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Removing of Some Heavy Metals from River Water Using Salvia rosmarinus Plant Extracts

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Abstract. Removing heavy metals from water is of great benefit because these elements pose great risks to human health. Over the past decades, many traditional methods have been used to remove these pollutants from water, such as: ultrafiltration, reverse osmosis, ion exchange, solvent extraction and chemical precipitation. However, these methods have disadvantages such as high costs. In this study, aqueous and ethanolic extracts of rosemary (*Salvia Rosmarinus*) were introduced to remove heavy elements from polluted river water as new environmental sustainability. Five heavy metals were monitored and tested their concentrations in river water by Inductively Coupled Plasma Spectroscopy (ICP). The results showed that the alcoholic and aqueous extract of rosemary proved to be highly effective in removing the element (Fe) by 100%, and it was also able to remove the element (B) by 92%, while the removal rate of (As) and (Mn) was 100%, while it was able to reduce (Sr) by 29%. The best removal rates were recorded at a pH of 8, with a removal time of 30 minutes, and an extract concentration of 0.2 g/L of polluted water. Rosemary extract has proven its effectiveness in removing heavy elements and is considered a promising future alternative in the field of water treatment.

Keywords: Water treatment, Heavy metals, Rosemary, Jar test, Ashar River

1. Introduction

Water is considered one of the most important natural resources in the world today, as it represents a vital source for the survival of all living organisms and human development as well. At a time when industrialization and urbanization are accelerating, water demand and consumption are increasing rapidly to the point that the problem of water scarcity has become an important obstacle to economic development. Meanwhile, water pollution, especially heavy metal pollution within water, has become an important global environmental issue for everyone (Yang *et al*,2019). Moreover, the discharge of sewage into water bodies has a significant negative impact on water quality and creates major environmental and health problems (Al-Nabhan *et al*,2021). Uncontrolled discharge of water is widespread in countries that are not developed in the field of environmental protection (Chasib *et al*,2021). This problem greatly affects countries that suffer from water scarcity problems (Zaboon *et al*,2022). Due to the presence of organic and biological materials, raw water is unfit for human consumption (Fajri *et al*,2023). Excessive release of heavy metals into the environment resulting from industrialization and urbanization has been a major problem all over the world. Unlike organic pollutants, most of which are susceptible to biological decomposition, heavy metal ions do not decompose into harmless end products in this case at all (Hegazi,2013). Toxic heavy metals can be distinguished from other pollutants in that they cannot be biodegraded but can accumulate in living organisms, resulting in a variety of diseases and disorders (Al-Gizzi *et al*,2024).

Heavy metals are non-biodegradable chemicals that can be linked to the food chain of an ecosystem (Taher *et al*,2023). The presence of heavy metal ions is a major concern due to their high toxicity and are dangerous to all life forms. Heavy metal contamination in wastewater is considered for many industries, such as metal plating processes, mining operations, tanning, chlor-alkali industry, refrigerant plants, smelting operations, alloy industries, and storage battery industries. Toxic heavy metals are considered pollutants that have a direct and significant impact on humans, animals, and the environment in general. Industrial wastewater containing heavy metals can directly pollute groundwater resources and thus lead to a serious groundwater pollution problem. Water of high quality is essential for human life and water of acceptable quality is essential for agriculture, industrial, domestic and commercial uses (Renge *et al*,2012).

Heavy metals found in wastewater and industrial effluents are a major environmental pollution concern. Heavy metals are generally considered to be those with a density greater than 5 grams per cubic centimeter. Most of these elements fall into this category. They are highly soluble in water and are known toxic substances and carcinogenic agents for humans (Zhao *et al*,2016). The conventional treatment techniques for removing heavy metals are chemical precipitation, ion exchange, oxidation, reduction, reverse osmosis, electrolysis and ultrafiltration. These techniques have side effects, such as generating a large amount of sludge, less efficient operation, sensitive operating conditions, and costly disposal. Thus, the adsorption method is a relatively new process that is emerging as a potential alternative for heavy metal removal (Renu *et al*,2017). Adsorption is one of the most widely used techniques in the process of removing heavy metals from water due to its low cost and simple operation (Yang *et al*,2019). Removal of pollutants by adsorbents can be achieved through ion exchange mechanism, hydrogen bonding interactions, ionic dipole interactions or by protonation interactions. Currently, biomaterials such as plant extracts have been explored as alternative sorbents that meet many of the mentioned requirements, Sorbents based on plant extracts are desirable because they are cost-effective, non-toxic, sustainable and multifunctional since plants contain a wide range of plant compounds, Recently, nanoparticle composites prepared by bio reduction of plant extracts have been used to remove toxic heavy metals and organic pollutants from wastewater. (Ituen *et al*,2021). Recently, there has been increasing awareness of the pollution of rivers with various pollutants, especially pesticides, hydrocarbons and heavy metals (Moyel *et al*,2015).

The aim of the study is focused on three different stations of the Ashar river (south of Iraq, Basra city) including study the physicochemical properties of the Ashar river water in multiple stations, in addition to using extracts of local plant *Salvia rosmarinus* (Rosemary) extracts (water and ethanol extracts) to get rid of heavy elements presents in the water.

2. Materials and methods

2.1 Materials

Rosemary powder, Soxhlet meter (China), pH meter (China), Jar tester (India), River water samples, HCl 36% (India), NaOH 99.8% (India), Water bath (Germany), Rotary evaporator (China).

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3. Method

3.1 Water sampling:

The most polluted internal rivers of Basra city were identified. It was found that the reports prepared by local and international organizations to measure the amount of pollution in the Ashar River, which is considered one of the oldest tributary rivers of the Shatt al-Arab Canal, and based on studies conducted by (Akesh,2017). And (Alhello *et al*,2020). And (Kahami *et al*,2023). Samples were taken as previously mentioned from the Ashar River in three different stations (Figure 1) along the river in the middle of the city, where pollution has been observed to have increased recently.

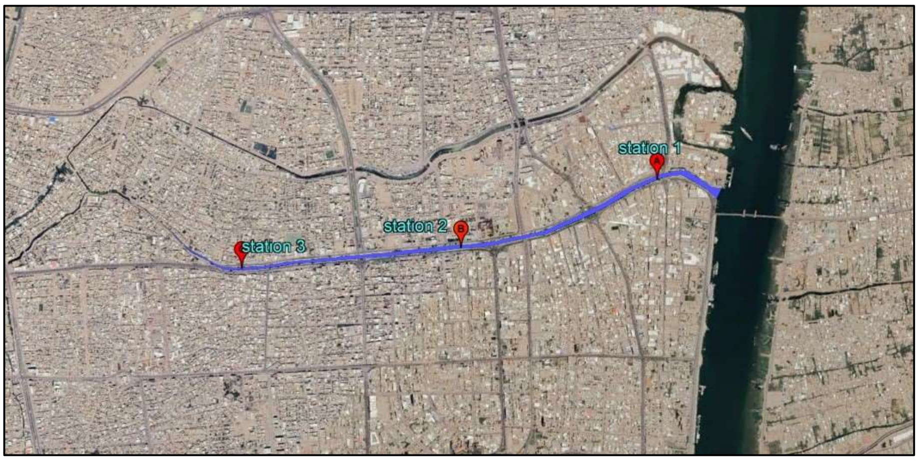

Figure 1. Location of study area

3.2 Plant collection and preparation

Rosemary was collected locally from selected nurseries in Basra city to ensure that it was not contaminated with heavy elements. Ten anvils were used to remove the plant from the soil, wash it, remove the roots and flowers, and dry the remaining green parts, the stem and leaves only. The plant was placed away from sunlight at room temperature to dry, where it took 5 days to dry completely. After that, it was ground in an electric grinder and placed in boxes to be ready for extraction.

3.3 Extraction method:

The method of extracting active compounds from plants was adopted by the Soxhlet extraction device (Dib *et al*,2021). It took eight hours for the rosemary plant to fully extract, using both aqueous and alcoholic extraction methods. 10 g of rosemary powder was weighed using a sensitive balance and placed in a paper thimble and fixed in the main chamber of the device (Extraction Tube). Then 150 mL of distilled water was added in the case of aqueous extraction and 150 mL of ethanol in the case of alcoholic extraction. Both the aqueous and alcoholic solvents were heated to boiling point to start the extraction process. Then the volume of the extracted solvent was reduced using a rotary evaporator to obtain a concentrated extract of about 20 mL was left to dry in room temperature (Figure 2). Then the resulting extract was weighed, and the extraction rate was calculated according to the following equation 1, as shown in Table 1.

$$Y\% = \left(\frac{We}{Wi}\right) \times 100 \tag{1}$$

Y%= Extraction yield

Wi= Weight the powder before extraction

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We= Weight of the resulting extract

Table 1. Rosemary Extraction Yield.

Extract type	Weight of the resulting extract (g)	Extraction yield (%)
Alcoholic rosemary	2.5	25
Water rosemary	2	20

Figure 2. Rosemary alcohol extract after drying

3.4 Gas Chromatography–Mass Spectrometry (GC-MS) Analysis

The Nahran Omar Laboratories of Basra Oil Company located in the north of the city were used to conduct the sample analysis by (GC-MS) using an Agilent Technologies 7890B GC system coupled with an Agilent Technologies 5977A MSD with an EI signal detector, using HP-5ms 5% phenyl, 95% methyl siloxane (30m * 250um * 0.25), where first the oven temperature was set at 40 °C for 4 minutes then it was raised to 10 °C / min to 300 °C for 20 minutes, where the helium carrier gas flow rate was 1 ml / min and the purge flow was 0.3 ml / min. Then the injection mode was divided with an injection temperature of 290 °C where the injection sample volume was 0.5 µl. Mass spectrometry, ion source temperature 230 °C, mass range 44-650 m/z, and data were recorded through NIST 2020 and 2014 databases, as an additional source to confirm the identity of the compound.

3.5 FT-IR Spectrophotometry Analysis:

Infrared spectroscopy was used to identify the phytochemicals through the active groups present in them and compared with GC-MS analysis. Infrared spectra of the plant extracts were tested in the 4000–400 cm^{-1} region using Shimadzu FTIR–8400 spectrophotometer applying the KBr disc technique.

3.6 Water Treatment

A jar test device was used by filling each beaker in the jar test with 100 mL of river water sample. Different concentrations of extract were added to each beaker from 0.01 - 0.04 g. The stirring speed was set at 200 rpm for 4 min followed by 40 rpm for 30 min (Lea, 2010).

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3.7 Optimization of biosorption parameters:

The required parameters for the optimum pH value for heavy metal adsorption were determined using 100 mL of mineral water. The biosorption experiment was carried out by the jar tester method to optimize the pH, metal ion concentration, contact time and at fixed temperature. Batch studies were carried out using different concentrations of plant extract 0.01 - 0.04 g, pH 4 - 9, and contact time 15 min - 60 min.

4. Results and Discussion

4.1 Gas Chromatography–Mass Spectrometry (GC-MS) results

The results of the examination of the plant extract sample showed the presence of many types of aromatic and aliphatic compounds, as Table (2) and Figure (3) indicate the components of the rosemary extract, as they showed that there are compounds with high concentrations belonging to the organic classes of cyclic esters, carboxylic acids, alcohols and phenols, whose concentrations ranged from 13.21-1.10%. Table (3) and Figure (4) represent the plant components of the aqueous rosemary extract, where the presence of active compounds, cyclic esters, alcohols, and ketones, in addition to aromatic and aliphatic compounds, is also noted, and their concentrations ranged from 15.24% to 1.45%.

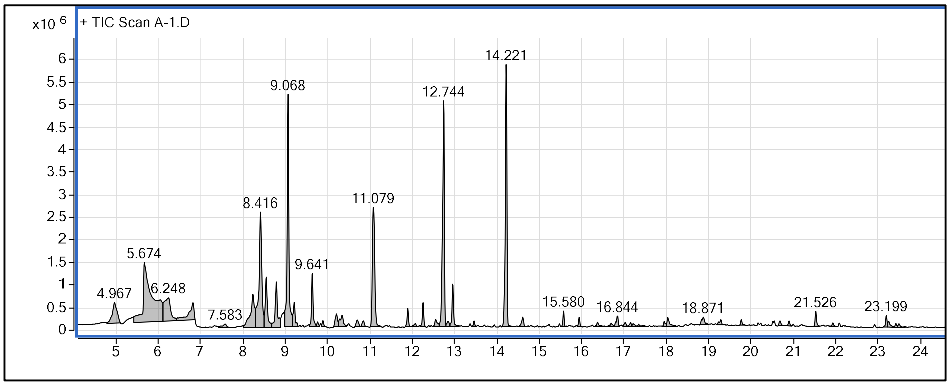


Figure. 3. GC-MS of alcoholic extract of rosemary.

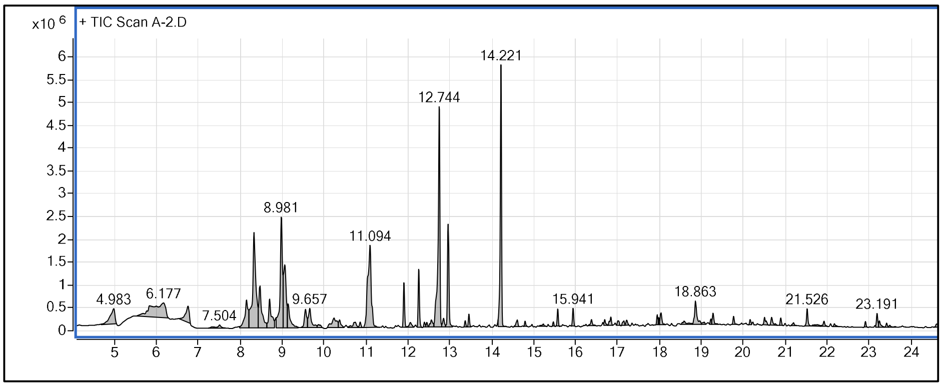
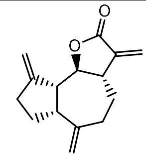
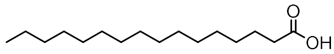
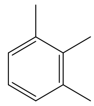
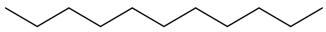
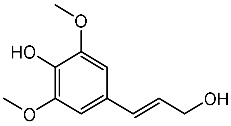
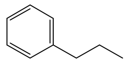
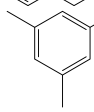
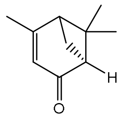
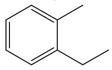
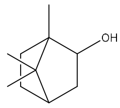
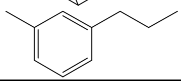


Figure 4. GC-MS of rosemary aqueous extract.

Table 2. GC-MS data of alcoholic extract of rosemary

Peak	**R.T.**	**Area Pct**	**Library/ID**	**Chemical structure**
26	12.744	13.2114	Dehydrocostus lactone	
30	14.221	11.8603	n-Hexadecanoic acid	
8	9.068	10.7558	Benzene, 1,2,3-trimethyl-	
20	11.079	9.6069	Undecane	
5	8.416	7.0454	trans-Sinapyl alcohol	
4	8.235	4.8544	Benzene, propyl-	
6	8.557	2.5394	Mesitylene	
28	12.956	2.1021	Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-trimethyl-, (1S)-	
7	8.785	1.9917	Benzene, 1-ethyl-2-methyl-	
24	12.257	1.2582	endo-Borneol	
14	10.207	1.1016	Benzene, 1-methyl-3-propyl-	

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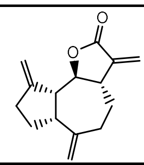
26	12.744	13.2114	Dehydrocostus lactone	

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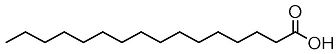
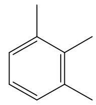
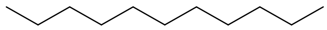
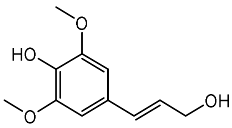
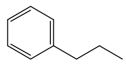
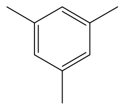
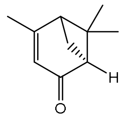
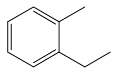
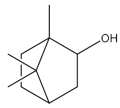
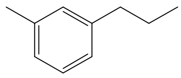
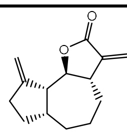
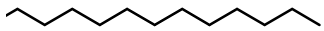
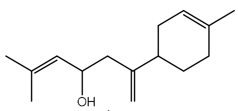
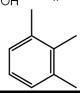
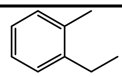
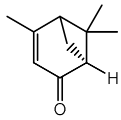
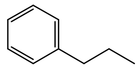
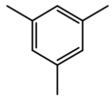
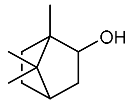
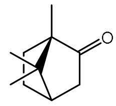
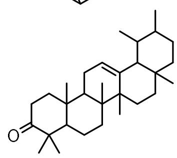
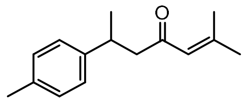
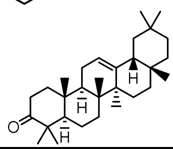
30	14.221	11.8603	n-Hexadecanoic acid	
8	9.068	10.7558	Benzene, 1,2,3-trimethyl-	
20	11.079	9.6069	Undecane	
5	8.416	7.0454	trans-Sinapyl alcohol	
4	8.235	4.8544	Benzene, propyl-	
6	8.557	2.5394	Mesitylene	
28	12.956	2.1021	Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-trimethyl-, (1S)-	
7	8.785	1.9917	Benzene, 1-ethyl-2-methyl-	
24	12.257	1.2582	endo-Borneol	
14	10.207	1.1016	Benzene, 1-methyl-3-propyl-	

Peak	R.T.	Area Pct	Library/ID	Chemical structure
22	12.744	15.2459	Dehydrocostus lactone	
29	14.221	13.443	Tridecane	
14	11.094	11.2173	6-Methyl-2-(4-methylcyclohex-3-en-1-yl)hepta-1,5-dien-4-ol	
6	8.981	6.8811	Benzene, 1,2,3-trimethyl-	

3	8.329	6.2377	Benzene, 1-ethyl-2-methyl-	
24	12.964	4.8158	Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-trimethyl-, (1S)-	
2	8.156	3.9682	Benzene, propyl-	
4	8.471	3.1244	Mesitylene	
18	12.257	2.879	endo-Borneol	
16	11.903	2.0587	Bicyclo[2.2.1]heptan-2-one, 1,7,7-trimethyl-, (1S)-	
69	33.223	1.7713	alpha-Amyrone	
45	18.863	1.565	ar-Turmerone	
68	32.791	1.4544	beta-Amyrone	

4.2 FT-IR Spectrophotometry results

The infrared spectroscopy data of the alcoholic extract of rosemary showed prominent absorption bands at 3386, 2861, 1687, 1452 and 1033 cm-1 (Fig. 5), while the nanoparticles formed using the aqueous extract of rosemary showed 3386, 2857, 1688, 1600, 1516, 1267 and 1034 cm-1 (Fig. 6). The absorption bands at 3286 cm-1 can be explained as O-H stretching vibration of carboxylic acid, alcohols or phenols (Daish et al,2024). The small band at 2861 and 2857 cm-1 is due to the vibrational frequency of alkane groups C-H. Also, absorption bands observed within 1687 cm-1 are due to the carboxyl group bond of esters or carboxylic acids.

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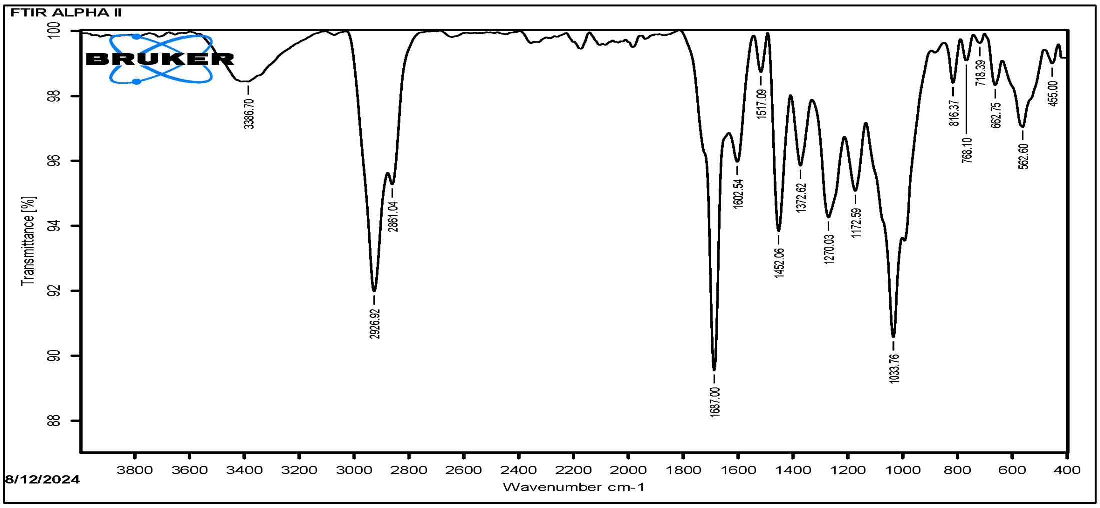

Figure 5. FT-IR of alcoholic extract of rosemary.

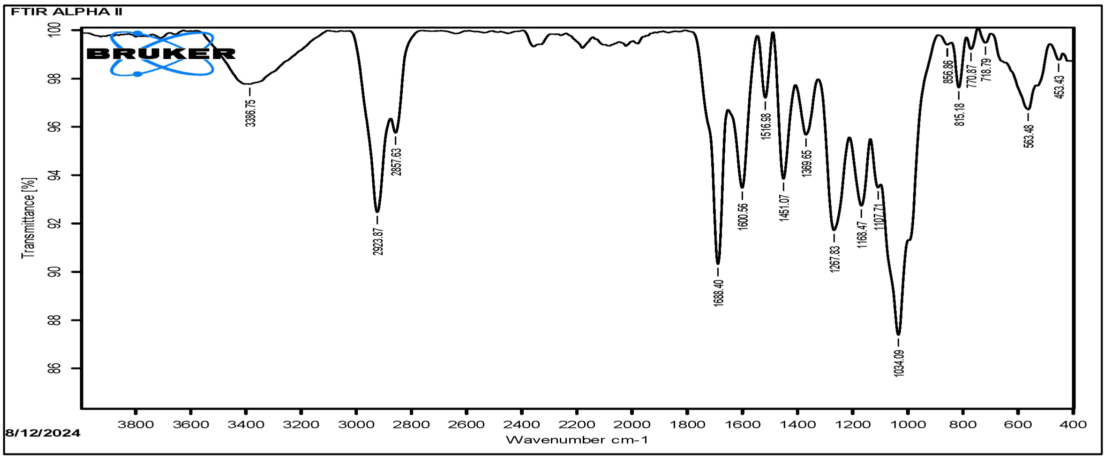

Figure 6. FT-IR of rosemary aqueous extract.

4.3 Removal of heavy metals by rosemary (RE)

4.3.1 Effect of contact time

After collecting the selected sample from Al-Ashar River, a test was conducted on the effect of contact time on the removal of heavy metals from the river water sample for a period 15 - 60 min. Table 4 presents the results obtained in this regard. As can be inferred, the results of the removal of heavy metals (arsenic, boron, manganese, iron and strontium) using both types of alcoholic and aqueous extracts showed that the highest removal rate at contact time 30 min was for all elements, as shown in Figure 7.

Table 4. Effect of contact time on removal percentage of heavy metals using 0.2 g/L of rosemary (RE) extracts

Extract type	Contact time (min)	Removing Efficiency (%)				
		As	B	Mn	Fe	Sr
Alcoholic	15	53.22	77.21	76.63	80.57	14.39
	30	100	83.53	100	100	14.61
	45	100	83.87	100	100	14.73
	60	100	83.77	100	100	14.67
Aqueous	15	62.18	72.06	80.83	89.36	70.45
	30	100	77.42	100	100	77.21
	45	100	77.81	100	100	77.06
	60	100	77.62	100	100	77.25

The process of absorbing heavy elements from water using the active compounds present in the plant extract depends on several basic factors, including sufficient time for interaction, diffusion, distribution and accumulation of these elements in the water. This is what the results in Table 4 showed, where at the beginning of the process at 15 min, the removal rate was low, and this is because the plant extract was not saturated with the targeted heavy element. The heavy elements may also be unevenly distributed in the water. As time passes, specifically at 30 min, we notice the highest removal rate of the elements, and this is because the distribution became more homogeneous, which allowed for increased opportunities for interaction with the plant extract. However, after a longer time at 60 min, the heavy elements begin to spread again in the water because of reaching a state of saturation by the extract molecules, which leads to their separation from the surface of the molecules to which they were attached. Especially since the absorption processes or chemical interaction between the plant extract and the heavy elements need a certain time to reach a state of equilibrium.

4.3.2 Effect of pH level

The effect of pH levels (4, 6, 7, 8, 9) on the removal efficiency of heavy metals was tested using two types of rosemary extracts, and the results are presented in Table 5. The data on the removal of metal ions show a clear convergence in the removal rate at pH 7 and 8. At pH 8, the removal rate increases significantly, which may be attributed to the fact that the basic pH increases the ability of plant extracts to absorb heavy elements due to the change in electrical charges on the surface of the adsorbents, which enhances the interaction with heavy elements (Huang *et al*,2017). In addition, many heavy elements are less soluble in a basic environment compared to an acidic environment. That is, at pH 8, an accumulation of insoluble complexes or hydroxides may form that can be removed from water more easily.

Table 5. Effect of pH level on removal percentage of heavy metals using 0.2 g/L of rosemary (RE) extracts

Extract type	pH	Removing Efficiency (%)				
		As	B	Mn	Fe	Sr
Alcoholic	4	53.28	65.71	49.35	42.49	11.06
	6	69.02	70.25	53.55	58.61	10.76
	7	77.02	78.12	86.09	89.35	20.94
	8	100	83.57	100	100	23.92
	9	71.44	79.33	60.59	68.40	13.50
Aqueous	4	55.61	68.12	34.66	39.56	75.35
	6	61.92	72.97	63.70	58.62	75.73
	7	72.35	78.12	76.65	82.04	76.17

8	100	78.73	100	100	77.10
9	71.44	71.16	71.39	73.25	76.28

4.3.3 Effect of extract Concentration

The concentration selection effect was conducted to obtain the best efficiency for removing heavy elements using the two types of extract. The quantities 0.1 – 0.4 g/L were tested as shown in Tables 6 and 7 and using the jar test device. The results showed that the appropriate extract quantity for the alcoholic extract of rosemary is 0.2g/L, while the appropriate extract quantity to achieve the best removal rate for the aqueous extract was 0.3g/L. The reason for the difference in the extract quantity between the two types may be due to the difference in the active compounds extracted from the plant. The results of GC-Ms and FT-IR showed that the alcoholic extract contains high concentrations of carboxylic acids, alcohols and phenols, which ranged from 13.21-1.10% which may be acted as complexing agents with metals. While the aqueous extract contained high concentrations of alcohols and ketones in addition to aromatic and aliphatic compounds, and their concentrations ranged from 15.24%-1.45%. This may be the possible reason why lower concentration of alcoholic extract became more efficient while aqueous extract required higher dosage to achieve optimum removal efficiency of heavy metals from water.

Table 6. Effect of Concentration on removal of heavy metals using Aqueous rosemary (RE)

Concentration (g/L)	Removing Efficiency (%)				
	As	B	Mn	Fe	Sr
0.01	33.76	19.34	25.10	40.87	37.88
0.02	41.19	45.43	57.32	74.35	31.86
0.03	100.00	77.42	100.00	100.00	77.22
0.04	100.00	73.63	100.00	100.00	63.52

Table 7. Effect of concentration on removal of heavy metals using alcoholic rosemary (RE)

Concentration (g/L)	Removing Efficiency (%)				
	As	B	Mn	Fe	Sr
0.01	78.18	64.43	63.70	51.28	11.73
0.02	100.00	83.54	100.00	100.00	14.62
0.03	100.00	85.09	100.00	100.00	14.71
0.04	100.00	84.48	100.00	100.00	14.74

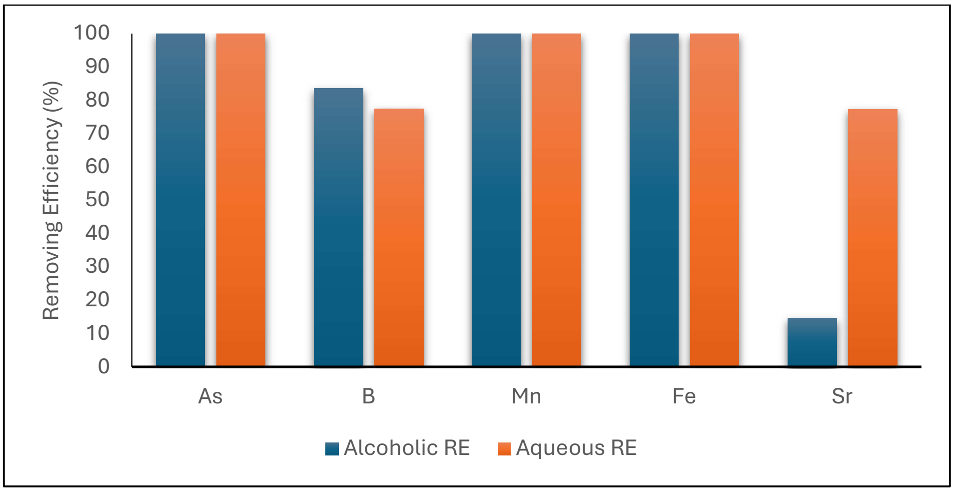

Figure 7. Removing efficiency of alcoholic (0.2 g/L) and Aqueous (0.3 g/L) rosemary (RE) extracts on heavy metals at pH 8 and 30 min of contact time

From the results of adsorption capacity of the alcoholic and aqueous extracts of rosemary on toxic heavy metal ions, Fe, B, As, Sr, Mn, we noted some important points. The extract characteristics shown by FTIR and GC-MS results indicate that the rosemary extract has good adsorption properties and is effective in removing heavy metals from aqueous solutions. The results also showed the iron removal rate in the three stations reached 100%, and this result was completely consistent with what was reached by (Ali, 2020). While the percentage of boron removal was 92%, which is what reached (Azhar, 2012). It showed a 100% removal rate of manganese, and this result is relatively close to what (Melaku, 2023). reached when using the Moringa plant to remove manganese from wastewater. The results showed a 100% removal rate of arsenic, which was approximately consistent with the results reached by (Karimi *et al*,2019). While the strontium element showed a weak response to removal, as the removal rate was 29% (Hassan *et al*,2020). The effects of different parameters known to influence the adsorption were investigated, and the results revealed that under the specified experimental conditions, all elements showed the highest removal percentage at pH 8, contact time 30 min, and concentrations of 0.2 g/L for the alcoholic extract and 0.3 g/L for the aqueous extract.

5. Conclusion

Rosemary has been shown to be a good means of removing heavy metals from highly polluted river water. Through its two parts, alcoholic and aqueous extract, this plant was able to achieve excellent rates of heavy metal removal, and with the increase of the heavy metal removal rate until the optimum concentration was used. Moreover, the removal of elements was within the optimum pH of river water, where the pH value was not affected, and there was no need to adjust the pH. Therefore, rosemary extract can be recommended as a good coagulant and heavy metal removal agent from water.

Conflict of interest statement

The authors declared no conflict of interest.

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