Marine Science Center-University of Basrah

Mesopotamian Journal of Marine Sciences
Print ISSN: 2073-6428
E- ISSN: 2708-6097
www.mjms.uobasrah.edu.iq/index.php/mms

Optimal Conditions for Lead Ions Adsorption using Powdered *Typha domingensis* Plant

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Article info.
- ✓ *Received: 10 August 2024*
- ✓ *Accepted: 22 September 2024*
- ✓ *Published: 29 December2024*

Key Words:
Flame atomic absorption Spectrometry
Fourier transform infrared spectroscopy
Lead
Scanning Electron Microscope
Typha domingensis

Abstract - *Typha domingensis* is considered one of the most important plants endemic in Iraq and is widely used in adsorption due to its global distribution in natural environments and its ability to survive in contaminated sites. The samples were collected from various areas in Basrah province southern Iraq and were exploited for removed of lead ions (Pb+2) from its aquatic solution. Several parameters (pH, contact time, temperature and mixing speed) were studied to obtaining optimal conditions for Pb+2 removals. Flame atomic absorption Spectrometry (FAAS) was used to measure the concentration of lead and the adsorption ratio on adsorbent surface, which showed high efficiency to remove Pb+2 (95.9 %) at pH 6.5, 10 min, 15 ºC, and 200 rpm. Scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FT-IR) were used to determine the surface morphology and functional groups. The optimum conditions were applied to a real sample of industrial waste and showed an adsorption ratio of 32.4%.

الظروف المثلى لادمصاص ايونات الرصاص باستخدام مسحوق نبات *Typha domingensis*

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المستخلص - نبات *Typha domingensis* هو من أهم النباتات المستوطنة في العراق ويستخدم بشكل واسع في الامتزاز بسبب انتشاره الكبير في البيئات الطبيعية وقدرته على البقاء في المواقع الملوثة. جمعت العينات من مناطق مختلفة في محافظة البصرة جنوب العراق وتم استغلالها لإزالة أيونات الرصاص (Pb+2) من محلولها المائي. تمت دراسة العديد من المعلمات مثل الرقم الهيدروجيني ووقت التلامس ودرجة الحرارة وسرعة الخلط للحصول على الظروف المثلى لإزالة أيونات الرصاص. استخدم مطياف الامتصاص الذري لقياس تركيز الرصاص ونسبة الامتزاز على سطح المادة الماصة والذي أظهر كفاءة عالية في إزالة أيونات الرصاص بنسبة (95.9 %) عند الرقم الهيدروجيني 6.5 ووقت التلامس 10 دقائق ودرجة الحرارة 15 درجة مئوية وسرعة الخلط 200 دورة في الدقيقة. كما تم استخدام المجهر الإلكتروني الماسح (SEM ومطيافية الأشعة تحت الحمراء FT- IR لتحديد شكل السطح والمجموعات الوظيفية. بعد ذلك تم تطبيق الظروف المثلى على عينة مخلفات صناعية وقد أظهرت نسبة امتزاز 32.4%.

الكلمات المفتاحية: مطياف الامتصاص الذري، مطياف الأشعة تحت الحمراء، ايون الرصاص، المجهر الإلكتروني الماسح، *Typha domingensis*

Introduction

Heavy elements are considered priority inorganic pollutants such as lead, cadmium and nickel even at low concentrations due to their high toxicity, stability and harmful effects on humans and ecosystems (Cimá-Mukul *et al.,* 2020).

DOI:https://doi.org/10.58629/mjms.v39i2.372 ,

Lead is renowned metalloid found in the earth's shell. It has exceptional properties including smoothness, flexibility, low melting point, and corrosion resistance, so it convenient for many industries (Awual, 2019; Giri *et al.,* 2022). Lead is present in industrial wastewater and at times in drinking water, acid batteries, metal coating and finishing, printing, and tanning are consider industrial origins of lead in water (El-Chaghaby *et al.,* 2020).

The accumulation of lead in the human body can cause serious hurt to the brains, tissues, and organs. In addition to that it also damages wetlands and agricultural lands. Lead uptake by plant roots is accompanied by calcium uptake and has a negative effect on plant morphology and growth. By penetrating tissues of plant, lead could alter activity of enzymes and balance of hormones, causing poor nutrient and water absorption, growth slowing and necrotic lesions (Abdel Hafez *et al.,* 2023; Khan *et al.,* 2023). Several mechanisms were reported to eliminate trace elements, such as solvent extraction, oxidation, electroflotation, membrane filtration, reverse osmosis, and coagulation. These procedures have some damages, like complexity of treatment and towering energy requests, as well as high costs. That is why it is important that we try to find a solution that is practical, cost-effective, and above all, available locally (Hazarika *et al.,* 2023).

Adsorption is an excellent remediation mechanism depending on the low cost, elasticity and simplicity of design and running, insensitivity to toxic contaminants, and the fact that, unlike most processes, it does not produce detrimental materials (Mohamed *et al.,* 2019). The use of biosorbents has increased in recent decades due to their high efficiency, affordability, and environmental friendliness (Liu *et al.,* 2021; Kumari *et al.,* 2024).

Macrophytes play a critical role in removing contaminants from surface water systems by providing typical habitat for growth microbes around the root region, and they filter, trap suspended particles, and excrete harmful materials that could be poisonous to pathogenic microbes. Many plant genera, such as *Cyperus papyrus, Typha domingensis, Phragmites australis, Panicum elephantipes, Scirpus* sp.*,* and *Canna* sp.*,* have been used as primary components of constructed wetlands and terrestrial wastewater treatment systems (Hamad, 2020; Hamad, 2023).

The cell walls of several plants, algae, and microorganisms are mainly composed of polysaccharides, proteins, and lipids that contain diverse functional groups (hydroxyl, carboxyl, amino, ester, and carbonyl groups). These have a large significance to the combination of metal ions in the solution (Khan and Khan, 2023).

Typha is a perennial aquatic or semi-aquatic plant common in the northern hemisphere and is quite applied for remediation water and soil pollution due to its nature of growing in swamps (Gupta *et al.,* 2023). Plant species such as *Typha domingensis* and *Canna indica* were used for remediation of Cr, Zn, and Ni under vertical flow situations (Vymazal *et al.,* 2021). Researchers found the most popular *Typha* sp that remove heavy elements from water, soils, and sediments in natural and constructed wetlands are *T. domingensis*, *T. latifolia*, and *T. angustifolia* (Bonanno and Cirelli, 2017; Hejna *et al.,* 2020). In the present study, our objective is to use Typha domingensis, a plant represented in several regions of Basrah province and widespread in southern Iraq, as a biosorbent to eliminate lead ions from aqueous solutions.

Materials and Methods

Samples Collection

Samples of *T. domingensis* were collected from different regions of Basrah province, southern Iraq, and put in sterilized plastic bags before transporting to the laboratory to prepare it for examination by performing the following operations, such as washing, drying, grinding, and sieving.

Prepare the Adsorbate Surface

All plant samples were washed few times with distilled water, desiccated at ambient temperature and pulverized with a grinder to obtain powder and then sieved at 63 µm to get homogeneous particles.

Prepare the Adsorbent Solution

The stock solution (1000 mgL^{-1}) was prepared using $PbNO_3$, and the standard solution (25 mgL^{-1}) was prepared through dilution it using deionized water.

Recommended Procedure

The adsorption method was used for elimination of Pb from their aquatic solution. The constant weight (50 mg) of *T. domingensis* powder was mixed with 20 mL of the standard solution in Erlenmeyer flasks size 100 mL. The optimal conditions have been studied, such as pH (4.5, 5.5, 6.5, 7.5 and 8.5), contact time (5, 10, 20, 30, 60, 90) min, temperature (15, 25, 35, 45 and 55) ºC, and mixing speed (50, 100, 150 and 200) rpm. After finishing each parameter, all solutions filtered with filter paper No. 40 (Maki *et al*., 2020). To determine the adsorption ratio, the concentration of lead was measured using flame atomic absorption spectrophotometer (Shimadzu AA7000).

Calculate of Adsorption Capacity and Adsorption Ratio

The capacity of adsorption (Qe) was calculated through the equation:

$$Qe = ((A-B) \times V) / M$$

Qe is the total of metal adsorbed (mgg^{-1}), V is the volume of the standard solution by unit L and M is the amount of the adsorbent by unit mg.

The adsorption ratio has been calculated by the equation:

$$\%\text{Adsorption ratio} = ((A-B) / A) \times 100$$

Where A is an elementary concentration of standard solution of Pb (25 mgL^{-1}), B is an equilibrium concentration of Pb in aquatic solution.

FT-IR Spectrum

The surface adsorbent of *T. domingensis* was analyzed to identify the active groups using Fourier transform infrared spectroscopy (Shimadzu FT-IR-8400s), using KBr disc method at wave numbers 200-4000 cm^{-1}.

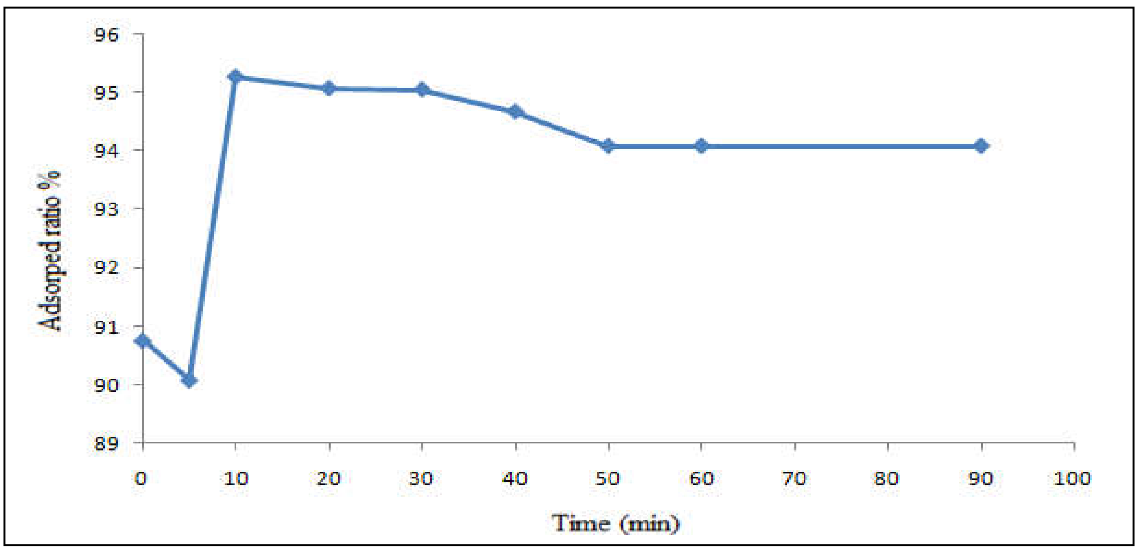
Scanning Electron Microscopy (SEM) Analysis

The morphology of the adsorbent material of *T. domingensis* was analyzed using SEM (TESCAN MERA III). The samples were fixed to brass parts using double-sided adhesive tape. SEM images were captured in various magnifications about ×800 - ×7500, voltage of 50 kV and the working area was kept between 4 - 5 mm.

Results and Discussion

Effect Contact Time of Pb Ions Removal by *T. domingensis* Powdered

For determining the optimal contact time for removing lead ions from their aqueous solution, the solution was exposed to shaking at 200 rpm, 20 °C, pH 6.5, and different times (0-90) min. The maximum adsorption ratio was obtained after 10 min. of shaking, and the balance time was 50 - 90 min. (Figure 1).



(Figure 1) The effect of time on the elimination of Pb.

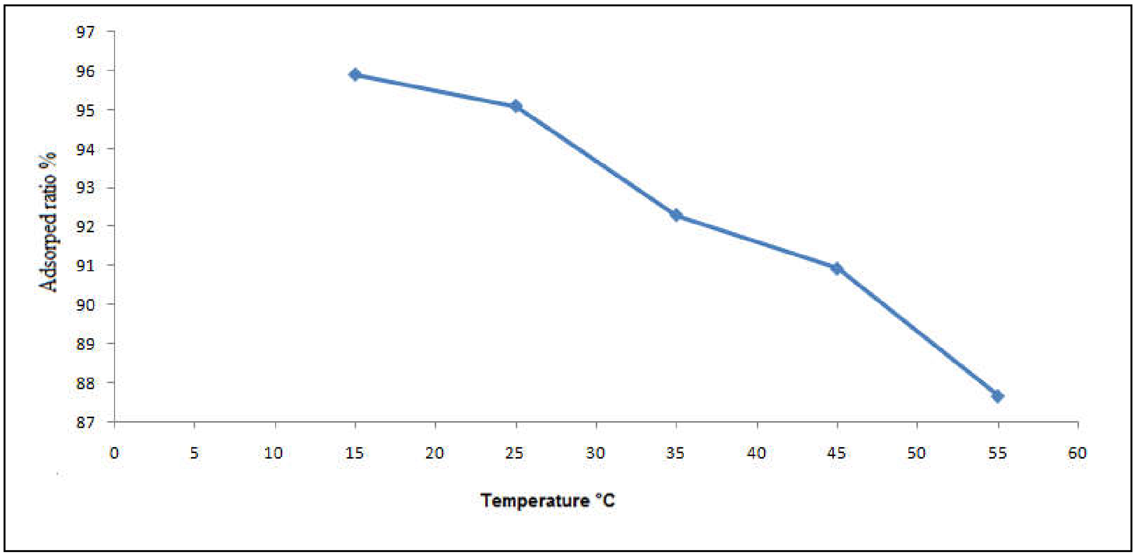
The removal of Pb occurs rapidly, versus regularly decreasing in the adsorption process with time until reaching the stability state, so the metal ion removal rate become higher depending on the number of free binding sites available, resulting in a bigger surface area for Pb removal. With time, the adsorption process becomes slower because of the reduction in active sites, which are occupied by competing metal ions (Abdel-Ghani *et al*., 2009; Sazali *et al*., 2020).

Contact time and kinetic parameters were required to determine the finest operating conditions in a continuous ion removal process. According to Abdel-Ghani *et al*. (2009), the removal of Pb by *T. domingensis* biomass rises with contact time and attains saturation in about 120 min.

Equilibrium state was achieved due to the repulsive interactions between the bulk phases and the metal ions adsorbed on the sorbent surface (Negi *et al.*, 2012).

Effect of Temperature for Lead Ions Removal by *T. domingensis* powdered

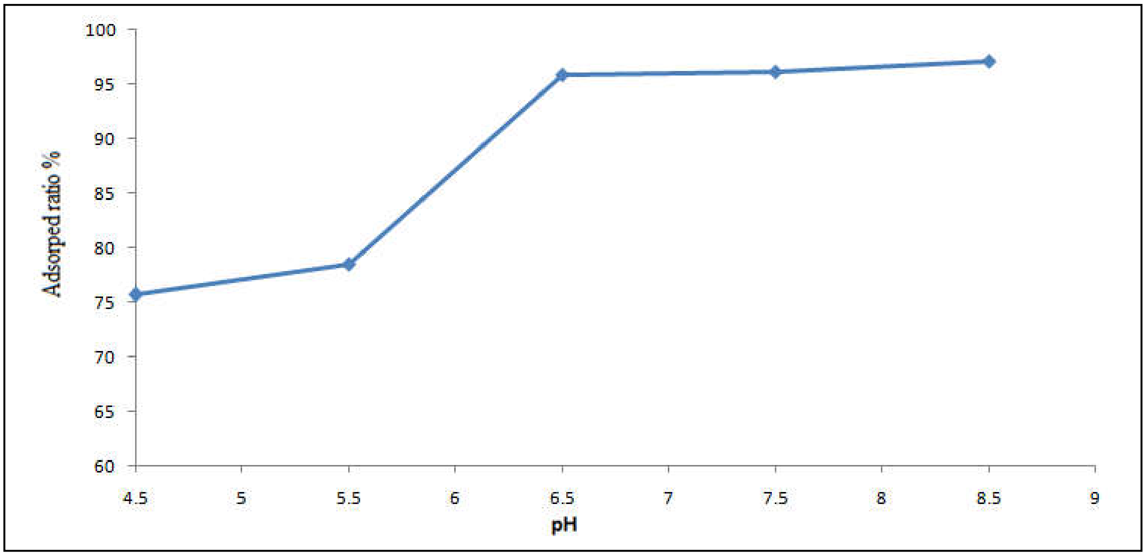
To investigate the temperature effect, the solution was left shaking at 200 rpm for 10 min, pH 6.5 and at different temperatures. The best temperature that has been selected for the removal of lead ions was 15 °C (Figure 2). There was a decrease in adsorption ratio with increasing temperatures, according to adsorption theory; increasing temperature followed a reduction in adsorption, which indicated a weakened adsorption between the biomass and the metal ion (Horsfall and Spiff, 2005). This result was consistent with a study by Mahdi *et al*. (2019) who found stronger adsorption for each pinnae and blade of date palm leaf at 15 °C.



(Figure 2) The effect of temperature in Pb removal.

Effect of pH in Pb removal by *T. domingensis* Powdered

The results of pH (Figure 3) show increasing in the adsorption ratio with the increasing pH values at 10 min, 200 rpm, and 15 °C. Once pH is greater than 6, the lead atoms will precipitate (Mahdi *et al*., 2018), so the increasing of pH values above 6 does not reflect the correct value of the adsorption ratio. It is generally accepted that the pH of the solution shows a crucial role in the design of an effective biosorption system. Onwordi *et al*. (2019) found that the changes in the pH of the adsorbate had significant effects on the surface functional groups of the adsorbent as well as the Pb existing in the solution, so pH is an active parameter to control adsorption. As the pH of the solution increases, there is greater elimination of Pb, which is probably due to the decrease in the concentration of hydronium ions. These results are consistent with previously reported results on lead removal in various biosorbents (El-Chaghaby *et al*., 2020; Abdel Hafez *et al*., 2023).

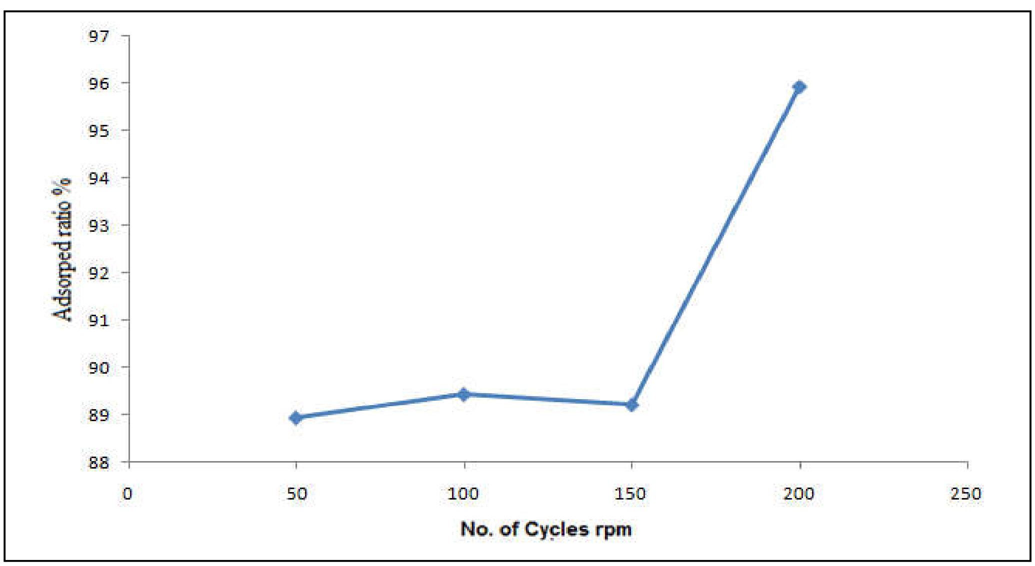


(Figure 3) The effect of pH in Pb removal.

Effect of Mixing Speed for Lead Ions Removal by *T. domingensis* powdered

The mixing speed effect of eliminating lead efficiency was studied through various speeds at 10 min, pH 6.5, and 15 °C. From Figure 4, optimizing the shaking is a critical factor in the biosorption process in which the resistance to mass transfer reduces with the increasing stirring speed, so the removal rate increases only to a certain rate due to biomass fragmentation (Parvathi *et al*., 2007; Martinez-Garcia *et al*., 2006). This result agrees with Maki *et al*. (2020) who found that the adsorption of Pb is effective in date palm seeds of Zahdi when the agitation rate increases to 200 rpm, and agrees with Singh and Zahra (2014) who noticed the best mixing speed of the solution of the *Emblica officinalis* pinnae powder mixture was 170 rpm.

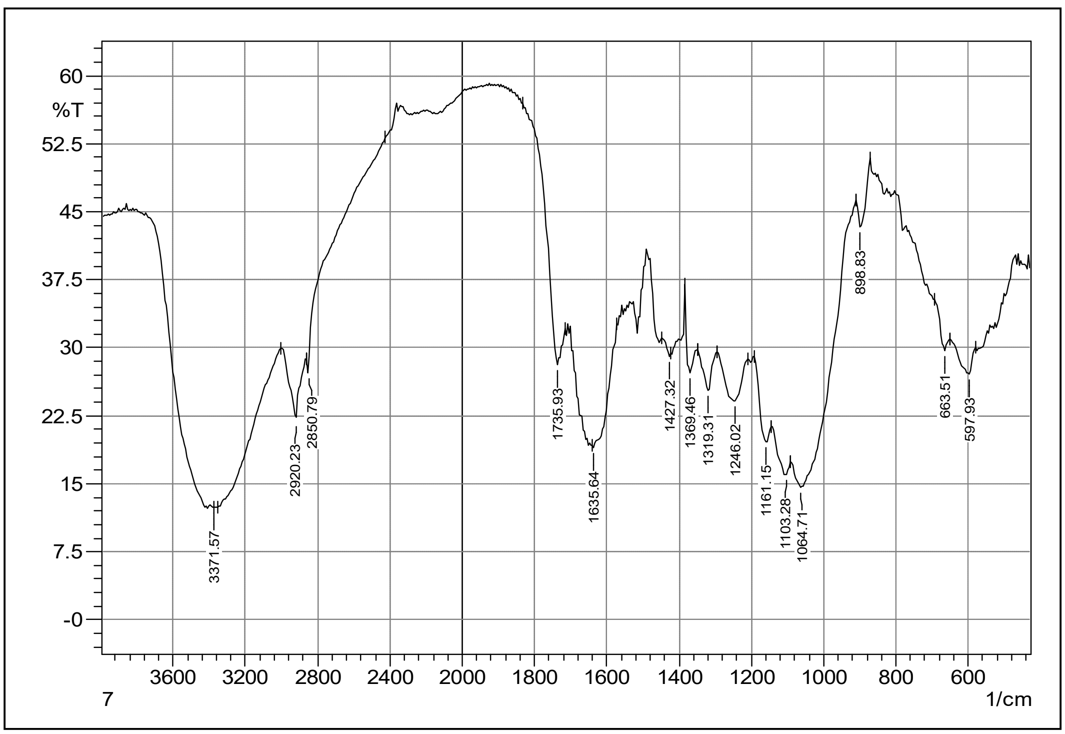
So that mixing speed of 100-200 rpm is adequate evidence for ease binding for all metal ions in active sites (Maki *et al*., 2020).



(Figure 4) The effect of mixing speed in Pb removal.

FT-IR Spectrum

To recognize the functional groups present on the surface of *T. domingensis*, FTIR spectral analysis was performed. Figure 5 shows various bands indicating the presence of active groups with different wavenumbers on the outer surface of the adsorbent used under study, where the hydroxyl (OH) groups appear in a strong and broad band at the wavenumber 3371 cm^{-1} which is involved in the synthesis of cellulose through its presence in the glucose molecule and considered the structural unit of cellulose, as well as the appearance of a band at 2920 and 2850 cm^{-1} belongs to the alpha group (–C-H). Meanwhile, the band that appeared at 1735 cm^{-1} is due to the existence of the carbonyl group (– C=O) in addition to the presence of a strong and broad beam belonging to the groups (C=C) and (C=N), that appeared at a wave number of 1635 cm^{-1}. The groups (C-O) and (C-N) appeared at 1246 cm^{-1} and 1319 cm^{-1}, respectively, and were also found in the *T. domingensis* leaf and fiber of Pandey *et al*. (2022), and in the *T. domingensis* leaf of Abdel-Ghani *et al*. (2009). All of the functional groups were able to connect with Pb through the chemical or physical adsorption process.

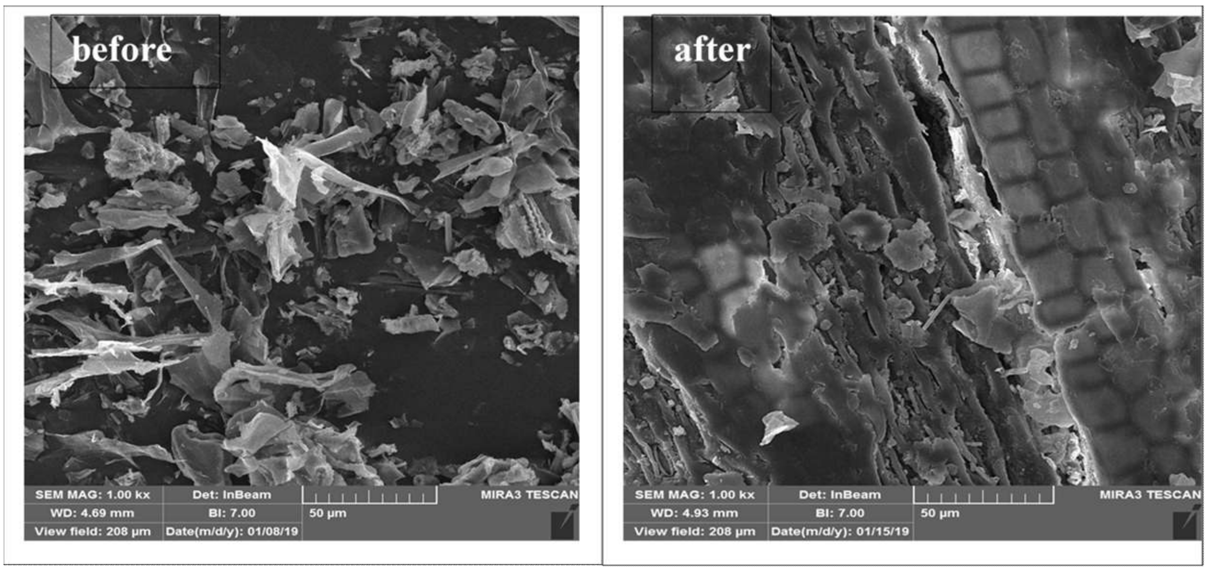


(Figure5) FTIR spectra for *Typha domingensis* powder.

Functional groups extant in waste include carbonyl, acetamide, phenol, amide, carboxyl, amine, alcohols, esters, and sulfhydryl (Baby *et al*., 2019). These groups have an affinity for forming complexes with heavy elements ion. Through spectroscopic analysis, many adsorption studies have confirmed the existence of functional groups and the binding capacity of these groups with elements ions (Banchhor *et al*., 2021).

SEM Analysis

The SEM image of *T. domingensis* is shown in picture 1. The surface texture and morphology of the biosorbent can be observed after and before contact with lead ions.
Various shapes and sizes of particles on the surface topography of *T. domingensis* before adsorption were shown. The particles have irregular surfaces with numerous unequal holes and small slots with wavy and so-broken verges; those features could give more surface area. The surface of *T. domingensis* turns shiny and soft and closes the pore structures after Pb adsorption. This may be to the binding between lead ions and the functional groups of the surface (Amin *et al*., 2017).



(Picture 1) SEM for the *Typha* biomass before and after lead adsorption

Application Adsorption on an Industrial Waste Sample

The waste industrial samples were collected from Nahran Omar field, western Basrah, Iraq. All the optimal conditions of the aqueous solutions were used in this experiment (Table 1).

(Table 1) Adsorption of lead ions from waste industrial samples using *T. domingensis* powdered.

adsorbent Surface	pH	Time (min)	Temp. °C	Mixing speed rpm	Standard solution of Pb mgL^{-1}	Adsorption ratio %
Typha domingensis	6.5	10	15	200	25	32.4%.

Removal of Pb in the waste sample was 32.4%; this was less of the optimal condition used in standard solution removal, which may be due to the fact that the sample contained oil residues, so that caused interference with other factors that interacted with lead for binding to the active sites.

The influence of various contaminants: if the coexisting contaminant contends or composes a complex with a base contaminant at link positions, a higher concentration of the second contaminant would lessen the biosorption distance of the base contaminant (Abdi and Kazemi, 2015). Gomes *et al*. (2014) found that the high efficiency of the aquatic macrophyte *T. domingensis* in constructed wetlands for the remediation of water polluted for mercury. While Pandey *et al*. (2022) found that *T. domingensis* fibers, like most fibers of plant origin, are composed mainly of cellulose. The structures of biochemical substances are partly joined to various functional groups, such as carbonyl, hydroxyl, and methyl groups, as a sorbent for the adsorption of Pb (Sulyman *et al*., 2017).

Conclusions

Typha domingensis powdered is considered a suitable adsorbent for eliminating Pb from industrial wastewater. Furthermore, *T. domingensis* is a natural, economic, and affluent substance. Its leaf powder may be exploited to simultaneously eliminate lead ions from aqueous solutions. The fast adsorption rate, high adsorption ability, optimal pH, contact time, temperature, and optimal speed rate were screened, and the adsorption percentage was 32.4%.

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