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ALUMINUM SEPARATION THROUGH REVERSE OSMOSIS: A CASE STUDY IN DRINKING WATER FROM THE GUADALQUIVIR BASIN, TARIJA, BOLIVIA

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The presence of heavy metals in the drinking water of the Guadalquivir basin, Tarija, Bolivia, has been generating a process of increasing and permanent contamination. This has caused water sources to be paralyzed, generating a water crisis in several months of the year. The separation of heavy metals is a complex process that, in many cases, conventional systems do not solve the problem. Reverse Osmosis is an efficient process that can be used as a definitive solution to water pollution problems and the recovery of consumption sources. This work shows the results of an experimental process in the separation of aluminum (Al) in drinking water from the Guadalquivir basin and its effectiveness in achieving the recovery of water quality. The research was carried out using an experimental reverse osmosis pilot plant with a Keensem brand polyamide membrane, model ULP-2540. The experimental plant has been designed to simulate the atmospheric and climatological conditions of the study areas. The system also evaluates the efficiency of contaminant separation and flux production.

Keywords: reverse osmosis; aluminum; water for consumption; heavy metals

SEPARACIÓN DE ALUMINIO MEDIANTE ÓSMOSIS INVERSA: UN ESTUDIO DE CASO EN AGUA POTABLE DE LA CUENCA DEL GUADALQUIVIR, TARIJA, BOLIVIA

La presencia de metales pesados en el agua potable de la cuenca del Guadalquivir, Tarija, Bolivia, viene generando un proceso de contaminación creciente y permanente. Esto ha provocado que fuentes de agua se paralicen generando crisis hídrica en varios meses del año. La separación de metales pesados es un proceso complejo que, en muchos casos, los sistemas convencionales no resuelven el problema. La Ósmosis Inversa es un proceso eficiente que puede ser utilizado como solución definitiva a los problemas de contaminación del agua y la recuperación de las fuentes de consumo. Este trabajo muestra los resultados de un proceso experimental en la separación de aluminio (Al) en agua potable de la cuenca del Guadalquivir y su eficacia para conseguir la recuperación de la calidad del agua. La investigación se llevó a cabo utilizando una planta experimental piloto de ósmosis inversa con una membrana tipo poliamida marca Keensem, modelo ULP-2540. La planta experimental ha sido diseñada para simular las condiciones atmosféricas y climatológicas de las zonas de estudio. El sistema también evalúa la eficiencia en la separación del contaminante y la producción del flux.

Palabras clave: ósmosis inversa; aluminio; agua de consumo; metales pesados

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1. Introduction

According to the United Nations, an estimated 1.2 billion people currently live in areas with water scarcity, which is projected to increase to 1.8 billion by 2025 Shankararaman y Mutiara, (2016) y UN, (2014).

Branchet et al. (2019) point out that urban or peri-urban centers face high population growth, the lack of sanitation infrastructure, and the need for good quality water resources, which have generated a high impact and contamination on water resources. (Chen et al., 2021), (Mora et al., 2021), and (Runkel, 2021) indicate that human activities have caused scarcity and contamination of drinking water.

Zak, 2012 ensures that some heavy metals in adequate amounts have an essential behavior in humans. However, excessive amounts have a negative impact, such as in the case of Cu, Zn, Fe, or Mn. For their part, Abdullah et al. (2019) point out that heavy metals such as As, Pb, or Hg are highly toxic, even at minimal levels.

Srinivasan et al. (1999) point out that aluminum (Al) is one of the inorganic trace metals present in drinking water. In addition to being naturally present in raw waters, Al-based coagulants, especially $Al_2(SO_4)_3$ (alum), often increase Al concentrations in treated water. It is a suspected agent causing neurological disorders such as Alzheimer's and presenile dementia. Al undergoes several transformations (also called Al "speciation") that are influenced by pH, turbidity, the temperature of the water source, and organic and inorganic ligands present in the water. Chemical precipitation, reverse osmosis, electrodialysis, and cation exchange are efficient processes for removing Al from water. On the other hand, Nieboer et al. (1995) mention that under critical evaluation of the epidemiological evidence, a natural association between aluminum in drinking water and dementia (including Alzheimer's disease, AD) cannot be ruled out.

Coplin and Galloway (1999) and Shankararaman and Mutiara (2016) point out that the deterioration of water quality in existing sources, the uncertainty in rainfall, and the unsustainable use of water require advanced treatment technologies to quench the thirst of a growing population. It is prudent to meet the ever-increasing demand using surface water, as excessive groundwater extraction causes land subsidence, damaging built infrastructure and wetlands and increasing the frequency and intensity of flooding. For his part, Edzwald (2010) indicates that surface water purification requires advanced water treatment technologies. This is because contaminants usually are loaded with high concentrations of particles, pathogenic microorganisms, and precursors of disinfection by-products, potentially carcinogenic, teratogenic, and mutagenic.

Garrido et al., 2017) and Quaghebeu et al., 2019, argue that Bolivia has shown high population growth and disorganized urban development. These problems originated from high contamination processes of natural resources, with water being the most affected.

For example, the Guadalquivir basin located in Tarija-Bolivia currently has water sources highly contaminated with heavy metals, which caused many of them to be abandoned to avoid public health problems (Villena et al., 2019).

Atab et al. (2012), Kin et al. (2018), Ray et al. (2018a), Fritzmann et al. (2007), and Chenghan and Han (2019) argue that membrane filtration water purification and treatment technologies are promising systems for the recovery of drinking water.

Chung et al. (2012), Khulbe and Matsuura (2018), Ray (2018b), and Saravanan et al. (2021) argue that Reverse Osmosis (RO) is one of the most widely used filtration technologies. January is being applied globally, becoming a tool with industrial applications. It is a process that is continuously researched to find the best options for energy efficiency and effectiveness in separating contaminants.

Various technologies exist to remove Al from drinking water. However, the overall effectiveness of water treatment processes varies considerably. On the one hand, Al is ineffective for Al is aeration/stripping, chemical oxidation/disinfection, and ion (anion) exchange. Coagulation, sedimentation, filtration (combined), and lime softening moderately effectively remove Al. At the same time, the cation exchange resin treatment, electrodialysis, and reverse osmosis are highly effective in eliminating aluminum. The RO would remove 90 to 100% of the Al present in the water Srinivasan et al. (1999).

There is little experience in removing aluminum in drinking water through reverse osmosis. However, an experimental process to remove Al by RO must consider this technology's physical/chemical aspects. RO (Feria-Díaz et al., 2021) is the most advanced technology used worldwide for water desalination. This, due to its relatively low energy consumption, high efficiency, flexibility, ease of operation, and process economy, led to advances in materials, better pump efficiency, and the creation of energy recovery devices.

However, the increase in polarisation that induces the loss of membrane permeability and fouling are some of the problems that must be analyzed and treated promptly (Sablani et al., 2001). It is stated that the main reason for the decrease in flux during the initial period of a membrane separation process is the polarization of the solute concentration at the membrane surface.

Research carried out by Alanod et al., 2020, confirms that a better recovery in an RO process occurs with increased pressure and temperature. However, it decreases when the feed flow rate increases. They also point out that lower energy consumption can be achieved with lower flow rates and pressures.

Lora et al., 2020 maintain that temperature is a parameter that must be controlled in an operation process in RO plants. This allows evaluating the effect on the operation and behavior of the RO process.

For his part, Mulder (1996) argues that transport through dense films can be seen as an activated process that can generally represent an Arrhenius-type equation. This implies that temperature can significantly affect the speed of transport. The equation expressed by Arrhenius shows the temperature dependence of the membrane permeability in the processes.

Membrane fouling is unavoidable in membrane-based water treatment processes and can significantly affect process performance, operation, sustainability, and economic viability. Due to the complex physical and chemical interaction between the different feed components and the membrane surface, an excellent way to predict membrane performance and energy requirements for pumping is through a proper understanding of the process that occurs. in the membrane (AlSawaftah et al., 2021), (Gu et al., 2021), (Salcedo et al., 2014) and (Saravanan et al., 2021).

This work shows the aluminum separation process in drinking water by reverse osmosis. Synthetic water and a pilot plant with a polyamide membrane from China, brand Keensen (2020), were used for the experimental process. The study considers the concentrations of aluminum found in the water from sources of supply in the Guadalquivir basin. During experimental tests, the process was adjusted for temperature effects to assess permeability variability and membrane fouling.

2. Goals

This paper aims to show the results of the aluminum separation process in drinking water from surface sources of the Guadalquivir basin in Tarija, Bolivia, using Reverse Osmosis and an experimental pilot plant in the laboratory.

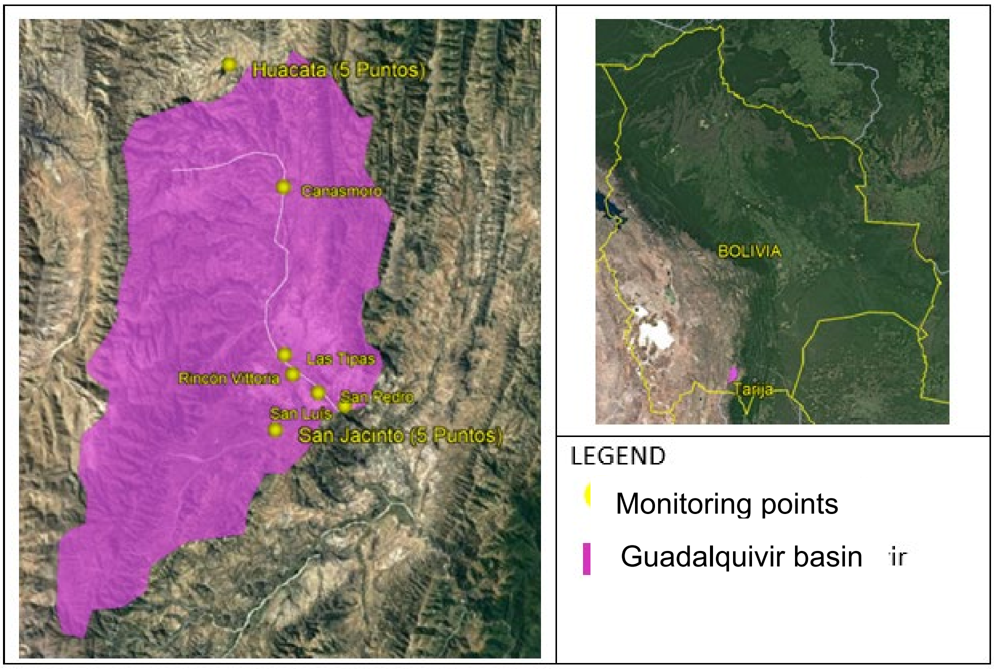
2. Materials and methods

This section describes the water problems of the study area, and the previous work carried out for the investigation, such as the monitoring of the water sources and the assembly of the RO pilot plants to the inquiry. Likewise, the membrane characteristics and the chemicals used are detailed, and the model used to analyze the system’s behavior is described.

3.1. Study area

In recent decades, Bolivia has had significant economic growth and, therefore, a population increase, which has caused a degradation of water quality in various country regions. The Guadalquivir basin is the most important area for its natural drinking water sources for the municipality of Tarija. During 2018 and 2019, within the framework of a doctoral research project with the Polytechnic University of Valencia, follow-up work was carried out to determine the quality of the water resources in the study basins. The study results exposed the presence of some heavy metals with concentrations that exceeded the permissible limits of Bolivian regulations (Villena, 2019). Denoting a significant water problem to be addressed in the study basin. Figure 1 shows the location of the study basins.

Figure 1: Location of the Guadalquivir basin, Tarija-Bolivia



The separation of metallic cations present in water for human consumption requires efficient processes and methodologies to achieve compliance with the parameters established in the Bolivian Standard and international water quality standards. In 2019 and 2020, within the framework of doctoral research with the Universitat Politècnica de València, an experimental reverse osmosis pilot plant was installed at the headquarters of the Universidad Católica Bolivia in Tarija to investigate and evaluate the process of separating heavy metals in drinking water (Villena, 2019).

The proposed experiment evaluates the RO process to separate aluminum in synthetic waters combined with sodium chloride in concentrations representative of those found during the monitoring work.

3.2. Chemicals and Membrane characteristics

The synthetic waters were made using aluminum sulfate 18-hydrate ($Al_2(SO_4)_3.18H_2O$) Panreack brand with 99.5% purity and VIOPACK brand sodium chloride. For membrane cleaning, VIOPACK brand citric acid was used.

The solutions have been prepared about the levels and concentrations of the metals found in the monitoring process. The conductivity measurement for the different samples obtained in the experimental procedure was carried out with a HACH model DREL/2088 mobile multiparameter equipment. To analyze aluminum concentrations in the practical waters, a HACH model DR 3900 spectrophotometer was used using the cromazurol S methodology with HATCH brand aluminum LCK 301 vials.

The RO pilot plant works with a Polyamide membrane-type ULP-2540, a Keensem brand of Chinese origin. The characteristics are shown in table 1.

Table 1: Technical characteristics of the membrane

Tipe	Model	Characteristics
Properties	ULP-2540	
	Composition	Polyamide
	Permeate capacity (m^3/day)	2.84
	Effective membrane area (m^2)	2.5
	Recovery rate (%)	8
	Effective membrane thickness (µm)	0.25
	Membrane pore diameter (µm)	<0.002
Terms of use	Maximum pressure (Mpa)	4.14
	Maximum temperature (°C)	45
	Maximum flow (m^3/hr)	1.4

Source: (Kennsen, 2020)

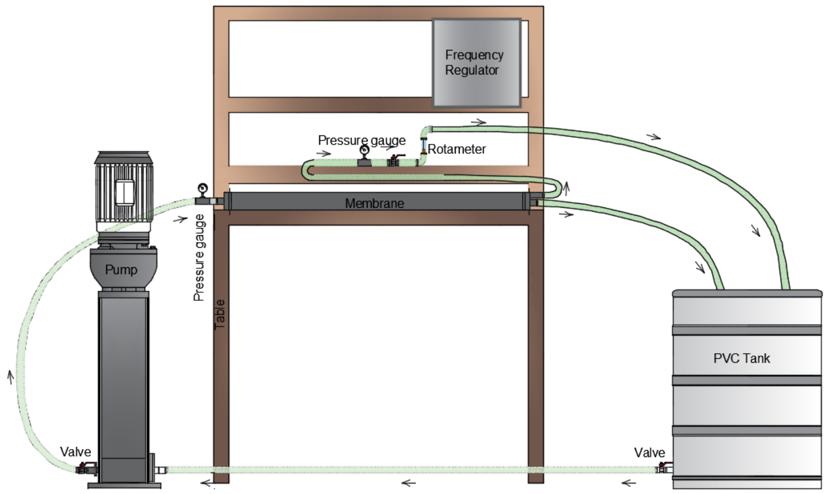
3.3. System operation

Figure 2 illustrates the schematic design of the pilot plant. It consists of a synthetic water storage tank with metal concentrations + NaCl, connected to a feed pump with a valve to regulate the entry to the system. It has a manometer at the inlet and another at the outlet of the concentrate that allows the verification of transmembrane pressures, a needle-type valve at the outlet of the concentrate to regulate the inlet flow, and two flow sensors to measure the outlet flows (permeated and concentrated). In addition, the pump has a frequency regulator to manipulate the pressures. Finally, there are the hoses for taking samples at the outlet of the permeate and concentrate.

3.4 Design of experiments and control variables

The orthogonal arrays methodology proposed by Taguchi et al., 2005 was used for the design of experiments. Three control variables were defined: pressure, inlet flow and solute concentration, and three work levels (low, medium, high) for each variable. Temperature is considered as an adjustment factor.

Figure 2: Design of the pilot experimental plant for RO



With the support of the Statgraphics Centurion software, the design of experiments was carried out, determining the performance of 9 experimental runs. For the experimentation process, 60 liters of synthetic water were prepared, using distilled water and the metal salts combined with sodium chloride; the concentrations are detailed in table 2 for the two experimental plants, respectively.

Table 2:Concentrations of solutes in synthetic water

Solutes	Experimental concentrations (mg/l)		
	C1 (low)	C2 (medium)	C3 (high)
aluminium (Al)	1.34	6.7	13.4
NaCl	23.31	23.31	23.31

Source: Own elaboration

The solutions (synthetic waters) were transferred to the membrane from the feed tank by means of a centrifugal pump with a closed circulation system. The experiments lasted approximately 30 minutes per-run. Samples of the permeate and concentrate were taken and analyzed to assess the effect of solute separation. This, through two procedures, the first, measuring conductivity with a conductivity meter and the second, through chemical analysis using a spectrophotometer.

Table 3 details the design of experiments with the different interactions between variables and levels.

Table 3: Design of experiment

ALUMINIUM

Experiment	Control variables			
	P (bar)	Income flow (l/hr)	Amount of solute	
			(g/60l) = Aluminium	(g/60l) = NaCl
9	10	440-450	19,9616	23,31
7	5	530-550	19,9616	23,31
2	7,5	530-550	1,9962	23,31
4	5	500-530	9,9808	23,31
1	5	440-450	1,9962	23,31
5	7,5	440-450	9,9808	23,31
8	7,5	500-530	9,9808	23,31
6	10	530-550	19,9616	23,31
3	10	500-530	1,9962	23,31

Source: Own elaboration

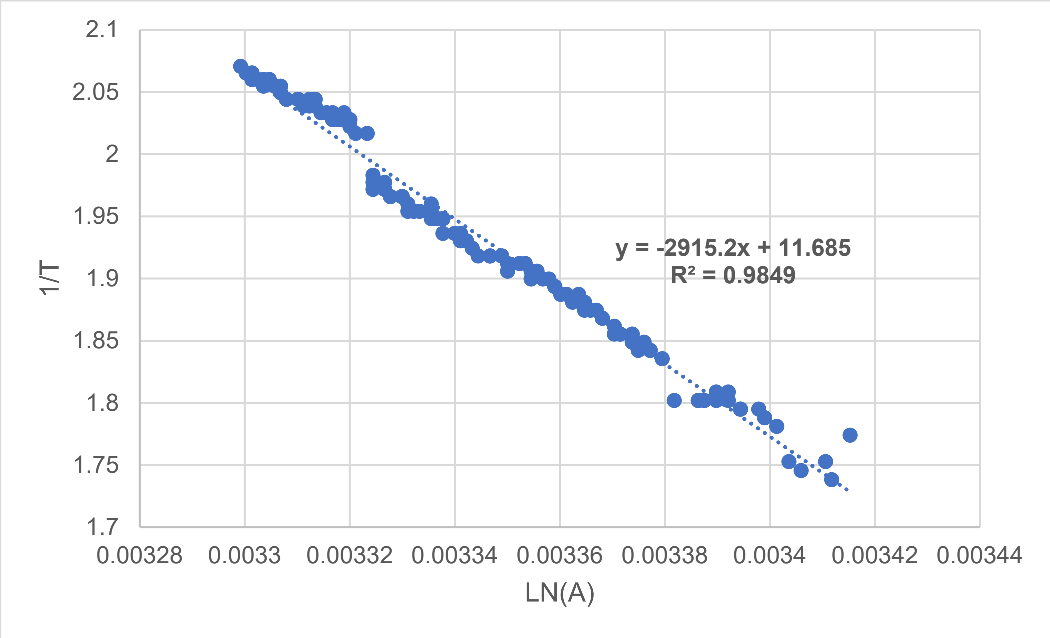
4. Results and discussion

The results obtained during the separation process of aluminum combined with sodium chloride in distilled water by means of an RO process carried out in the experimental pilot plant, set up on the campus of the Universidad Católica Boliviana San Pablo, are shown.

4.1. Determination of the adjustment factor by temperature

To adjust the results to the temperature variation, the adjustment factor was calculated using the method of Arrhenius Mulder, 1996, described in equation 1. Figure 3 shows the regression curve that determines the adjustment parameter.

Figure 3: Determination of the membrane permeability temperature adjustment factor



$$A = A_o * exp^{-\frac{\Delta H}{R}*\left(\frac{1}{T}-\frac{1}{To}\right)} \quad (1)$$

Where:

Ao = permeability over time To ($m^2 \cdot s.bar$)

ΔH/R = Activation energy obtained from the slope of the line plotted between Ln(A) y 1/T.

In this way, if we want to normalize the flux J_T, obtained at a temperature different from To, to its value Jn0, which in our adjustment is 293 K, we will have equation 2.

$$Jno \;\; = JT\, e^{-\frac{\Delta H}{R}\left(\frac{1}{T}-\frac{1}{293}\right)} \qquad (2)$$

Where:

JT = Flux ($l/m^2.hr$)

T = Temperature conversion from °K to °C = t+295

t = Temperature obtained during the experiment (°C)

Jno = Final flux after conversion ($l/m^2.hr$)

With the adjustment values shown in figure 4, the flow measured in a specific time and normalized to a temperature To = 293 K, the ΔH/R is 2915.2 K, obtaining equation 3.

$$Jno \;\; = JT\, e^{2915.2\left(\frac{1}{T}-\frac{1}{293}\right)} \qquad (3)$$

4.2. Behavior of membrane concerning pressure

According to the design of experiments, the levels established for the working pressures were 5x105, 7.5x105, and 10x105 Pa. Figure 4 shows the behavior of Jv (experimental) concerning the pressure increase ΔP. In contrast, figure 5 illustrates the behavior of Jv regarding metal concentrations.

Figure 4: Behavior of Flux Jv concerning pressure ΔP for Pb.

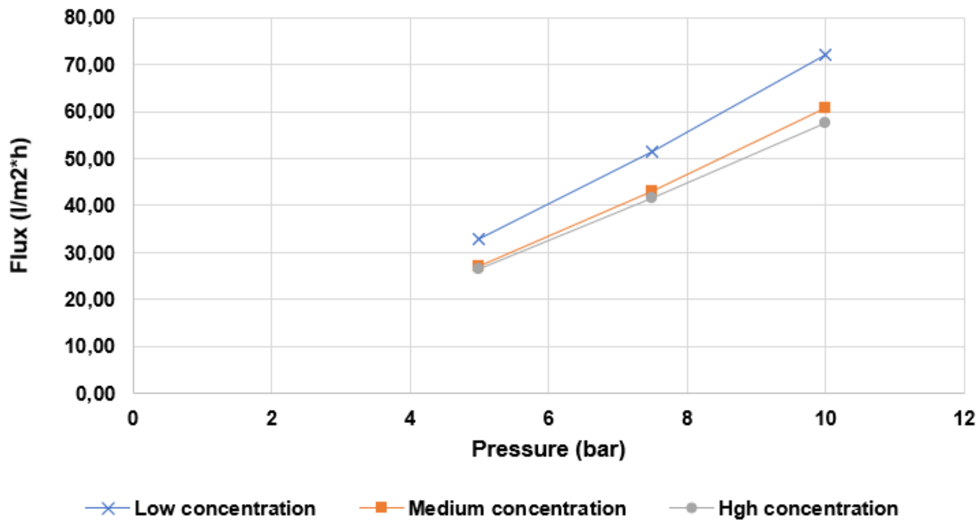
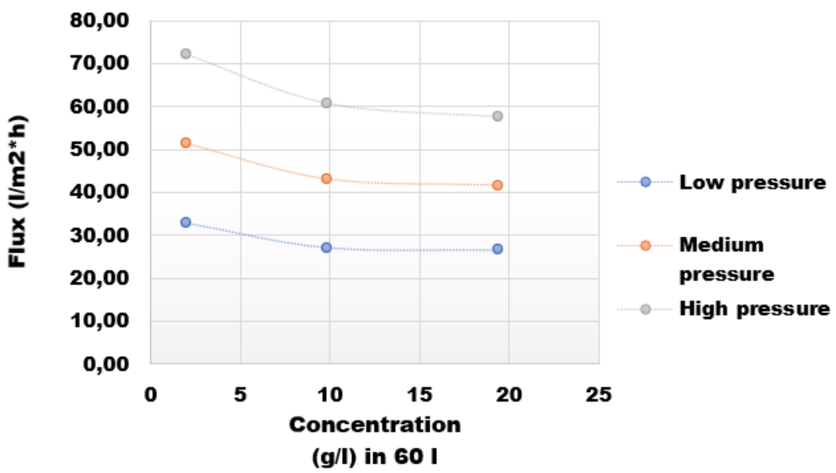


Figure 1: Behavior of Flux Jv concerning concentration to Al.



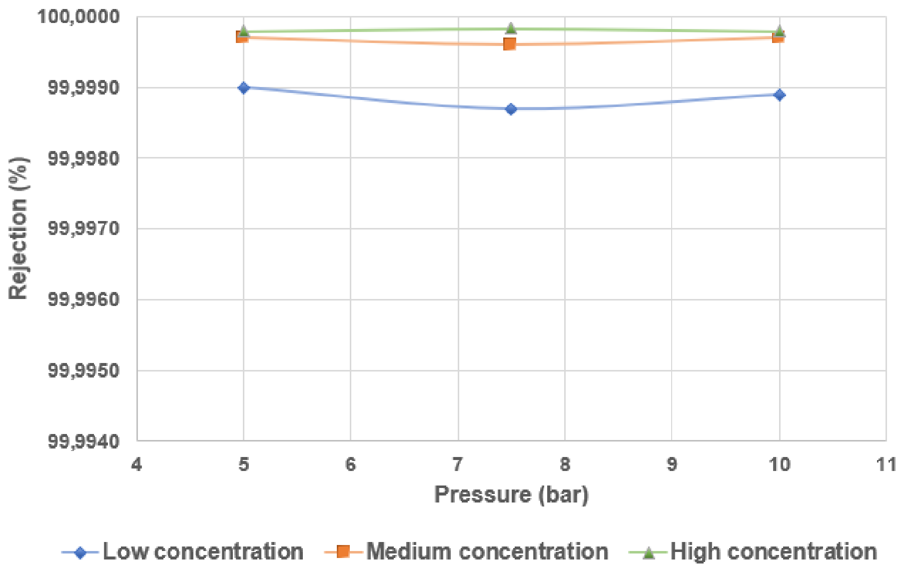
The experimental results show a good separation behavior between the components of the synthetic waters using the membrane. The behavior of Jv (experimental) was as expected for an RO process, observing that it is directly proportional to pressure and inversely proportional to concentration. This indicates that the membrane microstructure would be adequately interacting with the solution.

Under defined pressures, the flow behavior with pressure tends to be linear, so no physical compaction of the membrane is observed when solute concentrations are relatively low. However, the curve shows different behavior for higher concentrations due to the difficulty in the mass transfer process near the membrane surface.

4.3. Effect of pressure Δp on solute rejection Ro

In all cases, the rejection rates were higher than 98%, so the RO process allows safe water to be obtained, with concentrations of heavy metals below the permitted limits. Figure 6 shows the observed rejection concerning pressure.

Figure 6: Influence of the pressure Δp concerning the rejection Ro for Al

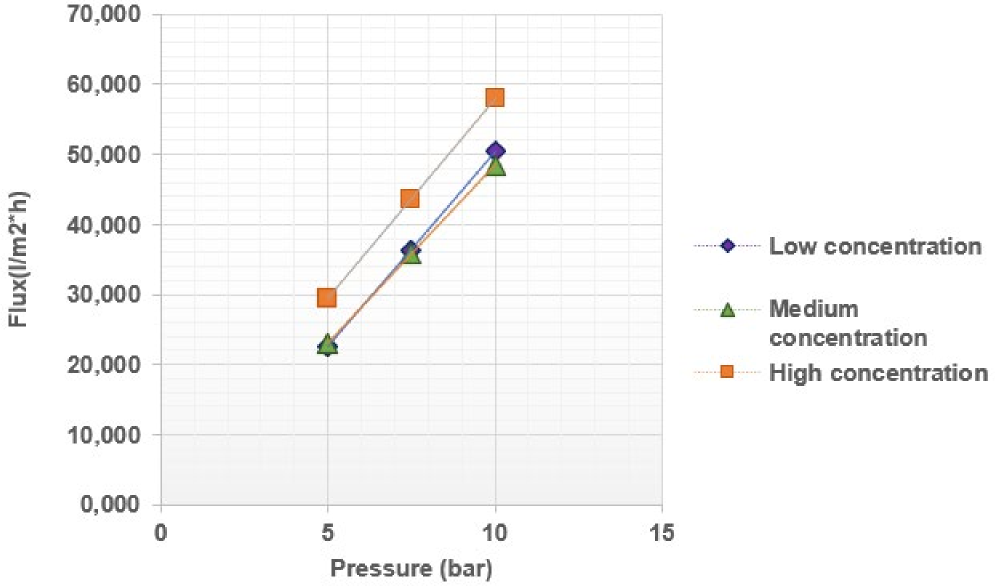


Regarding the efficiency in removing contaminants, Fig. 6 shows the Ro solute rejection index's good behavior; the rejections in most cases are greater than 99%. This allows establishing that the concentrations of metals in the permeate are below the parameters specified in national and international regulations for water for human consumption. There are no significant differences between the removal efficiencies operating at low, medium, or high pressures, which would imply that the energy cost could be reduced if one chooses to work at moderate pressures.

4.5. Effect of NaCl on Jv concerning pressure

Figure 7 shows the behavior of the Jv (experimental) concerning the increase in pressure when sodium chloride is incorporated into the synthetic water.

Figure 7: Influence of the NaCl on the behavior of Jv

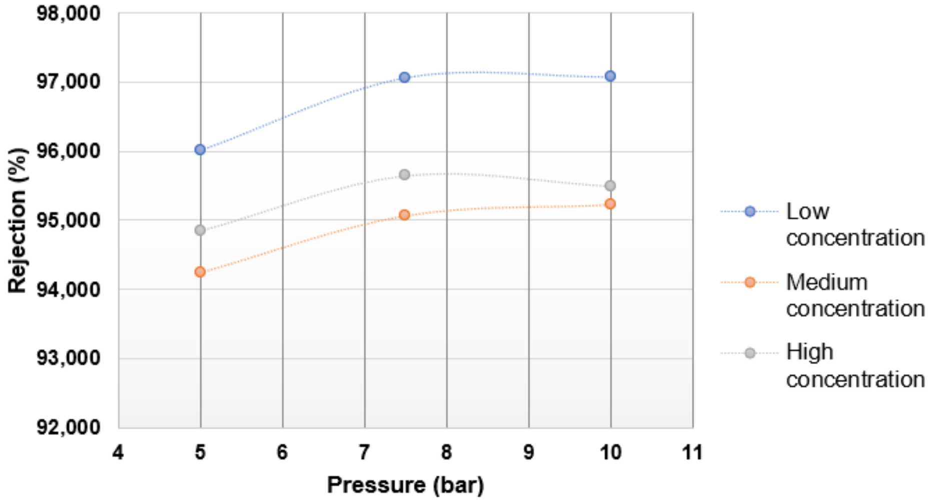


The expected behavior is observed in an RO process. However, the results show that the Jv decreases up to 20% when adding sodium chloride to the system.

4.6. Effect of NaCl on contaminant rejection

Figure 8 shows the system's behavior in rejecting contaminants when sodium chloride is incorporated.

Figure 7: Influence of the NaCl on contaminant rejection



It is observed that the presence of NaCl in the system hurts the rejection of solutes; with low and medium concentrations, the sacrifice has a linear increase; however, at high concentrations, it behaves differently; this can be explained by the process of concentration of solutes in the boundary layer of the membrane.

4.7. Membreana Cleaning

Membrane fouling in an RO process is a normal phenomenon. However, it is necessary to detect it promptly to avoid permanent damage to the membrane and reduce the efficiency of the process.

Figure 8 shows the behavior of the permeate at the beginning of the process and using distilled water.

Figure 8: Initial behavior of the permeate

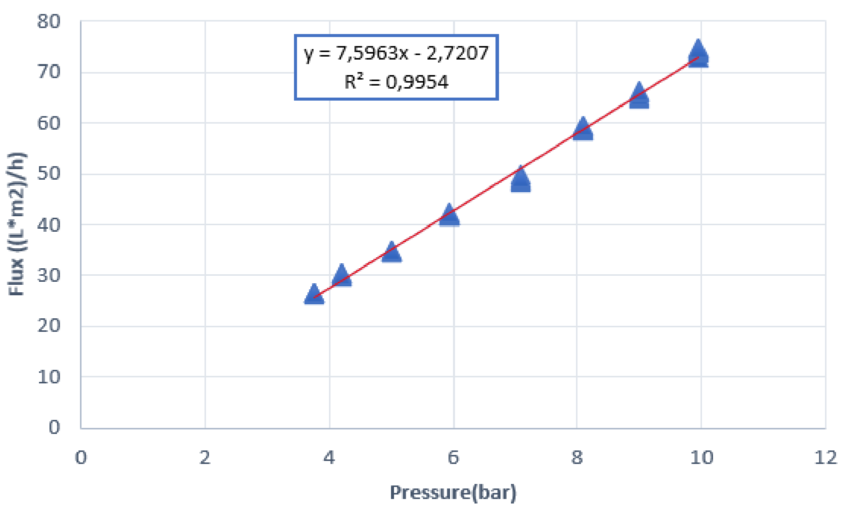


Figure 9 shows the result obtained after a cleaning process with distilled water.

Figure 8: MembraneThe membrane cleaning process

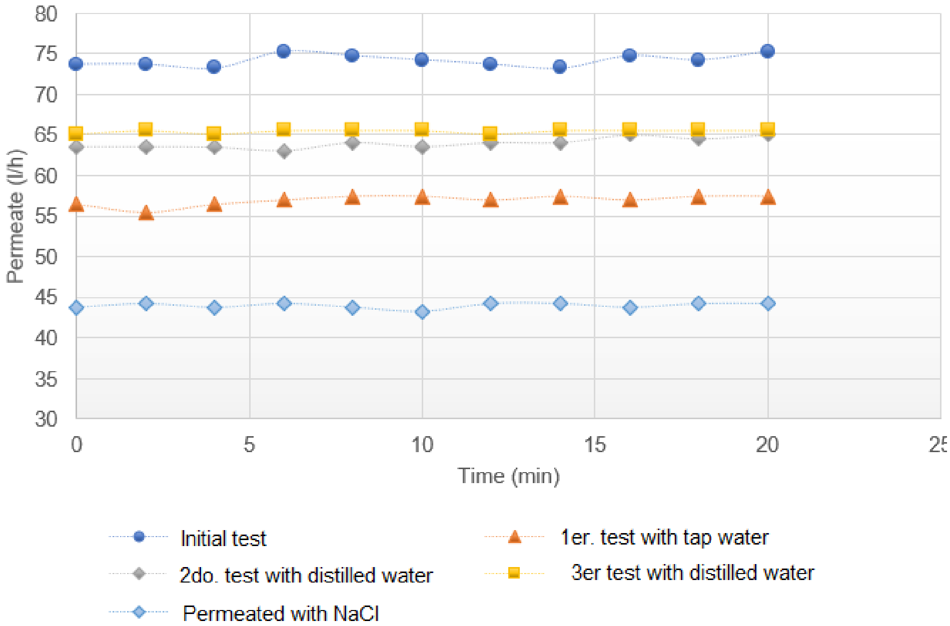


Figure 8 shows us that the permeate of the membrane had a permeate close to 70 l*m2/h. If we analyze figure 9 at the end of the experimental phase, the permeate falls to about 45 l*m2/h; it decreases by about 50%.

After three cleaning runs with only distilled water, the permeate recovers close to 100% concerning the initial behavior, which shows that the membrane maintains its efficiency for future work and research projects.

5. Conclusions

The experimental design used pressure, solute concentration, and inflow rate as control variables, which allowed the evaluation of the behavior of the feeding solutions.

The results of the experimentation show that at defined pressures and low concentrations, the behavior is linear and the membrane, therefore, does not present physical compaction. On the other hand, the flow has a different behavior at high concentrations due to the difficulty in the mass transfer process near the membrane surface.

The behavior of the membrane in the solute rejection process is as expected in an RO process; a good rejection of this is observed with "Ro" indices more significant than 99%. It is also observed that the presence of NaCl produces a decrease in the efficiency of the membrane; this is explained by the fact that the increase in solutes in the system generates more significant fouling in the membrane, also producing difficulty in mass transfer in the boundary layer.

Finally, it is shown that a timely cleaning of the RO membranes allows an optimal recovery, allowing it to have a longer useful life in rejecting contaminants and generating drinking water.

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