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Purification and characterization of reagent grade NaCl obtained from Crucita seawater

Purificación y caracterización de NaCl grado reactivo obtenido del agua de mar de Crucita

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Abstract
The main objective of this work is to analyze and apply the processes to obtain sodium chloride (NaCl) in a seawater sample form Crucita-Manabí and bring it to a higher purity in accordance with the Pharmacopoeia standards of the United States (USP). USP establishes quality standards and specifications for a wide range of pharmaceutical, chemical and health products. Therefore, the research was developed in three stages: the first corresponded to the purification of NaCl through the compilation of information from bibliographic sources that allowed investigating the different industrial and artisanal processes for the purification of NaCl, through this research it was possible to determine that to carry out this process separation, crystallization, distillation and filtration techniques were applied that allow the elimination of impurities present in the sample. The second stage corresponded to the analysis established based on the results obtained in the purification process; Therefore, it was essential to evaluate the conditions that allowed the impurities present to be eliminated, making it necessary to perform physical and chemical tests to determine the percentage of purity of the final product. Finally, the third stage consisted of analyzing the final product to establish whether it met the proposed objective based on the USP regulations on NaCl.

Keywords
Sodium Chloride, Purification, Crystallization, Distillation, Filtration.

Resumen
El presente trabajo, tiene como objetivo principal analizar y aplicar los procesos para la obtención del cloruro de sodio (NaCl) grado reactivo en muestra de agua de mar de Crucita-Manabí y llevarlo a una pureza más elevada acorde a los estándares de Farmacopea de los Estados Unidos (USP, por sus siglas en inglés). La USP establece estándares y especificaciones de calidad para una amplia gama de productos farmacéuticos, químicos y de salud. Por tanto, la investigación se desarrolló en tres etapas: la primera correspondió a la purificación del NaCl a través de la recopilación de información de fuentes bibliográficas que permitieron poder analizar los diferentes procesos industriales y artesanales para la purificación del NaCl, mediante esta investigación se pudo determinar que para llevar a cabo este proceso se aplicaron técnicas de separación, cristalización, destilación y filtración que permiten la eliminación de impurezas presentes en la muestra. La segunda etapa, correspondió al análisis establecido en base a los resultados obtenidos en el proceso de purificación; por tanto, fue fundamental evaluar las condiciones que permitieron eliminar las impurezas presentes, siendo necesario realizar pruebas físicas y químicas para determinar el porcentaje de pureza del producto final. Finalmente, la tercera etapa consistió en analizar el producto final para establecer si este cumplía con el objetivo propuesto basado en las normativas USP sobre el NaCl.

Palabras clave
Cloruro de Sodio, Purificación, Cristalización, Destilación, Filtración.

1. Introduction

NaCl is a simple chemical compound, composed of a sodium ion (Na+) and a chlorine ion (Cl-), known as common or table salt; it is a crystalline solid and soluble in water [1].

Reactive sodium chloride is a purified and superior variant of the conventionally used NaCl. This NaCl is used in laboratories and industries that demand a higher level of purity than that offered by common salt. For its use, reactive NaCl must meet specific purity requirements (99% - 100.5%) according to USP standards and be free of impurities that could affect the chemical or biological processes in which it is applied.

This compound has a wide application in sectors such as the chemical and pharmaceutical industry, where it is used as a reagent, buffering agent and in the production of various chemical products. It is also used in scientific research and in the production of food, medicines and cosmetics.

The present research focused on obtaining reagent grade NaCl in Ecuador, the purpose was to obtain this product and bring it to the highest possible purity since the process and purification of this product has not been done before in the country despite having enough raw material available, and to study and analyze the theoretical principles behind the NaCl purification process, including physical analysis and the chemical reactions involved.

Manabí is a province with saline resources that are used for salt production; however, at present the national industry does not produce NaCl for reactive use and proof of this is

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that the country's universities are forced to acquire NaCl in its reactive grade from chemical companies that import it for use in various analyses or studies carried out in laboratories at the university level. In this sense, it is necessary to carry out an investigation on the NaCl purification process and analyze it in seawater samples from Crucita-Manabí to determine its purity level and bring it to a higher purity according to the mentioned standards.

For this, a punctual sampling of the water was carried out in which representative samples were obtained and physical-chemical analyses were carried out to determine the concentration of NaCl before and after the purification process and then analyze the purity of the NaCl obtained by the simple evaporation method, evaluating the effectiveness of the process and comparing the results obtained with the USP standards. The conditions to eliminate the impurities present in the NaCl obtained were evaluated [1].

2. Materials and methods.

Before the composition of matter was discovered, the word salt referred to any soluble, non-flammable solid, especially when referring to that which was formed as a result of the evaporation of seawater. Despite its ancient etymology, the word salt is still used today with two distinct meanings. One is the specific name of the chemical compound sodium chloride, while the other is the generic name of the group of chemical substances formed from acids in which metals have partially or completely replaced hydrogen atoms [2]. The salt purification process begins with brine deionization or evaporation, where the ions are treated to remove impurities and sometimes purified before crystallization. It occurs in open-air salt pans that favor sodium chloride crystallization when evaporation begins [1].

A new technique to purify sodium chloride is by two-dimensional gel electrophoresis. The technique allows obtaining NaCl nanoparticles uniform in size and of high purity, which could be used in a variety of areas such as medicine and catalysis [3]. In the present work, a study was conducted that analyzed how operational parameters affect the purification of NaCl by ultrasound-assisted crystallization. The results showed that this technique can produce high purity NaCl crystals with lower energy consumption than conventional techniques [4]. A very novel technique was found to account for the use of modified zeolites as adsorbents to purify NaCl from wastewater. The technique allowed the recovery of high purity NaCl while reducing the impact of wastewater effect on the environment [5]. Similarly, purification of reactive NaCl by evaporation, simple distillation and ion exchange was performed.

The detailed process for obtaining reagent grade NaCl from seawater is described below. The method employed involves a series of successive stages, including initial physical-chemical analysis, evaporation, recrystallization, dissolution, filtration, distillation, ion exchange, washing of crystals and finally drying. The NaCl obtained was characterized by physicochemical analysis to determine purity, reaching a classification that is considered reactive.

The initial sampling and analysis consisted of collecting 20 liters of seawater and performing initial physical and chemical analyses to determine the characteristics of the seawater. Then, precipitation and primary crystallization were performed, which consisted of evaporating 12 liters of seawater at 100ºC for 8 hours. Evaporation caused the precipitation of dissolved chemical compounds, resulting in primary crystallization; 500 ml of distilled water were added to dilute the sample, and then vacuum filtration was performed to eliminate the non-soluble solids that precipitated as the temperature increased. Next, distillation and softening were carried out in which a simple distillation was implemented to obtain a concentrated brine which was analyzed detecting a high hardness in which an ion exchange was applied using a cationic resin to eliminate calcium and magnesium ions.

After this, fractional recrystallization and washing was applied, where the brine was diluted again with 500 ml of distilled water, evaporated at 100ºC for 10 minutes with constant agitation, the solution was passed through columns of regenerated cationic resin to eliminate the calcium and magnesium ions (repeated 3 times), Evaporation was carried out to obtain a fractional recrystallization, the crystals were washed with 500 ml of additional distilled water applying once again a new vacuum distillation and the evaporation and recrystallization stage was repeated. Finally, the drying and final analysis was carried out, which consisted of taking the sample obtained to an oven at 105ºC for 5 hours. Once the sample was dry, it was weighed and 562.45 g of NaCl was obtained, and finally, to determine its percentage of purity, physical-chemical analysis was carried out, where 97.23% of NaCl GR was obtained.

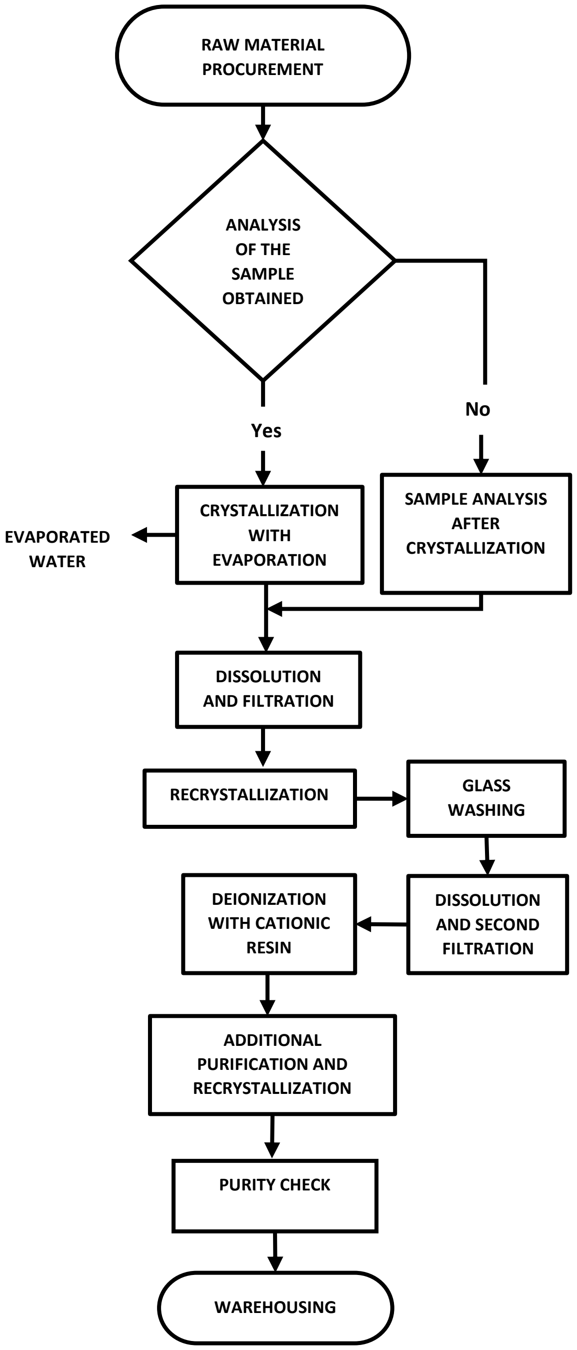

Fig. 1. Reagent-grade NaCl purification process flow diagram

2.1. Composition of seawater.

Seawater is a solution in water (H_2 O) of many different substances. Up to 2/3 of the natural chemical elements are present in seawater, although most of them only as traces. Six components, all of them ions, account for more than 99% of the solute composition [6].

Table 1. Percentage composition of solid solutes in seawater.

Anions	%	Cations	%
Chlorides (Cl^{-})	55,07	Sodium (Na^{+})	30,62
Sulfates (SO_4^{2-})	7,72	Magnesium (Mg^{++})	3,68
Bicarbonates (HCO_3^{-})	0,41	Calcium (Ca^{++})	1,18
Bromide (Br^{-})	0,19	Potassium (K^{+})	1,14
Fluorine (F^{-})	0,01	Strontium (Sr^{++})	0,02

Source: Osorio Arias & Álvarez Silva, 2006.

Information was gathered that allowed us to broaden our knowledge about the different methods and techniques that can be used in the process of obtaining NaCl Reactive Grade.

2.2. Selection of raw material

The seawater selected for the extraction of NaCl G.R. was collected in the sea of Crucita in the canton of Portoviejo, province of Manabí, Ecuador (Fig. 1). These samples were transferred to the chemistry laboratory of the Faculty of Engineering and Applied Sciences.

Fig. 2. Panoramic view of the sea locality of Crucita.

2.3. Experimentation

The experimental work began by taking seawater samples from the parish of Crucita for their respective physical-chemical characterization in the aforementioned laboratory.

Physical-chemical analysis: alkalinity, chlorides, conductivity, hardness, salinity, total solids, pH and temperature.

Table 2. Physical and chemical analysis of seawater samples.

Components	Results	Units
Alkalinity	96	mg/L
Chlorides	20020	mg/L
Conductivity	64000	us/cm
Hardness	4764	mg/L
Salinity	0,14	%
Total Solids	1380	mg/L
pH	6,27	
Temperature	27.5	ºC

The main methods applied during the experimentation were gravimetric and volumetric: in the volumetric method the ion contained in a given product was determined quantitatively; by measuring the volume of a solution of known concentration or standard solution that reacts with a known amount of solution containing the element under study and gravimetric method which is based on the precise and accurate measurement of the mass to be determined, which was separated from the rest of the components of the sample being NaCl. Although most of the substances were found in very low concentration, there were two important substances that are commercially extracted from seawater: sodium chloride (table salt) and magnesium [7].
The different physical analyses of the water sampled were obtained using the multi-parameter equipment (BLE-9909).

Alkalinity: The alkalinity of seawater plays a crucial role in buffering the pH of the ocean against acidification caused by CO2. The alkalinity of seawater is mainly determined by two components: carbonate ions and borate ions. These ions neutralize the hydrogen ions produced by CO2 dissolution, which limits the decrease in pH [8].

Alkalinity consists of the ability to neutralize acids and is the sum of all titratable bases, it prevents water pH levels from becoming too acidic or basic, giving rise to carbonates and bicarbonates in seawater [9].

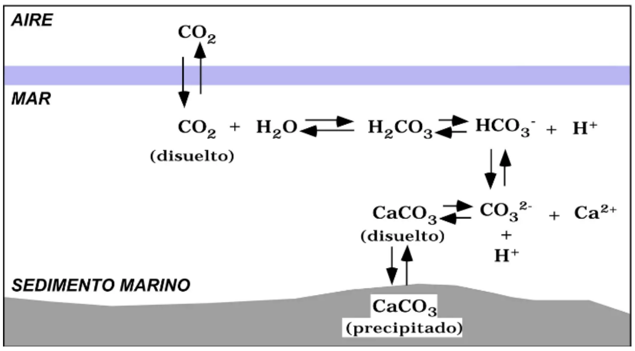

Fig. 3. Origen de bicarbonato y carbonato en el ambiente marino.

Hardness: Although seawater is an essential resource for life on earth, its high salinity and hardness prevent it from being used for human consumption or agricultural irrigation. The removal of salt from seawater, or desalination process, has become an increasingly important technology. The ability of seawater to dissolve a soap is measured in terms of because of its hardness. The presence of magnesium and calcium ions is the main cause. The hardness of seawater in the Pacific Ocean ranges from 200 to 400 mg/L CaCO3, with higher values in coastal areas and lower values in the open ocean [10]. Hardness can be temporary or permanent; the water may contain calcium and magnesium bicarbonate, iron or magnesium. It is characterized because its softening is achieved by boiling, which consists of the bicarbonate precipitating, releasing carbon dioxide and lowering the pH value by carbonic acid formations [11].

The analysis of total hardness by titration with EDTA (ethylenediaminetetraacetic acid), allowing the quantification of Ca and Mg ions and their subsequent conversion to total hardness expressed as CaCO3 was carried out as follows:

10 ml of the sample water was taken in a flask, 1 ml of Buffer solution pH 10 was added and as an indicator a pinch of eriochrome black T (ENT) was used, the same which is intended to form a purple colored mixture; to proceed to titrate with EDTA; until the appearance of blue color.

Reactions:
$Ca^{+2} + Mg^{+2} + \text{Buffer pH10} \longrightarrow$

$Ca^{+2} + Mg^{+2} + \text{ENT} \longrightarrow (Ca^{-}\ Mg^{-}\ \text{ENT})$ purpura

$(Ca^{-}\ Mg^{-}\ \text{ENT}) + \text{EDTA} \longrightarrow (Ca^{-}\ Mg^{-}\ \text{EDTA}) + \text{ENT}$

During the titration 56.2 ml of 0.01M EDTA were consumed, where 53.5 ml corresponded to MgCO3 and 2.7 ml for CaCO3.

Calcium hardness: With the Buffer pH 10 the total concentration of the sample hardness such as Ca and Mg was determined. To determine the concentration of calcium present in the sample, 1 ml of Buffer pH 10 was added to the sample and then 20 drops of KOH were added to regulate the pH 10 to pH 12. From this result, the total hardness was subtracted to obtain the Mg concentration value.

Consumption = 2,7 ml de EDTA 0,01M en $CaCO_3$.
Consumption = 53,5 ml de EDTA 0,01M en $MgCO_3$.

$$CaCO_3 = \frac{C * M * pM * 1000}{Vm} \tag{1}$$

$$Ca = \frac{2{,}7 * 0{,}01M * 100 * 1000}{10} = 270\frac{mg}{L}$$

$$MgCO_3 = \frac{53{,}5 * 0{,}01M * 84 * 1000}{10} = 4494\frac{mg}{L}$$

$$(270 + 4494)\frac{mg}{L} = 4764\frac{mg}{L} Total\ hardness$$

$$Ca = \frac{2{,}7 * 0{,}01M * 20 * 1000}{10} = 54\frac{mg}{L}$$

Total hardness: 4764 mg/L - Calcium hardness: 270 mg/L= 4494 mg/L of Mg
MgCO3 = 4494 mg/L

It was determined that the hardness present in the sample was found in higher percentage in Mg.

Sulfates (SO4-2): With an average concentration of about 2.7 g/L, sulfate is one of the most present inorganic anions in seawater. It plays an important role in seawater chemistry and has a major impact on marine life. The complex process of sulfate cycling in the Pacific Ocean involves a variety of physical, chemical and biological interactions [12]. Sulfates are minerals whose structural unit is (SO4-2) groups, cations of Al, Na, Ca, K, Mg, Fe, and others can be bonded to sulfates. Among them are anhydrite and gypsum, which are quite common in the earth's crust [13].

Table 3. Absorbance and concentration data for sulfate

Absorbance	Concentration
280,904	0,481
281,455	0,486
284,789	0,487

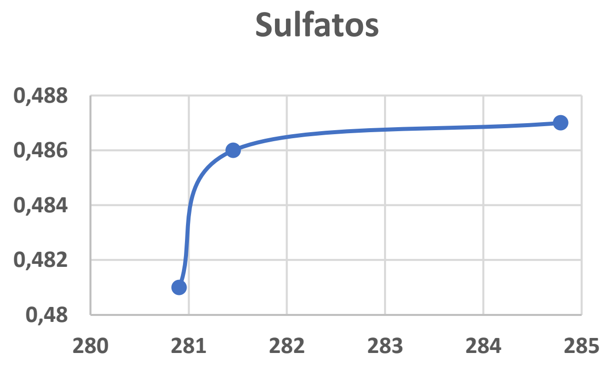

Fig. 4. Sulfate ion calibration curve.

The linearity obtained experimentally in the calibration curve for the sulfate ion shows a correlation coefficient r2=0.991 (Fig. 3). The curve was prepared with five concentration levels and a blank in the concentration range 48,000-312,000 ppm.

Chlorides: With an average concentration of about 19.4 g/L, chloride is the most present inorganic anion in seawater. It plays a fundamental role in seawater chemistry with great impact on marine life. The chloride cycle in the Pacific Ocean is a complex process involving a series of interactions [14].
Its analysis and concentration determination can be performed by several methods; in the present work, Mohr's method was used, which involves the quantitative determination of chloride, bromide or cyanide ions by titration with a standard solution of silver nitrate using Na2CrO4 or K as endpoint chemical indicator [15].

5 ml was taken and diluted in 95 ml of distilled water; from this dilution, 10 ml was used, 3 drops of K2CrO4 was added as end point indicator because AgCl is less soluble than Ag2CrO4 since the latter cannot be formed until Cl is fully reacted. There are three ways to determine the end point of the reaction: when a precipitate is produced, color change and disappearance of turbidity.

When the color change occurred, the titrant AgNO3 made a consumption of 20 ml, then the chloride calculation was performed:

$$Cl^{-1} = \frac{C * N * mEq * 100}{Vm} \qquad (2)$$

$$Cl^{-1} = \frac{20 * 0{,}0141 * 0{,}0355 * 100}{0.05} = 20{,}02g\ Cl^{-1}$$

100 ml M ------------ 2,002 g Cl^{-1}
1000 ml M ----------- x = 20,02 g Cl^{-1}

Interpretation:
For every 100 ml of sample there was 2.002g Cl-1.

Sodium chloride (NaCl) extraction: Water is a liquid, odorless, colorless and tasteless; it has a bluish tint and can only be detected in very deep layers. At atmospheric pressure, the freezing point of water is 0°C and the boiling point is 100°C. Water reaches its maximum density at 4°C and expands as it freezes, thus reducing its density, the same happens when the temperature increases from 4°C [16].

The evaporation of the water that was applied in the NaCl extraction process was that of the boiling point, in which 12 L of sea water was evaporated (fig.4); in the course of hours; forming a first crystallization.

Fig. 5. Evaporation process.

Subsequently, NaCl was dissolved in distilled water to separate the insoluble solids that precipitated during the evaporation process; a vacuum filtration process was applied to this solution.

Vacuum filtration: is an instrumental technique used in laboratories to separate solids from liquids or solutions. This type of filtration is used when solids are of interest or when gravity filtration is very slow; it is also an essential technique in recrystallization processes [17].

Recrystallization: is a common purification technique for NaCl, where an impure NaCl solution is dissolved in a suitable solvent, allowed to evaporate slowly to increase the NaCl concentration and then induce the precipitation of pure crystals. The study of Li et al. (2023) proposes a new NaCl recrystallization process by controlled evaporation which consists of optimized control system and optimized vessel design [18]. It was performed by simple distillation (Fig. 5); this is the most widely used procedure for the separation and purification of liquids, and it is the one that is always performed aiming to separate a liquid from its impurities [19].

Fig. 6. Simple distillation process.

When the first recrystallization was obtained, we proceeded to perform the analysis of chlorides to determine the percentage of purity of the sample obtained; here we dissolved 0.5 g of sample with humidity in 95 ml of distilled water, obtaining the following calculation:

$$\%NaCl = \frac{V * N * mEq * 100}{Pm} \qquad (3)$$

$$\%NaCl = \frac{51 * 0{,}0142 * 0{,}0585 * 1000}{0{,}5} = 8{,}4734$$

100 ml ----------- 8,413g NaCl
1000 ml -----------x = 84,13g NaCl

Obtaining a purity of 84.13% of NaCl.

Ion exchange: According to Degremont (1979), ion exchange is a reversible chemical reaction that takes place when an ion in a solution is exchanged for another ion of the same sign that is reattached to an immobile solid particle. Ion exchangers are a process that consists of taking advantage of the ability of resins to exchange ions between a solid phase and a liquid phase in a reversible way, that is to say that it returns to its original state and without permanent change in the structure of the solid. Generally, the great usefulness of ion exchange lies in the fact that ion exchange materials can be used over and over again since the exchanger material can be regenerated as the change it undergoes in the 'operation phase' is not permanent.

Strong acid cationic resin: Although abundant, seawater is not suitable for human consumption or for numerous industrial uses due to its high concentration of dissolved salts, including Ca and Mg ions, which cause it to have hardness. Ion exchange with cationic resin has become an effective method to remove these ions, softening seawater and making it useful for a variety of applications. Seawater contains Ca and Mg, salt-forming salts and increasing the hardness of the water; for this, the cationic resin of strong acid was used with its abbreviation SAC which is used in the form of sodium (Na), which performs an ion exchange allowing the hardness of the water produced by Ca and G and when saturated with hardness allows its regeneration with NaCl.

The reaction is as follows:
$2R_1\text{-}Na+Ca^{2+} \longleftrightarrow R_2\text{-}Ca+2Na^{+}$

$2R_1\text{-}Na+Mg^{2+} \longleftrightarrow R_2\text{-}Mg+2Na^{+}$

Producing a balance.

The hardness of the water decreased from 39.6 ml of EDTA consumption as a very high hardness; subsequently after the reaction with the resin the consumption was 2.1 ml of

EDTA, presenting high results in the removal of the hardness of our sample:
Hardness without resin:

$$Hardness = \frac{C * M * pM * 1000}{Vm} \qquad (4)$$

$$hardness\ CaCO_3 = \frac{1{,}9 * 0{,}01M * 100 * 1000}{10} = 190\frac{mg}{L}$$

$$hardness\ MgCO_3 = \frac{37{,}7 * 0{,}01M * 84 * 1000}{10} = 3166{,}8\frac{mg}{L}$$

At 2.1 ml of 0.01 M EDTA consumption, where the decrease in hardness of the sample water can be seen.

Hardness with resin:

$$Hardness\ CaCO_3 = \frac{0{,}10 * 0{,}01M * 100 * 1000}{10} = 10\frac{mg}{L} \qquad (5)$$

$$Hardness\ MgCO_3 = \frac{2 * 0{,}01M * 84 * 1000}{10} = 168\frac{mg}{L}$$

Total hardness: 178 mg/L - Calcium hardness: 10 mg/L= 168 mg/L of Mg.
MgCO3=168 mg/L.
The hardness present in the sample is almost null.

For the analysis of chloride in the sample obtained, the following calculation is analyzed:

$$Cl^{-1} = \frac{C * N * mEq * 100}{Vm} \qquad (6)$$

$$Cl^{-1} = \frac{24{,}3 * 0{,}0141 * 0{,}0355 * 100}{0{,}5} = 2{,}432673g\ Cl^{-1}$$

100 ml M ------------- 2,432673 g Cl^{-1}
1000 ml M ------------ x=24,32673 g Cl^{-1}

Avogadro's law states that “equal volumes react in the same way as long as their concentration is equal”.

Calculation for sodium chloride (NaCl):

$$V * N * mEq = g \qquad (7)$$

$$100 * 0{,}0142 * 0{,}0585 = 0{,}0830$$

$$25 * 0{,}0142 * 0{,}0585 = 0{,}02076$$

Reactions with silver nitrate for the determination of the purity of NaCl by titration of the sample obtained.

$NaCl+AgNO_3=AgCl+NaNO_3$

$$\%NaCl = \frac{V * N * mEq * 100}{Pm} \qquad (8)$$

Consumption: 24,3 ml de $AgNO_3$

$$\%Purity\ NaCl = \frac{24{,}3 * 0{,}0142 * 0{,}0585 * 100}{0{,}02076} = 97{,}23\%\ de\ Pureza\ de\ NaCl$$

3. Results.

Table 4. Final physical-chemical analysis of NaCl obtained from the seawater sample.

Components	Results	Units
Alkalinity	60	mg/L
Chlorides	24326,73	mg/L
Conductivity	1558	us/cm
Hardness	50	mg/L
Salinity	0,14	%
Total Solids	779	ppm
pH	6,22	
Temperature	27.5	ºC

The presence of CaCO3, CaSO4, NaCl, MgSO4, KCl, MgCl2 and MgBr2; indicates that the water sample is mineralized, i.e., it contains a significant amount of dissolved salts. The initial purity of NaCl in the sample was 84.7314%, which means that there was a considerable amount of impurities present. The sample was observed to have a high hardness, possibly due to the presence of calcium and magnesium ions. These ions can have negative effects in various industrial and domestic processes. Ion exchange with a strong cation resin was effective in removing calcium and magnesium ions, which increased the purity of the NaCl. Vacuum filtration and recrystallization were performed to obtain purer crystals.

0.0824 g of the NaCl crystals were dissolved in 100 ml of distilled water and the NaCl concentration was calculated, obtaining a value of 97.23%. The final purity of NaCl, 97.23%, is significantly higher than the initial purity, demonstrating the effectiveness of the purification process.

The different analyses and techniques used were carried out with the purpose of obtaining the highest degree of purity of NaCl; and to be able to use it in the laboratories allowing to develop and improve the techniques used for the purification of reagent grade NaCl decreasing the excessive use of chemicals that can affect the composition of NaCl. Sulfate results were 0.0 in the final NaCl sample. The market is also driven by the extensive use of NaCl as a raw material in the chemical industry to produce various chemicals, including chlorine, sodium hydroxide and sodium carbonates. These chemicals have a wide range of applications, which is further driving the demand for salt.

3.1. Comparison of initial and final analyses.

Table 5. Initial Analysis

Components	Units
Alkalinity	96 mg/L
Chlorides	20020 mg/L
Conductivity	64000 us/cm
Hardness	4764 mg/L
Salinity	0,14%
Total Solids	1380 ppm
pH	6,27
Temperature	27,5 ºC

Table 6. Final Analysis.

Components	Units
Alkalinity	60 mg/L
Chlorides	24326,73 mg/L
Conductivity	1558 us/cm
Hardness	50 mg/L
Salinity	0,14%
Total Solids	779 ppm
pH	6,22
Temperature	27,5 ºC

4. Discussion.

The salt market has few formally constituted Ecuadorian companies that share the local market for the supply of all the industrial and human consumption salts required at the national level, as we can see below:

Table 7. Distribution of the salt market.

Companies	Products	Annual Production Tons	Participation %	Average Dollar Price
Ecuasal C.A.	Cris-Sal	150.000	76%	18.000.000.00
Famosal S.A.	Sea And Salt	12.000	6%	1.440.000.00
Jueza S.A.	Pacific Salt	15.000	8%	1.800.000.00
Proquipil S.A.	Delisa	20.000	10%	2.400.000.00
Total, Sales And Production In Ecuador		197.000	100%	23.640.000.00

Source: Superintendencias De Compañías. Year 2013 Prepared by: Tammy Rodríguez Balseca.

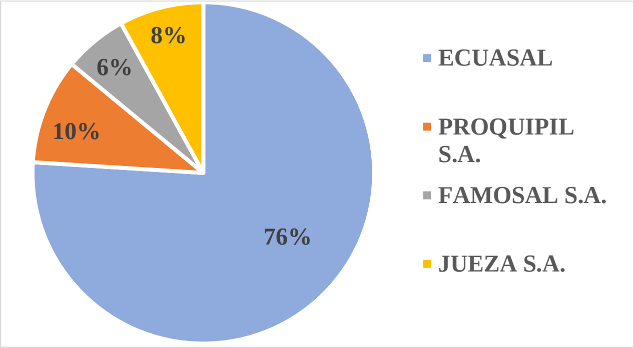

Fig. 6. Annual Production Tons.
Source: Superintendencia De Compañía. Year 2013 Prepared by: Tammy Rodríguez Balseca.

Artisanal salt production has become a neglected trade because traditional salt producers are unable to compete with the large production industries established in the market. Faced with such problems, the association of salt producers of Manta, Ecuador has developed a proposal for a refined salt production process that will contribute to the achievement of this objective. A technical and operational study is provided which is structured in the following components: product characterization, flow chart of the salt production process, geographic and micro location, and study of salt production capacities. The aim is for the association of salt producers to establish a business model to enter the local and national market [22].

NaCl is an important raw material in the chemical industry, since it has several uses. In the laboratory involved in the study, NaCl is used as raw material to manufacture parenteral solutions, such as: 0.9% NaCl injection and injection of 5% dextrose with 0.9% NaCl (mixed solution). These solutions are of great importance in the health field, they are used in rehydration therapies in cases of acute diarrhea and cholera, also for trauma, burns, when patients have a deficit of body Na+ and control the distribution of water in the organism.

According to the study conducted by Wang et al. (2020), a novel method for purification of reagent grade NaCl is by a combination of simple distillation and ion exchange membranes. This technique combines the advantages of these methods to effectively remove impurities such as organic compounds, heavy metals and inorganic salts [23].

It can be stated that the standards for reagent grade NaCl have a percentage of 99% to 100.5% purity both for pharmacopoeia and those that require even higher percentages such as ISO, however, there are standards that indicate that reagent grade NaCl can have a minimum of 95% purity, these are usually specific especially in local standards of certain countries which can accept this minimum level of purity such as the Mexican Official Standard (NOM) and the Ecuadorian Technical Standard (NTE) INEN 1204.

A comparison of NaCl purity standards established by the USP, the Codex Alimentarius and INEN 1234 showed some significant differences:

The USP indicates that its standards are the most demanding, especially when it comes to the degree of purity, since these grades are used in applications where high purity and reliability are required, such as in the manufacture of drugs, the minimum purity grade allowed by the Pharmacopeia is 99.0% [24].

The Codex Alimentarius standards are focused on food safety and establish a purity level that guarantees that the NaCl used in food does not compromise the health of the consumer; the accepted purity grade is 97.0% as a minimum [25].

INEN 1234, which is a national standard, establishes specific requirements for industrial grade NaCl, which is used in a wide variety of applications. The required purity level is lower compared to pharmaceutical and food standards in this case it allows 95% purity as a minimum [26].

5. Conclusions.

The initial characterization that was carried out allowed obtaining the values of the different components present in the sample to be analyzed and to start the present research, with this it was demonstrated that it is possible to obtain reactive grade NaCl using different techniques and analysis where the use of reagents is not excessively applied, since they change the composition of the different components found in the sample.

It was possible to obtain reagent grade NaCl from seawater from Crucita-Manabí, Ecuador, with a purity of 97.23%, although it did not exceed USP international standards (99% - 100.5%), it was demonstrated that it does exceed local standards, which allows its application. The key finding of this research shows that the seawater from Crucita-Manabí has a chemical composition suitable for NaCl extraction.

The purification technique used, which combines evaporation, recrystallization and ion exchange, was effective in eliminating the impurities present in the seawater and obtaining high purity NaCl, which determined that the ion exchange stage with cation resin was crucial for eliminating calcium and magnesium ions, The final physical-chemical analysis of the NaCl obtained confirmed that it meets purity standards, which makes it suitable for use in laboratories and industries that demand a high degree of purity.

The contributions of the present study demonstrate the feasibility of obtaining reagent grade NaCl from Ecuadorian seawater, which represents a significant contribution to the local industry, since this study can now be replicated to obtain high purity NaCl from other seawater sources, with potential application in various industries. The results obtained provide valuable information on the chemical composition of Crucita-Manabí seawater and its potential for NaCl extraction, which can contribute to the development of new industrial initiatives in the region. The relevance for local industry consists of the great impact of being able to obtain reactive grade NaCl from Ecuadorian seawater, since it reduces dependence on imports of this product, which translates into foreign exchange savings for the country. Promote the development of new industries and even existing industries that require high purity NaCl, such as the pharmaceutical, cosmetic and chemical industries, generating new employment opportunities in the industrial and scientific sector.

The regulations based on the present research are high quality international regulations; however, there are local regulations in different countries where a minimum percentage of 95.0% of reagent grade NaCl is allowed, which shows that the 97.23% of NaCl obtained in the present work can be useful as a reagent use.

In short, this study opens new possibilities for the use of seawater as raw material to obtain reactive grade NaCl, contributing to the industrial and scientific development of Ecuador.

6.- Author Contributions.

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3. Formal analysis: Segundo García; Ariana García
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8. Resources: N/A.
9. Software: N/A.
10. Supervision: Antonella Ferrin; Ramon Cevallos
11. Validation: Antonella Ferrin; Ariana García
12. Visualization: Thalía Caicedo; Ariana García

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