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REVERSE OSMOSIS PILOT PLANT TO REMOVAL HEAVY METALS Pb, Fe, As, Zn AND Mn IN CONSUMPTION WATERS IN BOLIVIA

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The presence of heavy metals in drinking water in the Guadalquivir and Milluni basins located in Tarija and La Paz, Bolivia, requires the need to investigate adequate water treatment systems and technologies. One of the most efficient systems is reverse osmosis whose filtration technology will ensure a process of elimination of these pollutants efficiently and sustainably. This work describes the design and operating procedure of the first pilot plant experimental to reverse osmosis for the cities of Tarija and La Paz-Bolivia and the characterization of a Keensem aromatic polyamide type membrane, model ULP-2540 for efficiency evaluation of this in the elimination of Pb (Lead), Fe (iron), As (Arsenic), Zinc (Zn) and Manganese (Mn) in surface water of consumption in concentrations of equivalent metal cations in synthetic waters. The experimental plant has been designed so that its operation simulates the atmospheric and climatic conditions of the study areas and whose control variables such as temperature and pressure can be manipulated with the regulation of a centrifugal pump, allowing to analyze the response variables such as the efficiency of the membrane and the flux of the system.

Keywords: pilot plant; reverse osmosis; heavy metals; drinking water

PLANTA PILOTO DE ÓSMOSIS INVERSA PARA REMOVER METALES PESADOS Pb, Fe, As, Zn, Mn EN AGUAS DE CONSUMO EN BOLIVIA

La presencia de metales pesados en agua de consumo de las cuencas del Guadalquivir y Milluni ubicadas en Tarija y La Paz, Bolivia, requiere la necesidad de investigar sistemas y tecnologías adecuadas de potabilización. Uno de los sistemas más eficientes es la ósmosis inversa cuya tecnología de filtración permitirá garantizar un proceso de eliminación de estos contaminantes de manera eficiente y sostenible. Este trabajo describe el diseño y procedimiento operativo de la primera planta experimental piloto de ósmosis inversa para las ciudades de Tarija y La Paz-Bolivia y la caracterización de una membrana del tipo poliamida aromática marca Keensem, modelo ULP-2540 para la evaluación de la eficiencia de esta en la eliminación del Pb (Plomo), Fe (hierro), As (Arsénico), Zinc (Zn) y Manganese (Mn) en aguas superficiales de consumo en concentraciones de cationes metálicos equivalentes en aguas sintéticas. La planta experimental ha sido diseñada para que su funcionamiento simule las condiciones atmosféricas y climatológicas de las zonas de estudio y cuyas variables de control como la temperatura y presión puedan ser manipuladas con la regulación de una bomba centrífuga, permitiendo analizar las variables de respuesta como la eficiencia de la membrana y el flux del sistema.

Palabras clave: planta piloto; osmosis inversa; metales pesados; aguas de consumo

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1. Introduction

The availability of water in a basin does not imply that its quality allows the use for human consumption, the constant processes of natural and anthropogenic contamination of water resources generate the need for water treatment before its use for human consumption, agricultural and even industrial consumption. The quality of the water varies according to the region and by the type of subsoil of the pollutants (Germán E. et al. 2012).

One of the biggest problems in water is the presence of heavy metals. Some are essential nutrients for plants and animals such as Mn (Manganese), Mo (Molybdenum), Cu (Copper), Co (Cobalt), Zn (Zinc), Sc (Scandium) and V (Vanadium)) while other elements like Ni (Nickel), Sn (Tin) and Cr (Chrome)) are essential only for animals. But when these elements are present in environmental systems at high concentrations due to natural imbalances or anthropogenic introduction, these metals can be toxic to living beings. (Domenech and Peral, 2008).

The toxicity of heavy metals depends on their mobility in the medium, which, in turn, depends on their chemical speciation, persistence, and tendency of accumulation or bioaccumulation. (Domenech and Peral, 2008, and Kumar, et al. 2012).

According to the "National Report on Water Management in Bolivia" (Mattos and Crespo, 2000), most of the mining activity in Bolivia is carried out in the endorheic slopes and upper areas of the Río de la Plata, since almost all the exploited mining deposits are located in this closed basin. This report indicates that tin deposits exist in the form of cassiterite or in the form of tin sulfide minerals, related to other metals (zinc, manganese, lead, and silver), contaminating rivers by discharging the waters used in the extraction and processing or by erosion and dissolution of slag from mines.

Some results have been published related to contamination by heavy metals from natural sources or from mining runoff waters, and studies were conducted on the removal of heavy metals from rainwater from mining from Cerro de Potosí using beds clays (Strosnider and Nairn, 2010).

In the basin of Lake Poopó, one of the Bolivian basins most affected by water stress, the sources of heavy metal contamination from mining were studied (Ramos Ramos et al. 2012). Previously, a different work proposed a methodology for the evaluation of mining contamination in the Pilcomayo river that was published in the Virtual Library of Public Health (Medina R, 2005).

In the upper basin of the Guadalquivir river, hardly any studies have been carried out on the presence of heavy metals on the quality of its waters. Researchers from Bolivia and the University of Seville published in the Virtual Library of Public Health an analysis of the water quality of the San Jacinto Dam in Tarija based on preliminary reports of which there is no record of its publication (Medina R, et al. 2006). Said analysis reveals the lack of truthful information about the state and quality of drinking water. The lack of appropriate facilities for the purification of water from the most important aquifers in the region has led in some cases to the shortage of populations or the use of sources of dubious quality.

There are several techniques that make it possible to convert contaminated or brackish water (seawater) into drinking water for human consumption, which is generally carried out by two types of technologies, thermal and membrane separation or filtration. In the first one, there are techniques such as multi-stage flash distillation (MSF), multiple-effect distillation (MED) and steam compression distillation, while in the second one we can name the electrodialysis technique (ED) or the osmosis inverse technique (OI) (Voutchkov, 2013).

The techniques for treating heavy metals in waters depend on different factors that allow them to be classified as conventional, referring to the techniques that are usually used for the

removal of these contaminants, such as membrane filtration such as electrodialysis, nanofiltration, ultrafiltration, and reverse osmosis, ion exchange, adsorption, chemical precipitation or coagulation-flocculation, and unconventional ones such as the metal adsorption process with the use of natural materials, phytoremediation, biopolymers, hydrogels or fly ash (Caviedes, D. et al. 2015).

Reverse osmosis is a process of membrane permeation for separation by controlled diffusion or screening. It has the ability to select elements as small as 0.0001 mm, giving it a wide range of treatment capabilities (Nemerow and Dasgupta, 1998).

The design of a reverse osmosis plant is much more complex than considering only types of membranes and high-pressure pump. It is necessary to define and optimize parameters and determine methods of pretreatment of the feed water that affect the life of the membranes and affect the energy consumption, generally, these require disinfection systems, filtration, acidification, precipitation inhibitors, microfiltration (Garcia, F. 2012).

Reverse osmosis technology is based on the osmosis process, a natural phenomenon that occurs when two solutions of different saline concentrations, brought into contact through a semipermeable membrane, tend to equalize their concentrations (Mallevialle et al. 1999 and Kucera, 2010).

Membranes are a fundamental part of the reverse osmosis process. Currently, the commercially available membranes correspond to those derived from two types of polymers: aromatic polyamides (PA) and cellulose acetate (CA). However, there are studies that indicate limitations in CA membranes such as a low operational pH range (4.5-7.5), susceptibility to biological attacks and a limited range of operating temperatures. (Li and Wang, 2010).

The reverse osmosis process uses semi-permeable membranes, allowing the purified fluid to pass while rejecting contaminants, it is one of the techniques capable of removing a wide range of dissolved species from the water. That represents more than 20% of the world's desalination capacity (Shahalam et al. 2002). It is an increasingly popular option in water treatment in chemical and environmental engineering. The use of reverse osmosis systems to remove heavy metals has been investigated but has not yet been widely applied (Fu F. and Wang Q., 2011).

Mohsen-Nia et al., (2007), they successfully removed Cu^{2+} and Ni^{2+} ions with a reverse osmosis process and the rejection efficiency of the two ions increased to 99.5%. On the other hand, Dialynas and Diamadopoulos (2009) applied a pilot-scale membrane bioreactor in combination with reverse osmosis and found that the removal of heavy metals was very high.

The use of OI in heavy metal removal has focused on the removal of cations such as Cu^{2+} and Ni^{2+} , (Mohsen-Nia, Montazeri, and Modarress, 2007), Cu^{2+} + (Zhang et al. 2009), As (Chan and Dudeney, 2008), Ni^{2+} + and Zn^{2+} + (Ipek, 2005) or Zn^{2+} + and Cu^{2+} + (Ujang and Anderson, 1996), among others.

There are few studies with OI with cations such as Fe, Hg, Sb, Pb, and Mn. OI has been used in the elimination of Hg in refinery waters (Urgun-Demirtas et al. 2012). In the case of Sb, the effect of pH on elimination in drinking water using OI has been studied with good results (Kang et al. 2000).

The Guadalquivir basin represents the most important hydrological and water supply zone in the central valley of Tarija, Bolivia. This for having the largest population in the department and for the quality and quantity of water in the area (Villena et al, 2019 and Villena et al, 2019).

The present work shows the physical, operational and experimental design of a reverse osmosis pilot plant for the removal of heavy metals such as Iron (Fe), Arsenic (As), Lead (Pb), Zinc (Zn) and Manganese (Mn) present in water for human consumption from different surface sources in the Guadalquivir, Tarija and Milluni basins, La Paz. The work evaluates the behavior

and efficiency of a semipermeable aromatic polyamide membrane under typical climatic conditions of the study areas.

2. Goals

The objective of the project is the physical and experimental design for a reverse osmosis pilot plant whose operation works under climatic and atmospheric conditions like those of the cities of Tarja and La Paz, Bolivia. The pilot plant will allow the evaluation and analysis of the behavior of a semipermeable membrane for the elimination of Iron (Fe), Arsenic (As), Lead (Pb), Zinc (Zn) and Manganese (Mn) present in drinking water of the main sources of supply for study areas.

The study includes the elaboration of the design of experiments and the protocol for the assembly and operation of the pilot plant.

3. Design

The following section describes the materials used for the physical design of the plant that includes the assembly, operation and commissioning of the pilot plant.

3.1. Materials

The materials used for the assembly and operation of the plant are described in this section.

3.1.1. Membrane

The system uses a commercial Keesem model ULP-2540 membrane and Wave Cyber (Shanghai) model 2540.300.1 housing. The membrane is of the Polyamide type, wound in a spiral with an active area of 25 m², allows a permeate of 2.84 m³ / d with a rejection of concentrate of up to 99.3%. The maximum working pressure (applied) is 600 psi (40 bar) and a minimum proportion of concentrate of 8%, according to manufacturer data. Figure 1 shows the detail and configuration of the membrane.

Figure 1: Membrane configuration



3.1.2. Control pump and accessories

The plant works with a 1.5 HP power multistage centrifugal/electric pump, 1.8 m³ / h and a maximum operating pressure of up to 35 bar.

The system is mounted on an aluminum test frame and a stainless-steel countertop. At the entrance of the pump, a water filter was installed capable of retaining possible materials in suspension, as well as having a safety valve against possible pressure changes that may damage the membrane.

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3.1.3. Systematization and flow measurement

For permanent and continuous control of the flows, flow measurement sensors were installed at the outlet of the permeate and concentrate, in addition to an analog rotameter at the outlet of the concentrate to have a visual control system.

The sensors are from the Gems Sensors brand, with scales of 0.5 to 5 l/min placed at the outlet of the permeate and from 1 to 25 l/min at the outlet of the concentrate. The sensors have been systematized and programmed to an Arduino board connected to a computer that allows the measurement and recording of flow data in real-time.

3.1.4. Test frame

The test frame has been made of aluminum with a stainless-steel operating table to prevent corrosion and contamination from the experiment.

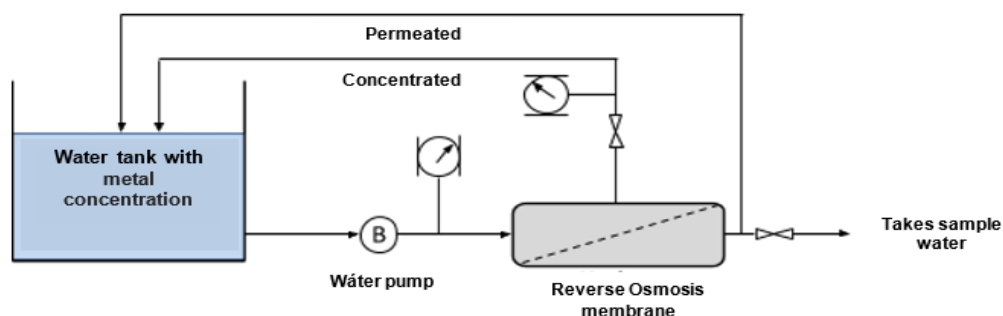
3.2. Plant Operation

To guarantee proper operation and obtain optimal results, the following plant operational process is proposed.

3.2.1. Conceptual operation of the plant

Figure 2 shows the conceptual approach for the general operation of the plant.

Figure 2: General scheme of operation of the pilot plant



The system has been designed to work in a closed-loop. The sample with the metal concentration is introduced into the tank and pumped to the RO membrane where the concentrate and permeate flow are recirculated to the tank. The process is kept running by recording the process data until the Jws (flux) remains constant. At that time, samples of the permeate are taken and the concentration of the metal cation is analyzed.

3.2.2. Operation and performance protocol

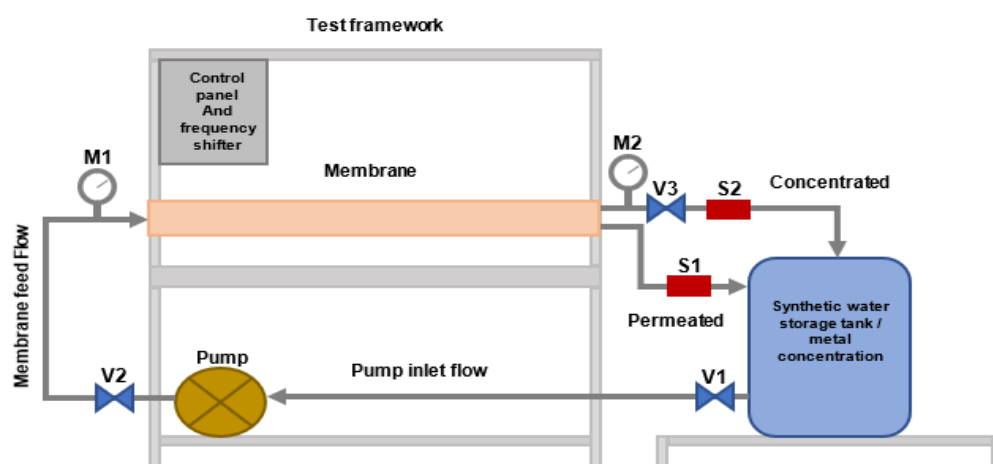
The study water is fed to the membrane by means of the pumping system at pressures and flows regulated with the frequency shifter and the needle valve installed at the outlet of the concentrate, figure 3 shows the general operating diagram of the plant:

The following operating protocol has been designed to guarantee optimal results:

- The synthetic water tank with the concentration of the metal cation in an amount of 60 liters is connected to the pumping system.
- To start the experimentation, the valves V1 for water entering the pump and V2 for feeding the membrane must be open.

- The frequency regulator located on the control panel must be in the initial position (zero) and the needle valve V3 that regulates the pressure located at the outlet of the concentrate at the lowest level.
- When starting the system start-up, it must be verified that the pressures indicated by the pressure gauges M1 and M2 located at the inlet of the membrane and at the outlet of the concentrate respectively are equal to or with the minimum difference in pressure drop in the membrane, indicated by the manufacturer less than 1 bar.
- The pressure variations can be manipulated or controlled with the opening or closing of the V3 valve, while the supply, concentrate and permeate flow rates are regulated with the frequency shifter. It is important to safeguard the supplier's conditions regarding the maximum working pressures and temperatures that the membrane and the protective casing withstand.
- Flow rates at the output of the system are visualized and systematized by flow sensors S1 and S2 located at the output of the permeate and concentrate respectively and connected to a computer and programmed to an Arduino board.
- Each experiment of each metal studied will generate information that will allow the evaluation of the efficiency and behavior of the membrane under site-specific operating conditions, such as the pressure, temperature, concentration of the metal in the water, and operating flow rates.
- When observing the stabilization of the flows that can last between 45 to 60 minutes from the beginning of the experiment, 3 water samples are taken in minimum quantities of 100 ml from the following sectors:
 - Income flow with the initial concentrate sample
 - The flow of permeate with the sample at the exit of the permeate
 - Concentrate flow with the sample at the concentrate outlet
- At the end of the tests, the shutdown must be carried out first by lowering the frequency regulator to zero and valve V3 at the minimum pressure, finally valves V1 and V2 are closed.

Figure 3: General operation design of the pilot plant



4. Experimental design and preliminary operational tests

The experimental design has been proposed following the response surface model proposed by Box and Hunter (1957), Box and Draper (1959) and Box and Wilson (1992) who developed theoretical-methodological criteria to provide greater precision in the response of different processes, which implies a search for the Model-Design combination of treatments, which is essentially what the Response Surface Methodology - MSR contains.

The response surface model is very useful for modeling and analyzing the results obtained in applications where the response of interest is influenced by different variables and the objective is to optimize this response. Its main advantage, compared to other models that relate a variable to three levels, is that it provides the minimum number of experimental runs, which makes it an economical design from the point of view of reagents, for quantification of samples, payment of external checks, energy, among others (Gil E., 2002).

The trend in the development of the methodology for response surfaces has been the construction of compact experimental designs, with a minimum of experimental units; the researcher concentrates on the properties of the estimators of all the parameters of the response function, properties that depend on the design used (Briones-Encina, F. and Martínez-Garza, A. 2002).

Kuehl (2001) points out that the MSR is useful for solving certain types of scientific problems, industrial and engineering processes, showing itself with greater application in the industrial, chemical and agricultural areas. The experimental design with the determination of control and response variables and the determination of the number of experiments are detailed below.

4.1. Determination of the variables

In this section, the variables needed for the design of the experiments are defined.

a) Control variables: The control variables are:

- **Pressure (Pr)**
- **Conversion (Y):** The conversion reflects the relationship between the permeated flow and the inflow flow, shown in equation 1:

$$Y = P / F \quad (1)$$

Where:

P = Permeated

F = income flow

- **Metal Concentration (C):** Concentrations vary according to the type of metal obtained from the previous monitoring process.

b) Experimentation levels by variables: Each control variable has 3 levels of experimentation according to the detail in table 1:

c) Response variables

The response variables area:

- **Flux (Jws) I:** Flux calculated based on the equation (2):

$$\text{Flux (l*m}^2\text{/hr)} = \text{Permeated} / \text{Membrane area} \quad (2)$$

- **Rejection (R) =** The rejection of the concentrations of the metal cations are calculated with the equation (3):

$$R (\%) = C_f/C_p \quad (3)$$

Where:

C_p = Concentration of the metal cation in the permeate

C_f = Concentration of the metal cation in the incoming water (synthetic)

Table1: Metal experimentation levels

Metal	Pressure Pr (bares)	Conversion Y (P/Flux)	Concentration C (mg/l)
M1	[5-10-15]	[5-7.5-10]	[C1-C2-C3]
M2	[5-10-15]	[10-12.5-15]	[C1-C2-C3]
M3	[5-10-15]	[15-17.5-20]	[C1-C2-C3]

4.2. The number of experiments

The data obtained and the determination of the number of experiments and their interactions were analyzed using Statgraphics Centurion commercial statistical software using the predictive response surface method. The analysis of variance, the graphs of effects and interactions, the coefficients of determination (R^2) and the graph of the response surface were generated with the same software. For this effect, the factorial fraction method with 3 levels was used, determining 9 tests for each metal. Table 2 shows the interactions between variables defined by the software.

In the case of concentrations, 3 levels are defined per metal analyzed, with C1 being the lowest, C2 the mean and C3 the highest:

- C1Pb, C2Pb y C3Pb corresponds to the 3 levels of lead
- C1Fe, C2Fe y C3Fe corresponds to the 3 levels of iron
- C1As, C2As y C3As corresponds to the 3 levels of arsenic
- C1Zn, C2Zn y C3Zn corresponds to the 3 levels of zinc
- C1Mn, C2Mn y C3Mn corresponds to the 3 levels of manganese

The 9 tests determined by the software for each analyzed metal, allow us to point out that the investigation will have 45 runs in general.

4.3. Initial tests to verify membrane specifications and temperature adjustment factors

The tests prior to the experimental phase have allowed verifying the supplier's (manufacturer's) specifications regarding the efficiency in salt removal and the determination of a temperature adjustment factor, which allows correcting the results at any experimental temperature.

4.3.1. Verification of the efficiency in salt removal

The commercial membrane control test indicates an efficiency greater than 99% in salt removal under initial conditions that the supplier performs with Sodium Chloride (NaCl) at a concentration of 1500 PPM and operating conditions of 10 bar, 25 ° C and at least 30 min of operation, these conditions have been verified obtaining the following results shown in table 3:

Table 2: Interactions between variables of the experiment design

Test	Variables		
	P (Bar)	Y % (P/F)	Cf (mg/l) - metal concentration
1	15	1-5	C3Pb, C3Fe, C3Zn, C3As y C3Mn
2	5	10-15	C3Pb, C3Fe, C3Zn, C3As y C3Mn
3	10	10-15	C1Pb, C1Fe, C1Zn, C1As y C1Mn
4	5	5-10	C2Pb, C2Fe, C2Zn, C2As y C2Mn
5	5	1-5	C1Pb, C1Fe, C1Zn, C1As y C1Mn
6	10	1-5	C2Pb, C2Fe, C2Zn, C2As y C2Mn
7	10	5-10	C3Pb, C3Fe, C3Zn, C3As y C3Mn
8	15	10-15	C2Pb, C2Fe, C2Zn, C2As y C2Mn
9	15	5-10	C1Pb, C1Fe, C1Zn, C1As y C1Mn

Table 3: Verification of NaCl removal membrane specifications

Test	Conductivity in the permeate (us/cm)	Ph	Temperature °C	Conductivity in the dissolution (NaCl) - 1500 PPM in 60 liters - us/cm	Removal of NaCl (us/cm)	% removal
1	12.39	7.13	25	2760	2747.61	99.55%
2	12.58	7.17	25	2760	2747.42	99.54%
3	13.3	7.76	26	2760	2746.7	99.52%
4	13.3	7.91	26	2760	2746.7	99.52%

For the verification test, 4 process samples were taken, measuring the conductivity of each one and comparing it with the concentration at 1500 PPM of NaCl, a removal greater than 99% is obtained, verifying the initial specifications of the membrane.

4.3.2 Determination of the temperature factor

To obtain a factor that allows an adjustment of the data obtained during the experimentation and avoid manipulation of the temperature, the temperature factor of the system must be calculated based on the temperature variation experienced by the water in the experimentation process in the plant, this factor is calculated by graphing the temperature variation with respect to the flux.

The temperature factor is carried out by feeding the system with distilled water with the minimum temperature registered in the monitoring areas and the minimum operating pressure. In the case of this project, it started with a temperature of 17 ° C and the constant pressure of 5 bars, during the operation of the closed-circuit system, the water experiences temperature

increases, causing variations in the permeate and concentrate flow rates. Permeate allows calculating the detailed flux in equation (2).

To obtain the temperature adjustment factor, the Arrhenius model (Huang y Lin, 1968) was used to change the permeability of the membrane by temperature, detailed in equation 4:

$$A = A_0 e^{-\frac{\Delta H}{R}(\frac{1}{T} - \frac{1}{T_0})} \quad (4)$$

Where:

A_0 = Permeability to time T_0

If the flux by reverse osmosis is defined according to the equation (5):

$$J = A(\Delta P - \Delta \Pi) = A_0 e^{-\frac{\Delta H}{R}(\frac{1}{T} - \frac{1}{T_0})} (\Delta P - \Delta \Pi) \quad (5)$$

Where:

ΔP = is the pressure difference between the faces of the membrane, that is, the average pressure between the inlet and outlet of the module if the permeate is at atmospheric pressure.

$\Delta \Pi$ = it is the difference in osmotic pressure of the food solution and that of the permeate. In this way, to normalize the JT flux, obtained at a temperature other than T_0 , at its J_{n0} value, which in our adjustment is 293 K, we will have the following expression (6):

$$\frac{J_T}{J_{n0}} = \frac{A(\Delta P - \Delta \Pi)}{A_0(\Delta P - \Delta \Pi)} = \frac{A_0 e^{-\frac{\Delta H}{R}(\frac{1}{T} - \frac{1}{T_0})} (\Delta P - \Delta \Pi)}{A_0 e^{-\frac{\Delta H}{R}(\frac{1}{T_0} - \frac{1}{T_0})} (\Delta P - \Delta \Pi)} = e^{-\frac{\Delta H}{R}(\frac{1}{T} - \frac{1}{T_0})} \quad (6)$$

Figure 3 shows the graph of the behavior of permeability with temperature change:

With the results obtained and from the graph, the adjustment values would be that, from the flux measured at a given temperature, the flux normalized to is 2459.5, obtaining the following model (7) for adjusting the permeability to any temperature:

$$J_{n0} = J_T e^{\frac{\Delta H}{R}(\frac{1}{T} - \frac{1}{T_0})} = J_T e^{2459.5(\frac{1}{T} - \frac{1}{293})} \quad (7)$$

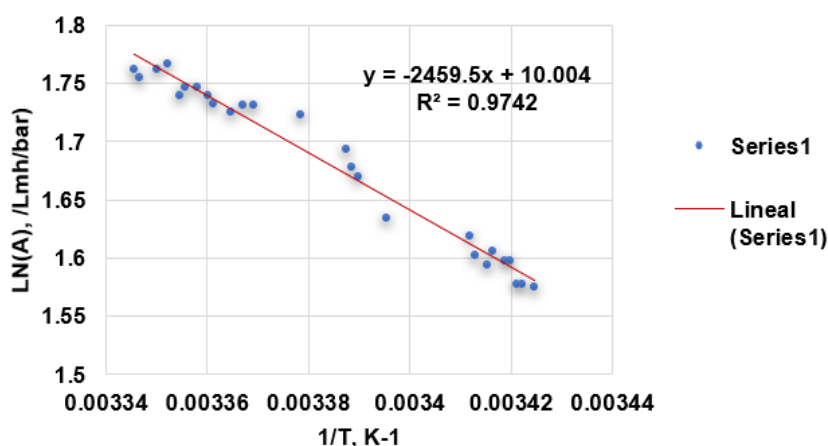
4.4. Experimental phase and operation

The experiments allow evaluating the efficiency of the membrane in the removal of metals, for this purpose 60 liters of synthetic water are prepared in concentrations defined in the design of the experiment for each of the study metals.

The systematization of the experiments generates a continuous and permanent record of the results that are stored in a computer for further processing. These results will allow evaluating the following response variables:

- The efficiency of the membrane in removing metal from the water.
- The flux.
- The simulation of the behavior of the membrane before an industrial scale operation process, which includes the analysis of membrane fouling.

Figure 3: Permeability vs. temperature



5. Conclusions

The design and assembly of the reverse osmosis pilot plant allow the operation and operation to simulate climatic and atmospheric conditions of the study areas, modeling the behavior of the system with pressures between 5 to 15 bar and temperatures that vary between minimums of 10 ° C up to maximums of 30 ° C.

The system allows the treatment of lead (Pb), iron (Fe), manganese (Mn), zinc (Zn) and arsenic (As) in waters for human consumption for research areas whose pilot experimental plant uses materials, equipment and supplies commercial centers, which guarantees an adequate operating process and sustainable maintenance.

Compliance with the initial conditions indicated by the membrane supplier was verified, guaranteeing optimal operation in the process. The Statgraphics has allowed us to successfully design the experimental protocol and the analysis of the results of the experimental phase for the analysis of the behavior of the membrane in the removal of heavy metals.

Under the Arrhenius model, a specific model was experimentally determined to adjust the permeability of the membrane to any operating temperature of the study areas, this allows eliminating the temperature variable in the factorial design of the experiments.

The pilot plant, the experimental design and the results of the process will provide important and necessary information for the future approach of an industrial phase that guarantees the production of drinking water in the study areas under criteria of energy efficiency and efficient operation and maintenance.

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