mn oxides, clay minerals, and organic matter can influence on the presence of arsenic in the small particles. They indicated adsorption of metals using Iron oxide can occur through oxidation–reduction reactions and surface coordination due to large surface area of iron oxide (Liao et al., 2016).

Table 1. Main elemental composition of sepiolite sample

Element	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	MgO
%	52.9	2.56	1.23	0.10	0.01	23.6
Element	K ₂ O	TiO ₂	MnO	P ₂ O ₅	LOI	
%	0.05	0.09	0.09	0.01	19.8	

Table 2. Main elemental composition of sepiolite sample

Treatment type	Sample	
	S1*	S2*
Exchangeable Fraction	15.3	1.74
Organic Fraction	8.36	7.26
Fe/Mn oxide fraction	208	185
Carbonate Fraction	30.7	17.0

* S1: contaminated sepiolite soil-treatment 7 with days incubation

* S2: contaminated sepiolite soil-treatment 28 with days incubation

Over time, arsenic enters the non-exchangeable phases, with less arsenic in the exchange phase and lower arsenic extraction and higher adsorption. Also, according to the microstructural results presented in the following sections, the presence of iron and manganese in soil samples is evident. The exchangeable species are usually related to adsorbed elements (in the form of electrostatic surface adsorption and specific surface adsorption) on the surface of the sediment sample. Carbonates are important adsorbents of many elements. A metal in the carbonate form does not have a strong bond and can enter the environment in the face of changing environmental conditions. The nature of the source of contamination, especially in terms of mineralogical composition, has a great influence on the chemical separation pattern and the potential for mobility and bioavailability of pollutants in the water and soil environment. The reactivity of metals is directly related to their physical and chemical forms and their position and is the main parameter governing their mobility and bioavailability.

The results from this study show that textural properties of soils can serve as useful indicators for assessing As availability, mobility and toxicity in the soils. Our results showed that coarse textured soils released higher amounts of easily labile As. In heavily-textured soils high in clay sized particles, As was commonly immobilized by Fe oxides. The study findings could clarify the low and high phytotoxicity of As observed in crops grown on fine- and coarse-textured soils (Gerdelidani et al., 2021).

3.3. Total extraction results

The results of the arsenic content during total extraction by atomic absorption spectrometry in S1 and S2 soil samples have been shown in Fig. 1. The highest extraction values for S1 and S2 samples with nitric acid were 62.6% and 59.8%, respectively. In sample S1, which has a shorter duration of contamination, arsenic is directly adsorbed by fine-grained sepiolite particles. Also, according to the results of (selective) sequential extraction, the highest amount of exchange phase is related to sample S1; as a result, the percentage of arsenic extracted is higher. In the S2 soil sample, arsenic is released into the internal channels of sepiolite and more arsenic is transferred from the exchange phase to the iron oxide phase; therefore its adsorption is higher that is in accordance with (Lombi et al., 2000) study. Dos Anjos et al. (2014) also used different extractions to remove heavy metals such as arsenic and found that nitric acid and hydrochloric acid have high extraction ability (dos Anjos et al., 2014).

Deng et al. (2018) combined use of sepiolite and dehydrated sludge as a repair agent to passivate arsenic contaminated soil. Experiments were taken out on 1 day, 5 days, 10 days, 20 days and 40 days. Based on the dissolution strength, five reagents were chosen in the method to perform the sequential extractions of arsenic. They stated that after passivation experiment, the best remediation period was 1–10 days (Deng et al., 2018). Yang et al. (2021) evaluated the remediation efficiency of nZVI-BC for Cd and As co-contaminated soil. For the incubation experiments the nZVI-BC was added to

the contaminated soil at rates of 0.05%, 0.10%, 0.25%, 0.50%, 0.75%, and 1.00% (w/w) and eventually, 80 g of the soil was sampled after 1, 3, 7, 14, 21, 30, 60, 120, and 180 days of incubation. As was extracted with 0.05 M $\text{NH}_4\text{H}_2\text{PO}_4$ at a soil to water ratio of 1:25 for 16 h. After 14 days of incubation. Over 93% of the total As existed in the residual form (Yang et al., 2021).

In a comprehensive study by Jiang et al. (2023), montmorillonite modified by amorphous ferrihydrite was synthesized and its capability for immobilization of As, Sb, and Pb in soil by SBET, DTPA, and TCLP extraction was investigated. SBET reached 87.34% for As. The DTPA method achieved a reduction of over 80 % from 0.81 mg/kg to 0.17 mg/kg after 3 days and almost reduced to 0 mg/kg after 56 days. Also, As was not detected in the TCLP leachate after 10 days, and its concentration remained stable for 56 days (Jiang et al., 2023).

In our previous study, we assessed the extractive treatment of arsenic from vermiculite as the contaminated soil in 7 and 28 days intervals. The amount of extracted arsenic from the samples with 7-day incubation was 21.3, 27.6, and 70.5 mg/Kg for exchangeable, Fe/Mn oxide, and carbonate fractions, respectively. Moreover, no As was detected in Organic Fraction after 7 days. For 28 incubation samples, Fe/Mn oxide fraction and carbonate fraction methods had 34.3 and 35.2 mg/Kg of extracted As. It should be noted that extracted arsenic was not detected in exchangeable fraction and organic fraction after 28 days of treatment (Abbaslou et al., 2022).

3.4. Micro-structural results

Scanning electron microscopy (SEM): Figures 2 (a and b) showed scanning electron microscopy of sepiolite sample after 28 days exposure to arsenic contamination. The fibrous structure of sepiolite didn't affect by pollution.

Presence of arsenic peaks in polluted sepiolite sample confirming arsenic pollutant in this soil (Fig 2c). The results of the SEM of arsenic polluted clay sample (Fig 2b) after acid washing showed partial acid dissolution in both octagonal and tetrahedral sheets of sepiolite. First, by breaking the fibers in the center, a deep gap has created along the axis. Finally, acid extraction results in a complete loss of fiber as reported before by (Yebera-Rodriguez et al., 2003). Arsenic content has also decreased significantly after extraction (Fig. 2d).

X-ray Diffraction (XRD): According to the XRD results, any irregularity in the clay mineral layers and the interlayer space can reduce the intensity of the X-ray diffraction peaks due to the presence of elements (Jeon and Nam, 2019; Rabiei and Hashemi, 2019). The increase in peak intensity can be seen in the contamination. Extraction has also reduced the intensity of the peaks in the extraction (Figure 3). Panda et al. (2010) showed the effect of acid treatment on kaolinite structure and observed reduction of peak intensity of the clay. They concluded that acid can affect the crystalline character of the clay (Panda et al., 2010).

3.5. Physical, chemical and mechanical soil properties

The results of the different properties of sepiolite clay soil through treatments have been presented in Table 3. Soil texture after acid extraction has shifted to clod and coarse grains. The fine grains were reduced and the coarse grains were added. Reduction of soil clay fraction reduces surface tension and water adsorption capacity of soil and elements. Increasing sand increases the water to move downward and dissolved elements in soil water (Yi and Sung, 2015). The plastic index has decreased after extraction. Acidification of the S2 sample did not change the specific gravity based on the results reported in Table

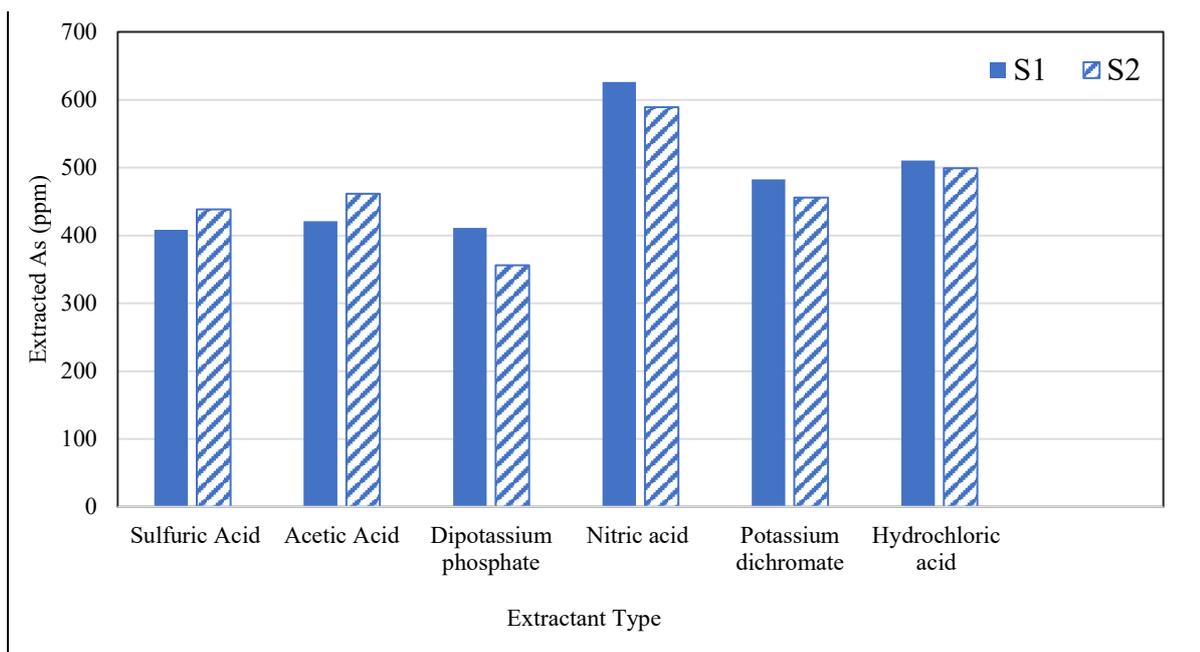


Fig 1. The concentration of extracted As with different extractant (ppm)

3.

Acid extraction resulted in a significant decrease in pH of the ES2 sample, due to the release of H⁺ in the soil medium, which is increased by adding acid to the medium and acidifying the medium.

The soil plasticity index (PI), should generally be greater than 10 percent. However, soils with very high PI, (greater than 30 percent), are cohesive and sticky and become difficult to work with in the field. When high PI soils are too dry during placement, they tend to form hard clumps (clods) that are difficult to break down during compaction. Preferential flow paths may be created around the clods allowing leachate to migrate at a relatively high rate (EPA, 1993).

Soils typically possess a range of physical characteristics, including particle size, gradation, and plasticity, that affect their ability to achieve a hydraulic conductivity of 1 x 10⁻⁷ cm/sec. Testing methods used

to characterize proposed liner soils should include grain size distribution, Atterberg limits, and compaction curves depicting moisture and density relationships using the standard or modified Proctor, whichever is appropriate for the compaction equipment used and the degree of firmness of the foundation materials.

Although the soil may possess the required properties for successful liner construction, the soil liner may not meet the hydraulic conductivity criterion if the construction practices used to install the liner are not appropriate and carefully controlled.

Liner soils usually have at least 30 percent fines (fine silt- and clay-sized particles). Some soils with less than 30 percent fines may be worked to obtain hydraulic conductivities below 1 x 10⁻⁷ cm/sec, but use of these soils requires greater control of construction practices and conditions.

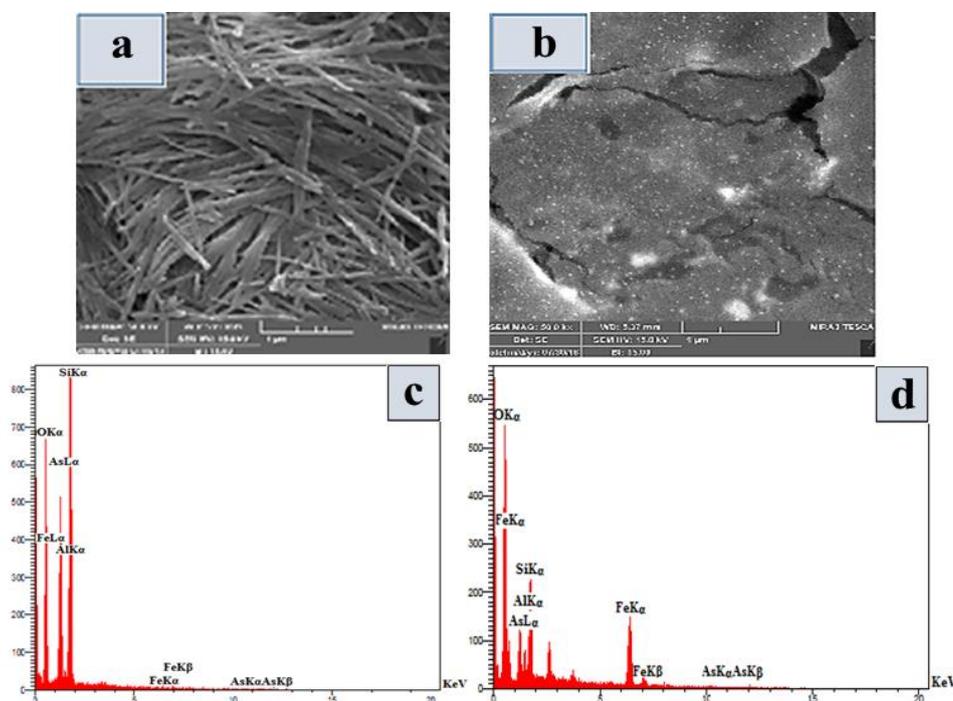


Fig 2. (a) SEM result of S2 sample (Arsenic polluted sepiolite), (b) SEM result of ES2 sample (polluted soil after acid washing), (c) EDX spectra of S2 sample, (d) EDX spectra of ES2 sample

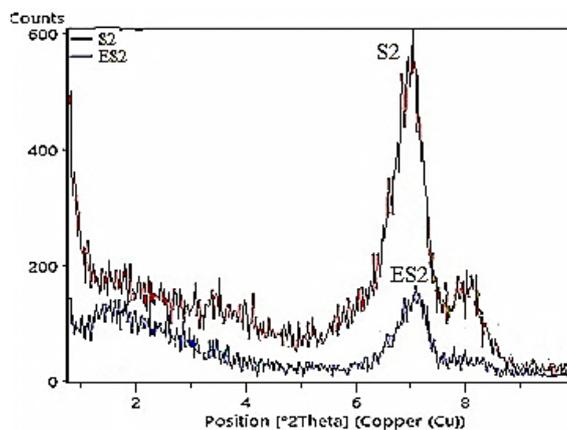


Fig 3. X-ray diffraction (XRD) of S2 and ES2 sample

Table 3. Comparison of some physiochemical properties of S0, S1, S2 and ES2

Soil sample	Clay (%)	Silt (%)	Sand(%)	Gs
S0	9.00	83.0	8.00	2.24
S1	21.0	68.0	11.0	2.33
S2	27.0	61.0	12.0	2.40
ES2	26.0	56.0	18.0	2.40
Soil sample	ω_{opt} (%)	γ_{dmax} (KN/m ³)	pH	UCS (kPa)
S0	37.5	13.1	7.65	250
S1	37.2	13.2	7.02	280
S2	36.1	13.2	7.11	405
ES2	33.8	13.8	5.03	445
Soil sample	PI	K (cm/s)	Soil classification	
			USCS	AASHTO
S0	14.1	10-8×3.0	MH	A-5
S1	24.4	10-8×6.7	MH	A-7-5
S2	24.6	10-8×3.9	MH	A-7-5
ES2	12.1	10-7×5.0	MH	A-7-5

S0: sepiolite soil, S1: contaminated sepiolite soil-treatment with 7 days incubation, S2: contaminated sepiolite soil-treatment with 28 days incubation, ES2: As extracted sepiolite soil

4. Conclusion

1. The results of sequential As extraction experiments showed that arsenic is easily exchangeable in short periods; but over time, arsenic enters the non-exchangeable phases of the soil and reduces its ability to exit the soil and enter the groundwater. The most important factor for arsenic fixation is iron and manganese in the soil phase.

2. The highest extraction rate was obtained for sepiolite clay with nitric acid. Soil texture after extraction has gone toward coarse grains. Acid extraction caused changes in the samples, including a decrease in pH, plastic amplitude, and percentage of fine particles. It also increased the percentage of coarse grains in artificially contaminated soil.

3. Generally, the sepiolite soils have liquid limits and plastic limits greater than the recommended values. Therefore, this makes the sepiolite soils unstable and unsuitable for use as landfill liners. This research introduces the amending of sepiolite soil properties by mixing it with the silt of fine sand. This method could be efficient in improving the Atterberg limits for use as clay liners of geo-environmental constructions.

Conflict of interest

The authors declare that they have no conflict of interest.

Additional Information And Declarations

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Competing Interests

The author declare there is no competing interests, regarding the publication of this manuscript

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