

Cadmium ion (Cd^{+2}) removal from produced water using ion technique: Amberlite IR-120H and modified amberlite IRA-400 Cl^- resins

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International Journal of Science and Research Archive, 2024, 13(02), 1331–1338

Publication history: Received on 10 October 2024; revised on 19 November 2024; accepted on 22 November 2024

Article DOI: <https://doi.org/10.30574/ijrsra.2024.13.2.2253>

Abstract

This study to characterizing the physical and chemical properties of the produced water in Nafoora field, Gulf Company in the Libyan, also, level of concentrations of selected heavy metals have been measured. To do that, different techniques have used. Results have showed pH, Conductivity, salinity, TDS, and turbidity levels were 6.2 ± 0.19 ; $1473 \mu\text{S}/\text{cm}$; 1.8; $79000 \text{ mg}/\text{l}$; and $40 \pm 4.18 \text{ NTU}$ respectively. The levels of the anions ranged as follows: Cl^- ($15300 \text{ mg}/\text{l}$); NO_3^- ($1 \text{ mg}/\text{l}$) and SO_4^{2-} ($120 \text{ mg}/\text{L}$). While the concentration of some ions metals were found as follows: K^+ ($1400 \text{ mg}/\text{l}$); Na^+ ($360000 \text{ mg}/\text{L}$) and Ca^{+2} ($8570 \text{ mg}/\text{l}$). Some heavy metals were also estimated and found as follows: Cd ($1.2 \text{ mg}/\text{l}$); Fe ($3.1 \text{ mg}/\text{l}$); Cu ($2.3 \text{ mg}/\text{l}$); Ni ($0.3 \text{ mg}/\text{l}$) and Pb ($0.1 \text{ mg}/\text{l}$). Ion exchange technique was used to remove the cadmium metal ion from produced water using Amberlite IR-120H and modified Amberlite IRA-400 Cl^- resins. Optimization pH, temperature and contact time using lame Atomic Absorption Spectrophotometer (AAS) approach for measuring the concentrations of Cd ion. Experiments showed that the Amberlite IR-120H resin has maximum removal efficiency for Cd at pH 7, 80°C and 30 min respectively, while pH 7, 80°C and 120 min are the most appropriate conditions for modified Amberlite IRA-400 Cl^- resin. The results obtained show that the Amberlite IR 120 H as a strong cation-exchange resin has greater Cd removal ability (96.3%) and fast as compared to the modified Amberlite IRA-400 Cl^- resin (73%).

Keywords: FAAS; Cations, Anions; Resin; Cadmium; Produced water

1. Introduction

Produced water is generally hot and more saline than seawater, and it is produced as by product of oil and gas separtion operations. This water may include water from the reservoir, water injected into the formation, and any chemicals or surfactant added during the production and treatment processes [1,2]. In general, produced water has a complex composition that can be broadly categorised into organic and inorganic compounds including heavy metals, dispersed and dissolved oils, salts, grease, dissolved gases, radionuclides, waxes, dissolved oxygen, formation solids and microorganisms [3,4]. It may also contain process chemicals such as corrosion inhibitors and demulsifiers added during the operation of the well [1]. The types of chemicals and the concentration of the metals found in produced water can vary depending on the geology and age of the reservoir from which the oil and gas are produced. The most common metals detected in produced waters are cadmium, lead, zinc, iron, manganese, and barium [4,5].

The pollution caused by the discharging of produced water containing toxic metals has become a major environmental problem due to the importance of petroleum in energetic production [6]. The oil and gas industry generates around 250 million barrels of produced water each day worldwide which results in the release of 2.4 million tons of cadmium, arsenic, chromium, mercury, copper, nickel, lead, vanadium, selenium, and zinc annually. Unfortunately, more than 40% of this water is released into the environment without proper treatment [7]. This improper disposal can cause metals to spread through the food chain, leading to various health, environmental economic impacts [8]. H.R.H. Prince

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Alexander of the Netherlands stated that more than 80% of produced water is released into water sources without treatment, hindering efforts to combat pollution [9]. This untreated wastewater negatively affects human health, economic development, environmental protection, agriculture, and the sustainability of ecosystems [9].

Among the heavy metals found in produced water, cadmium is of particular concern due to its high toxicity, non-biodegradability, hydrophilic property ability to bioaccumulate in aquatic ecosystems, which makes it to easily enter the food chain [10,11]. Cadmium accumulates in the pancreas, kidneys, glands and intestines, modifying the metabolism of the elements necessary for the body, such as magnesium, copper, zinc, calcium, iron, and selenium. The main adverse effects in humans exposed to high concentrations of cadmium compounds include damage to the kidney, liver, lungs, placenta, endocrine tissues as well as reproductive problems. Additionally, data on Cd toxicity show that it can cause anosmia, apoptosis, anemia, eosinophilia, fractures and osteoporosis, diabetes mellitus, chronic pulmonary problems, oncogenes activation and Itai-Itai disease [12,13,14].

In order to protect the environment and living organisms, many countries and international organizations have established a set strict limits on the allowable concentrations of cadmium in concentrations of cadmium in drinking water and wastewater. For example, the WHO recommend a maximum cadmium concentration of 0.003 ppm in both wastewater and soils for agriculture [15,16]. While the maximum limit of Cd recommended by The U.S. Environmental Protection Agency (EPA) for food intake is 1 mg/kg/ day and drinking water is 0.5 mg/kg/day [11,17].

Several methods are being used for removing heavy metals from produced waters, such as membrane filtration, precipitation, sedimentation, ion exchange, electrocoagulation and electrochemical techniques [18]. Among these techniques, ion exchange is often regarded as the most effective option for various applications due to its high selectivity, higher thermal stability, suitability for a broad range of pH levels, insolubility in most aqueous and organic solutions as well as ability to regenerate and recovery of the removed metals [11,19,20,21].

Several researches have been conducted on the removal and extraction of toxic metals using a variety of resins with distinct functional groups [11,22,23].

The aim of this study was evaluation some physical and chemical properties of untreated produced water from Nafoura field-Gulf company in the Libyan Desert and examination the efficiency of Amberlite IR-120 H and IRA 400 CL⁻ ion exchange resins on the removal of cadmium as well as factors that affect the ion exchange process, such as solution pH, temperature and residence time.

2. Materials and Methods

2.1. Chemicals and reagents

Table 1 Properties of Amberlite IR 120 and Amberlite IRA-400 ion exchange resins

Parameter	Amberlite IR-120 resin	Amberlite IRA-400 resin
Ionic form	H ⁺	Cl ⁻
Functional group	SO ₃ ⁻	-N ⁺ R ₃
Matrix type	polystyrene DVB	Polystyrene DVB
Resin type	Macroporous strong acidic cation exchange resin	Macroporous, strong basic anion exchange resin
Particle size (mm)	0.6 - 0.8	0.3-0.9
Moisture (%)	45 - 50	40-47
Max. operating temp.(°C)	120 - 150	80
pH range	0 - 14	0 - 14
Total exchange capacity (meq/mL)	2.0	2.6-3

All the chemicals used in the study were analytical grade and were used without further. Stock solution of 100 ppm of cadmium (II) was prepared by dissolving the required amount of $\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ (Sigma-Aldrich) in distilled water, and solution of desired concentration was prepared by diluting the stock solution. Pure nitric acid (99%), hydrochloric acid (99%) and sodium salt of EDTA were supplied by Merck (USA) and other chemicals were ordered from Sigma-Aldrich. Amberlite IR-120 in hydrogen form and Amberlite IRA-400 resin in chloride form were acquired from Sigma-Aldrich (USA) The properties of these resins are given in Table 1

2.2. Instrumental and devices

ContraAA® 300 Atomic Absorption Spectrometer equipped with deuterium background correction and cadmium hollow cathode lamp was used for heavy metals analysis, under the following conditions: lamp current: 8 mA; flame: air/acetylene. A pH meter (Mettler Toledo) was used for pH measurement. Flame Photometer, turbidity meter, chemical oxygen demand (COD) unit, thermometer, oxygen cylinder.

2.3. Sampling

100 litres of untreated produced water sample was randomly collected in August, 2022, from Four different points of discharge lake in Nafoora field-Gulf company in the Libyan desert. This sample was collected in a sterilized glass bottle, covered and transported to the laboratory. After some physical and chemical properties of untreated produced water was measured, the sample was filtered using filtration papers. This process has been repeated several times to obtain water free from impurities.

2.4. Modification of Amberlite IRA-400 Cl⁻ resin

Amberlite IRA 400 Cl⁻ is a strongly basic, clear gel, anion exchange resin. In order to increase the efficiency of the recovery, Chloride ion that is associated with Amberlite IRA-400 Cl⁻ resin was substituted with EDTA²⁻ by shaking the resin with 0.01 M Na₂ EDTA solution for 24 h. The resin was separated from the solution and washed several times using demineralized water until the solution was found free from chloride ion and then was dried in an electric oven (60°C). To ensure the replacement was completed, the received solution was treated using silver nitrate (Mohr method) until obtain clear solution free from precipitate. The modified Amberlite IRA 400 Cl⁻ was obtained as insoluble reddish brown spherical granules and has two oxygen of carboxylic group and two nitrogen of amine group so, act as a cation exchange resin [24].

2.5. Column preparation.

To find the most selective resin for the removing of Cd(II) in the produced water, glass chromatographic columns were cleaned and dried. Each type of resins Amberlite IR 120H and modified Amberlite IRA 400 Cl⁻ was packed in these separated columns and then 5% nitric acid solution was pumped through the all columns, causing the resin to shrink, and the excess space was partially filled with additional resin. The total measured weight of the packed resins were (11.3 g). Before purging the Cd (II) sample through the column, the resin was cleaned with 5% nitric acid to remove any contaminating metal flushing. Ion exchange behaviour of these resins were investigated by passing 50 ml Cd(II) solution over 11.3 g of these resins packed separately in a columns. After that, the sorbate from the each columns were eluted with dilute HNO₃ solution until no further cadmium removal was detected.

A standard calibration curve was drawn to calculate the Cd concentration using an Atomic Absorption Spectrophotometer at wavelengths of 228.8 nm. The formula used to determine Cd removal efficiency (%) is as follows [23,25]:

$$\text{Removal (\%)} = \frac{C_i - C_f}{C_i} * 100$$

Where C_i = initial metal ion concentration. C_f = the final metal ion concentration

3. Results and Discussion

3.1. The physiochemical properties of Nafoora field-Gulf company untreated produced water

The physicochemical and metallic evaluation of the effluents provides an idea of the concentration of toxic metals and ions, pollutants contained in the produced water to be treated. The physicochemical characterization parameters of untreated produced water from the Nafoora field-Gulf company reported in tables 2 and 3. Standard methods were used, and sample temperature was kept between 20.0 to 25 °C. The results show that, the produced water sample has a

pH value of 6.2 ± 0.19 which is considered to be within the acceptable limits of 6-9, especially since the pH of pure distilled water is 7 [26]. The salt content of Produced water is measured in terms of the salinity, Electrical Conductivity (EC), and Total dissolved solid (TDS). The EC value is $1473 \mu\text{S}/\text{cm}$, which is within the limit of $4000 \mu\text{S}/\text{cm}$. This is significant considering that typical drinking water has an EC range of 1000 to $1500 \mu\text{S}/\text{cm}$ [26]. The mean TDS and turbidity values are recorded at $97000 \text{ mg}/\text{l}$ and 40 NTU , respectively, which exceed the acceptable limit of $5000 \text{ mg}/\text{l}$ for TDS and 15 for turbidity. It is important to note that the total hardness, measured as calcium carbonate, exceeded the acceptable limits of 120 to $170 \text{ mg}/\text{L}$, with a recorded value of $420 \text{ mg}/\text{L}$ [26]. As showed in Table 3, the concentration of heavy metal in untreated Nafoora field-Gulf company produced water is found to be higher than the WHO, 2008 heavy metal limits for drinking water. The prescribed limits for drinking water by WHO are as follows: Cd ($0.003 \text{ mg}/\text{L}$), Fe ($2.0 \text{ mg}/\text{L}$), Cu ($2.0 \text{ mg}/\text{L}$), Ni ($0.07 \text{ mg}/\text{L}$) and Pb ($0.01 \text{ mg}/\text{L}$) [27].

Table 2 Physical properties of produced water of Nafoora field-Gulf company

Parameter	value	Method of test
pH at 25°C	6.2 ± 0.19	pH meter (Mettler Toledo)
Salinity calculated as % NaCl	1.8	Thermo Scientific (Orion 720A)
Conductivity $\mu\text{S}/\text{cm}$ at 25°C	1473	Thermo Scientific (Orion 720A)
TDS(mg/l)	97000	Thermo Scientific (Orion 720A)
Turbidity(NTU)	40 ± 4.18	TurbidimeterJenway 6035
DO(mg/l)	10.2 ± 0.50	DO2 Jenway9500

Table 3 Chemical properties of produced water of Nafoora field-Gulf company

Parameter	Concentration (mg/l)	Method of test
Total hardness	420	CaCO_3 (mg/l)was measured by titration with EDTA according to ASTM Standard Method D 1126 - 02
Nitrate	1 ± 0.57	Standard methods for the examination of waters and wastewaters. APHA AWWA-WEF, Washington, DC.
Chloride	15300 ± 0.44	Determined using the silver nitrate titration method following ASTM D4458-009.
Sulphate	120 ± 0.18	Measured using ASTM D 516-68.
Calcium	8570 ± 0.86	Measured by titration with standard acid according to standard method D 1067-06.
Sodium	360000 ± 0.38	Flame photometer
Potassium	1400 ± 0.76	Flame photometer
Cadimium	1.2	Absorption Spectrophotometer at wavelengths 228.8 nm.
Iron	3.1	Absorption Spectrophotometer at the wavelength of 228.8 nm.
Copper	2.3	Absorption Spectrophotometer at the wavelength of 248.3 nm.
Nikel	0.3	Absorption Spectrophotometer at the wavelength of 396 nm.
Lead	0.1	Absorption Spectrophotometer at the wavelength of 283.3 nm.

3.2. Optimizations of ion exchange technique

Amberlite IR120 H and modified Amberlite IRA 400 Cl^- resins were studied in removal of Cd(II) ions from aqueous solution under different experiment conditions such as pH, temperature and contact time. The experimental results and the relevant observations are discussed in the following sections.

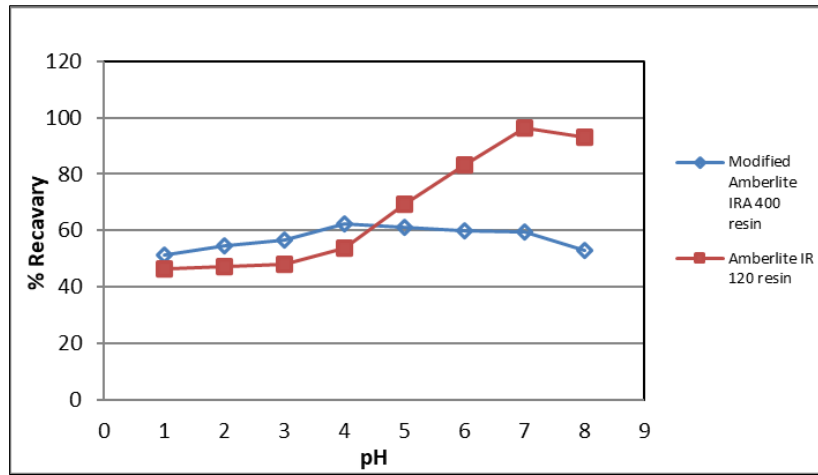


Figure 1 Effect of pH on Cd (II) recovery using Amberlite IR 120 H and modified Amberlite IRA 400 Cl⁻ resins

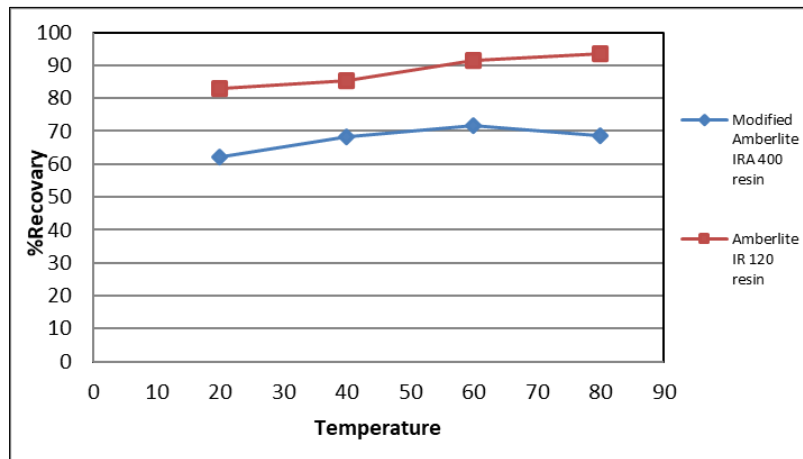


Figure 2 Effect of temperature on Cd (II) recovery using Amberlite IR 120 H and modified Amberlite IRA 400 Cl⁻ resins

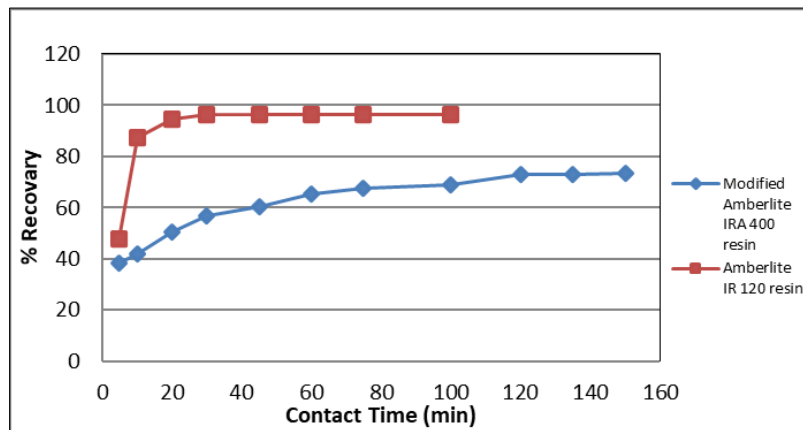


Figure 3 Effect of Contact time on Cd (II) recovery using Amberlite IR 120 H and modified Amberlite IRA 400 Cl⁻ resins

3.2.1. The effect of pH on recovery of Cd(II)

It is well known that the pH of the aqueous solutions is an important factors in the ion exchange process because it affects the degree of ionization of the resin, the concentration of the counter ions on the resin's functional groups, and the metal ions solubility during the exchange process [24, 28, 29]. The effect of pH on the removal efficiency of Cd(II) ions by the Amberlite IR120H and modified Amberlite IRA 400 Cl⁻ resins was investigated separately in the pH range 1.0– 8.0 at a constant Cd(II) ions concentration (10 ppm) and a constant amount of the resins (11.3 g) at room temperature, as shown in Figure 1.

The pH vs % recovery of the Cd (ii) ion curves indicated that the removal percentage increased with the rising pH values for both type of resin. The removal rate of Cd (II) increased slowly from 46.2 to 96.3% for Amberlite IR 120 H resin and increases from 51.4 to 62.6 % for modified Amberlite IRA 400 Cl⁻ , as the pH of solution increased from 1 to 7. These results validate that Amberlite IR 120 H, as a strong cation-exchange resin, has greater recovery efficiency for the Cd (II) than modified Amberlite IRA 400 Cl⁻ resin. At low pH values, cadmium removal was found to decrease because the resin's ion exchange surface area was more protonated and the competition on the resin exchange sites between Cd ions and H⁺, which leads to competition between Cd ions and H⁺ for the resin exchange sites, thereby reducing the number of binding sites available for Cd [30,31]. Conversely, at pH higher than 7, the amount of free Cd ion decreased due to precipitation as Cd(OH)₂, resulting in a decrease in the recovery of Cd by both resins [32]. This result is consistent with those of study conducted by Demirbas *et al.*, (2005), on the removal of Cu(II), Zn(II), Ni(II), Pb(II), and Cd(II) from aqueoussolution on Amberlite IR-120 H synthetic resin, where the greatest percentage removal was found in the pH range of 4–8, with about 99% of Cd(II) removed [33].

3.2.2. The effect of temperature on recovery of Cd(II)

The influence of temperature on removal of 10 ppm Cd(II) ions in aqueous media with the Amberlite IR120H and modified Amberlite IRA 400 Cl⁻ resins was studied in the temperature range of 20–80 °C, with 11.3g resin and a pH of 6. The results are shown in Figure 2. The findings indicate that metal removal increases with the temperature of the system. As can see in Figur 2, the removal efficiency of Cd increased with the rising temperature for both types of resin. The recovery for Cd using Amberlite IR120H resin increased from 82.8% to 93.4 % as the temperature rose from 20 to 80 °C, while the best recovery value of Cd with modified Amberlite IRA 400 CL⁻ resin was recorded at 71.8 % at 60°C. This result may be attributed to the endothermic nature of the ion-exchange process, where higher temperatures thermodynamically favor endothermic reactions [23]. A gradual increase in temperature significantly enhances the diffusion rate and mass transfer rate in metal ions in the solution, hereby increasing the likelihood of contact between the metal ions and the interior of the resin, which specifically boosts the removal rate [34]. Higher temperatures can reduce the viscosity of the solution and improve the solubility of Cd in the solution, allowing Cd ions to move more freely and interact more effectively with the resin [35]. It's important to note that excessively high temperatures may lead to thermal degradation of the resin, reducing its efficiency. Therefore, optimizing the temperature is crucial to maximize Cd recovery while minimizing resin degradation. This result is consistent with findings by Khalil *et al.* (2015) in their study on the equilibrium of cadmium ion removal using Amberlite IR 120 H sorbent [36].

3.2.3. The effect of Contact Time on recovery of Cd(II)

Contact time is a crucial factor for the effective use the ion exchange resins in practical application, as it requires rapid sorption [36]. The influence of contact time on the ion exchange of Cd ions by Amberlite IR 120 H and modified Amberlite IRA 400 Cl⁻ resins was studied at different time interval (2–120 min), using 11.3 g of resin with 50 ml of a 10 ppm Cd solution at pH 6 in different flasks. The experiments were conducted separately for each type of resin, and the results are shown in Figure 3. The results indicate that the percentage of Cd ion removal increased with prolonged contact time, reaching a plateau at 30 min with a removal rate of 96.3% for Amberlite IR120 H resin, and at 120 min with a removal rate of 73% for modified Amberlite IRA 400 Cl⁻ resin. As seen in Figure 3, the Cd exchange rate rose quickly during the first few minutes and then gradually increased until the equilibrium state was attained. After that, any additional increase in contact time had a negligible impact on the removal efficiency.

Initially, cadmium ions were rapidly adsorbed onto the resin due to the abundance of available binding sites, leading to a significant increase in the amount of cadmium accumulated on the resin surface. As the number of free sites decreased, the exchange rate slowed down due to repulsive forces between cadmium ions on the resin and in the solution. The exchange rate continued to decrease until it reached a plateau, indicating saturation [29,36,37].

4. Conclusion

The physical and chemical properties of untreated produced water samples from the Nafoora field-Gulf Company, such as pH, electrical conductivity (EC), total dissolved solids (TDS), total hardness, and heavy metal content, were analyzed. The results did not meet regulatory limits, particularly for cadmium, which was found to be above the acceptable limit of 0.003 mg/L set by the WHO. Without proper treatment, produced water poses risks to human health and other life forms. Amberlite IR 120 H and modified Amberlite IRA 400 Cl⁻ ion exchange resins were utilized as packing materials in column chromatography to remove cadmium ions from untreated produced water samples. Furthermore, this study investigated the effects of variables, including the pH of the cadmium solution, temperature, and contact time, to optimize the removal efficiency of the resins. The results of the investigation indicated that Amberlite IR120 H, as a cation-exchange resin, is more effective than modified Amberlite IRA 400 Cl⁻ for removing cadmium ions from produced water.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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