

Optimization of Rock Salt Washing Process to Remove Selected Chemical Impurities in Lake Katwe Rock Salt

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Abstract: Rich deposits of mineral salts, present in substantial economic quantities, characterize the geological bounty of Uganda. The primary location of this wealth is lake Katwe, a saline crater lake southwest of the country. However, all grades of rock salt extracted from Lake Katwe remain contaminated with chemical impurities such as calcium, magnesium, heavy metals and sulphate ions, even after solar evaporation process from the salt pans. There is a need to conduct research into possible contaminant removal methods which could increase the market value of the extracted salt, and add to the body of knowledge for future salt processing and chlor-alkali industries in Uganda. This study examined the effect of varying water volumes and temperature on the levels of Calcium, Magnesium and Sulphate ions in rock salt samples from Lake Katwe. The samples were methodically crushed and washed. The salt was then dissolved, crystallized, dried and meticulous analysis to determine the concentration of these impurities was carried out. The data was analysed and represented in Microsoft Excel and MATLAB. Results obtained revealed that a temperature of 40°C and salt-water ratio of 1:2 are the optimal parameters for maximum reduction of calcium, magnesium and sulphate impurities in rock salt. This analysis enhances the purity of salt harvested from Lake Katwe's solar evaporation process and offers optimal washing parameters to reduce impurities in brine, thereby improving plant yield and reducing production costs across various salt grades. It also provides new information to the mining communities at Lake Katwe on how to affordably improve the percentage purity of their rock salt. The study will also contribute novel industry-tailored data for future prospective salt processing and chlor-alkali industrial plants near the Lake while also filling a knowledge gap for further research on rock salt purification.

Keywords: Rock Salt, Chemical Impurities, Salt Washing, Temperature, Composition, Lake Katwe

1. Introduction

Uganda is home to an abundance of mineral salts that can be found in the brines and evaporite deposits of Lake Katwe. This closed saline crater is located in the southwestern region of the country, in Kasese district and within the Katwe-Kikorongo volcanic field. It sits at an elevation of approximately 885 meters and has a maximum area of 2.5 square kilometers, with a depth ranging from 0.4 to 1.6 meters and a circumference of around 9 kilometers. The water in Lake Katwe has a salt content of about 60%, and the salt deposits on the lake bed are estimated to contain around 12 million tonnes of salt, the composition of which varies with depth [1].

Lake Katwe salt consists of different salt grades and compositions including thenardite, burkeite, halite and glaserite [2]. For centuries, the communities living around Lake Katwe have relied on traditional, rudimentary methods to mine salt from the lake for various purposes such as human consumption, animal feeds, and preserving hides and skins. Today, these communities continue to mine salt, which remains the main economic activity in the area. Salt is extracted and purified at the lake using ancient, traditional, and artisanal methods that produce low-quality salt in limited quantities [3]. The production of salt is highest during the dry seasons, which are January to March and July to September, because most of the processes used in salt extraction and

purification rely on solar energy.

There are two methods used to harvest salt at Lake Katwe: underground mining and solar evaporation. Underground mining, also known as rock salt mining, involves mechanically extracting the salt deposits from the lakebed using drilling and blasting techniques. Iron poles are used to physically break and lift the rock salts from the bottom of the lake, which are then placed on a floating wooden rafter. This rafter is manually pulled to the shoreline, where the salt is offloaded, piled up, and sun-dried before being sold. This method of salt production is also referred to as rock salt mining [4].

The solar evaporation method for washing salt at the lake involves the use of salt pans with sizes ranging from 300 to 400 square meters, which are located in an area with a depth of around 1 meter. These pans have mud-lined walls that are supported by wooden stakes and woven with grass. The evaporation process relies on solar and wind energy, and involves allowing the brine to concentrate through solar evaporation before crystallization occurs. This method is used to produce salt at the lake using different salt pans [5].

During the salt washing process, solution salt from the lake is placed in a salt pan, which allows suspended solids to settle. After sedimentation and crystallization, the first grade of salt is obtained. This salt is then dissolved again and crystallized in a cleaner salt pan, resulting in cleaner and clearer salt crystals. Three grades of salt are produced using this method, which are classified as grade A, grade B, and grade C based on the level of visible impurities. Grade A is a high-quality crystallized salt that is extracted from clean salt pans during the long dry season and is primarily used for domestic consumption. Grade B is extracted from the bottom of the mud-lined salt pans in preparation for the end of the dry season and is mainly used for animal consumption. Grade C is mainly rock salt that is extracted from the lakebed below the surface brine and is primarily used for animal consumption, such as a cattle lick, and as a raw material for industrial use, such as in the processing of animal hides [6].



Figure 1. Different rock salt grades from Lake Katwe.

Lake Katwe rock salt is known to contain organic matter, dirt and chemical impurities like Calcium, Magnesium, Sulphate ions and heavy metals which must be reduced/removed before domestic and industrial use. In the chlor-alkali industry, the purification processes require brine with low concentrations of chemical impurities to prevent

corrosion, damaging of equipment and to have low cost of production with high yields. Although solar evaporation has been used to reasonably clean and improve the physical appearance of the different salt grades at the Lake, a lot of chemical impurities remain which can lead to high costs of chemical purification, as excessive consumption of water resources during dissolution and crystallization of different salt grades. Consequently, there is a need to develop optimum parameters that reduce some of the chemical impurities in Lake Katwe rock salt during the salt washing process.

This paper is intended to determine the optimum temperature and water volume required to remove Calcium, Magnesium and Sulphate ion impurities present in Lake Katwe rock salt. This information will help to improve the purity of harvested salt from the solar evaporation process in the Lake Katwe salt pans. In addition, this analysis will provide information that can improve effective plant yield and lower production costs of different salt grades by providing optimum salt washing parameters to reduce the chemical impurities in brine.

2. Related Work

Salt production occurs on a global scale, with annual production exceeding 200 million tons. Approximately one third of this total is produced through solar evaporation of sea water or inland brines, while another third is obtained through the exploitation of rock salt deposits both underground and on the surface. The remaining third is obtained in the form of brine, primarily through extraction of the solution [7]. The main constituents of raw brine are NaOH and Na₂SO₄, although it also contains magnesium and calcium ions [8]. There are mainly three conventional methods used to extract salt, classified according to the recovery method. Underground mining involves conventional mining of the underground deposits through drilling and blasting at depths between 100 and 1500 m below the surface, where the solid salt rock is removed and screened into various marketable sizes. Solar evaporation, which is the oldest salt production method, involves using solar energy to evaporate most of the water from salt pans, leaving behind salt crystals which are then harvested using mechanical means. Salt can also be extracted using large commercial evaporators called vacuum pans. Brine from underground wells is pumped into huge closed vessels under vacuum. The brine is then boiled at low pressure using injected steam, leading to formation of crystals [4].

After the salt has been extracted, the next step is to remove impurities like carbonates, sulphates using various methods including the SALEX method, vacuum salt manufacturing and chemical treatment. In the SALEX process, hydro-milling selectively ruptures the salt crystals where the impurities are embedded so that they are released, without formation of fines that increase salt losses. The ruptured crystals are then immersed in pure brine, leading to migration of impurities entrapped in the crystal crevices [8].

Vacuum crystallization involves evaporation of brine in a vacuum environment. Brine is first purified and then fed into a vacuum crystallizer at low pressure, leading to formation of salt crystals which are separated from the remaining brine. The crystals are then washed and dried to remove any remaining moisture, resulting in a high purity salt product. This method usually results in a purer product than other methods of salt production [9].

Various Ugandan scientists have undertaken research at the local level to assess the characteristics of Lake Katwe salt. Lake Katwe brines have been investigated to determine their chemical, mineralogical and morphological composition. Inductively Coupled Plasma–Atomic Emission Spectrometry (ICP–AES), Inductively Coupled Plasma–Sector Field Mass Spectrometry (ICP–SFMS), ion chromatography, and potentiometric titration were used to determine that the lake brine was highly alkaline and rich in SO_4^{2-} ions with varying amounts of Ca^{2+} and Mg^{2+} ions [1]. The potential of using Lake Katwe raw salt for salt extraction has been evaluated by evaporating brine through 12 evaporation stages, and analysing the precipitates using XRD and FEG-SEM [10]. It was noted that the first and second precipitates included both calcium sulphates with increasing density. It was also noted that lower evaporation rates of about 10% are required to precipitate sulphates, while carbonate ions precipitate at higher evaporation rates, usually along with calcium and magnesium ions.

Research has also been undertaken to evaluate the possibility of improving the salt extraction process from raw rock salt. Numerical models have been created to enhance the efficiency of solar salt pans in Lake Katwe for salt extraction from raw rock salt. [10]. The models were used to determine the optimal depth of salt pans, which was found to be 0.1 m, for higher salt densities. Phase models have also been developed to understand the phase chemistry of Lake Katwe brine [11]. Furthermore, a process was developed for extracting commercial salts from brine in Lake Katwe, and the salt pans were modelled to improve the quality and quantity of salt production. [12]. The process involved a precipitation sequence starting with sulphates, followed by carbonates of calcium and magnesium. The study revealed that halite was the dominant mineral crystal, followed by thernadite, trona, and glaserite. To obtain maximum crystal yield, the feed concentration, mixer speed, residence time, and heating load were determined to be 0.248 g of NaCl/g of solution, 830 rpm, 50 min, and 1400 W, respectively.

Significant research has been conducted globally for large-scale extraction and purification of salt, as well as optimizing the local salt purification process through solar evaporation. For example, salinity gradient solar ponds have been developed for applications in air conditioning and refrigeration [13], industrial heat processes [14], desalination [15], chemical production [16] and generation of electrical power [17] along with several one dimensional [18, 19] and two-dimensional models [20, 21] to evaluate their performance.

Little research has been conducted on improving the further

purification of salt grades extracted by the Lake Katwe salt farmers to affordably remove chemical impurities after evaporation and increase the market value of the salt. This is despite the fact that Grade A salt, which is the highest quality crystallized salt extracted from the clean salt pans, has been found to still contain significant amounts of Ca, Mg and S elements [2].

This paper is intended to address this gap by determining the optimum temperature and water volume required to remove Calcium, Magnesium and Sulphate ion impurities present in Lake Katwe rock salt. This information will help to improve the purity of harvested salt from the solar evaporation process in the Lake Katwe salt pans. In addition, this analysis will provide information that can add to the body of knowledge on improving effective yield and lowering of local production costs of different salt grades by providing optimum salt washing parameters to reduce the chemical impurities in brine.

3. Methodology

3.1. Sample Size

The study was mainly focused on rock salt obtained from Lake Katwe, and a total of about 40 samples were obtained from washing rock salt at different temperatures using different quantities of water. These were analysed for the quantity of selected impurities recovered.

3.2. Crushing the Rock Salt

Rock salt was crushed using a wood mortar and pestle to small particle size. It was dried in an air oven at 110 °C till no change in weight.

3.3. Crystallization of Salt

500 g of crashed rock salt was dissolved in 500 g of double distilled water. The mixture was left to settle and a clear salt solution decanted off. The solution was heated to near boiling and evaporated for 30 minutes. It was then left to cool below 0°C for about 12 hours till all the salt crystallized out. The crystallized salt was dried and kept in a desiccator. The salt solution was not allowed to boil.



Figure 2. (a) dissolved salt and (b) decanted rock salt.

3.4. Washing of Salt

Effect of water volume: The amount of salt crystallized was weighed and an equal volume of water (1:1 w/v) was added. The mixture was stirred to dissolve the salt, heated to near boiling and evaporated for 30 minutes. It was then cooled below 0°C for about 12 hours till all salt crystallized out. The procedure was repeated to wash the salt 2 more times. The above experiment was repeated with salt-water ratios of 1:1.5, 1:2, 1:2.5, 1:3 w/v.

Effect of temperature: Using salt/water ratio of 1:1 w/v, salt was washed using water at different temperatures i.e., 30, 35, 40, 45, 50°C.

3.5. Determination of Calcium and Magnesium

Preparation of sample: 5 g of the salt sample was dried in an air-oven at about 150°C for 4 hours and cooled in a desiccator for subsequent tests. 2 g of the dried sample were dissolved in 20 ml of water in a beaker. The mixture was heated to boiling, filtered and the filter paper was washed free of chloride. The sample salt solution was cooled and made up the solution to 100 ml. This solution was used for subsequent tests.

Determination of Ca and Mg: 100 ml of the salt solution was transferred into a 250 ml conical flask, 10 ml of ammonium chloride ammonium hydroxide buffer solution, 5 drops of Eriochrome black T indicator solution were added and titrated against standard EDTA solution till wine red colour of the solution changed to pure blue end point. The volume of the standard EDTA solution used in the titration was noted (V1). 100 ml of the salt solution was transferred into a 250 ml conical flask and 6 ml sodium hydroxide solution was added and stirred well. 0.2 g murexide indicator was added to the solution and it was titrated against standard EDTA solution till wine red colour of the solution changed to pure blue end point with murexide. The volume of the standard EDTA solution used in the titration was noted (V2).

$$\text{Calcium (Ca), percent by mass} = A * V2 * m \quad (1)$$

$$\text{Magnesium (Mg), percent by mass} = 6068 * A * (V1 - V2) * m \quad (2)$$

where A is the Calcium equivalent in mg of 1 ml of EDTA solution determined, V1 is the Volume in ml of standard EDTA solution used (for Ca & Mg), V2 is the Volume in ml of standard EDTA solution used for Ca and m = Mass in g of the dried taken.

3.6. Determination of Sulphate

Procedure 1: 20 ml of standard barium chloride solution were pipetted into a conical flask. 2 drops of hydrochloric acid and 10 ml of ammonium chloride ammonium hydroxide buffer solution were added. The mixture was diluted with water to 50 ml, 5 drops of Eriochrome black T indicator solution were added and titrated against standard EDTA solution to pure blue end point. The titre value was noted (A1).

Procedure 2: 100 ml of the salt solution and add 2 drops of hydrochloric acid were mixed and heated to gently boil. To the hot solution, 20 ml standard barium chloride solution were added. Boiled gently for about 5 min and then cooled to room temperature. 10 ml of ammonium chloride - ammonium hydroxide buffer solution and five drops of Eriochrome black T indicator solution were added and titrated against standard EDTA solution to pure blue end point. The titre value was noted (B1).

$$\text{Sulphate (SO}_4\text{), percent by mass (on dry basis)} = \frac{(0.96 A1 + V1 - B1)}{m} \quad (3)$$

where A1 is the volume in ml of standard EDTA solution used in Procedure 1, V1 is the volume in ml of standard EDTA solution used (total Ca + Mg), B1 is the volume in ml of standard EDTA solution used in Procedure 2 and m is the mass in g of dried salt sample.

3.7. Data Analysis

The average amount of Ca, Mg, and SO_4^{2-} content was determined using Microsoft Excel. The data was also represented in form of tables and line graphs using Microsoft Excel and MATLAB.

4. Results and Discussion

4.1. Effect of Amount of Water on Magnesium Ions in Lake Katwe Rock Salt

Table 1 shows the results obtained for the concentration of Magnesium ions after washing Lake Katwe rock salt with different volumes of water. Each of the salt samples was washed three times and the results are as shown;

Table 1. Composition of magnesium ions against water volumes for different washing times.

Composition of Magnesium ions after each wash (g/l)			
Amount of water (l)	1 st wash	2 nd wash	3 rd wash
0.5	0.1135	0.0973	0.0892
0.75	0.0973	0.0811	0.0730
1.0	0.0811	0.0649	0.0649
1.25	0.0811	0.0649	0.0649
1.5	0.0811	0.0649	0.0649

From Table 1, an increase in the amount of water for the three washing times, leads to decrease in the composition of Mg^{2+} ions in Lake Katwe rock salt and becomes constant at 1 litre (1:2 salt-water ratio). Further increase in water volume beyond 1 litre and number of washing time does not affect the change in composition of the Magnesium ions hence it remains at 0.065 g/l. Overall, the results suggest that the concentration of magnesium ions in Lake Katwe rock salt can be effectively reduced by washing it with at least 1 litre of water using a 1:2 salt-water ratio. Beyond this point, additional water or washing cycles do not further reduce the concentration of magnesium ions. A similar investigation was performed to determine dissolution of Mg^{2+} ions in different

solvents, including deionized water, and determined that 20 mmol of Mg^{2+} ions dissociated at a MgO concentration of 2000 $\mu\text{g/ml}$ [22]. This confirms that the concentration of Mg^{2+} ions in a solution can be affected by various factors, such as the solvent used and the concentration of other ions or compounds present.

4.2. Effect of Amount of Water on Calcium Ions in Lake Katwe Rock Salt

Figure 3 shows the results obtained for the concentration of Calcium ions after washing Lake Katwe rock salt with different volumes of water. Each of the salt samples was washed three times and the results are as shown;

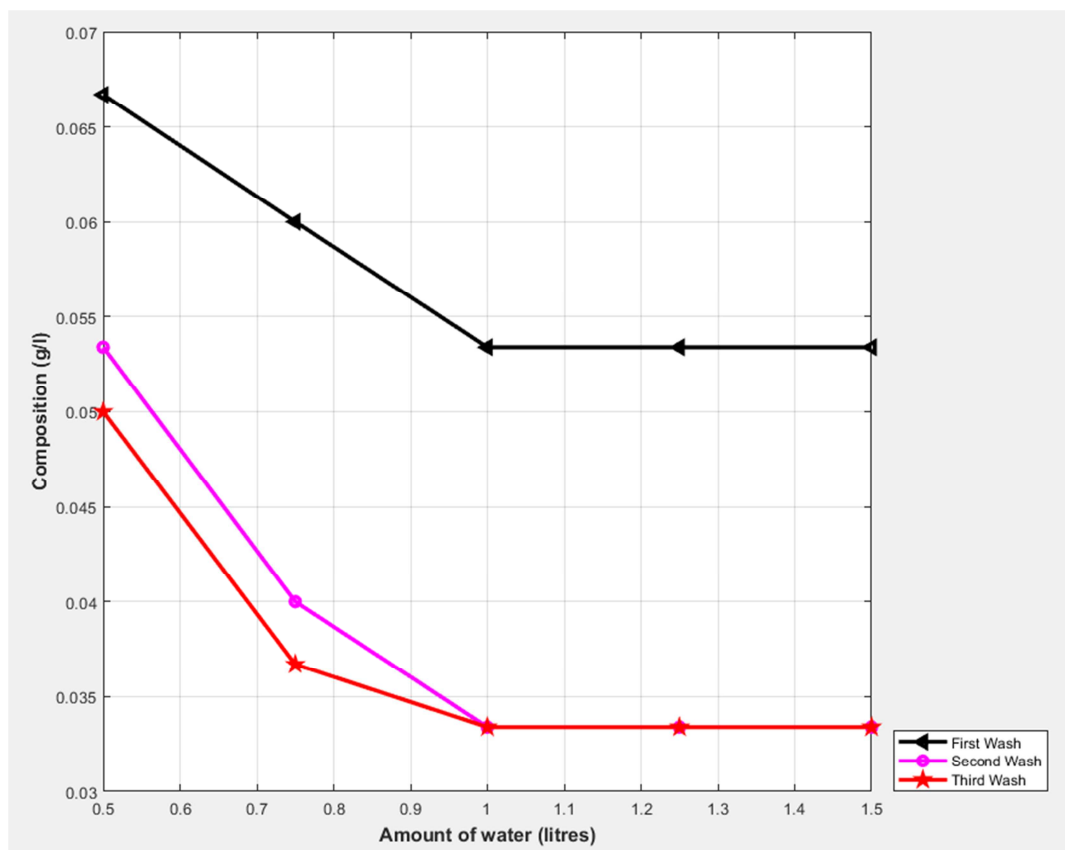


Figure 3. A graph of composition of Calcium ions against water volumes for different washing times.

An increase in water volumes for the three washing times, leads to decrease in the composition of Calcium ions in rock salt and becomes constant at 1 litre (1:2 salt-water ratio). Further increase in water volume beyond 1 litre and number of washing time does not affect the change in composition of the Calcium ions hence it remains at 0.034 g/l. R. D. R. Silva [23] removed calcium ions from well water by precipitation with sodium phosphate and showed similar percent calcium removal at pH of 11.5, saturation pressure of 4 bar, a recycling ratio of 30% and a sodium oleate concentration of 20 mg l^{-1} . These findings suggest that washing rock salt with at least 1 litre of water using a 1:2 salt-water ratio can effectively reduce the concentration of calcium ions. However, using more water or conducting more washing cycles does not result in further reduction of calcium ions in the solution.

4.3. Effect of Amount of Water on Sulphate Ions in Lake Katwe Rock Salt

Figure 4 shows the results obtained for the concentration of Sulphate ions after washing Lake Katwe rock salt with

different volumes of water. Each of the salt samples was washed three times and the results are as shown;

An increase in the amount of water for the three washing times, leads to decrease in the composition of Sulphate ions in the rock salt and becomes constant at 1 litre (1:2 salt-water ratio). Further increase in water volume beyond 1 litre and number of washing times does not affect the change in composition of the Sulphate ions hence it remains at 12.5 g/l. The results suggest that washing rock salt with at least 1 litre of water using a 1:2 salt-water ratio can effectively reduce the concentration of sulphate ions. However, using more water or conducting more washing cycles does not result in further reduction of sulphate ions in the solution. However, B. Sawadogo [24] showed higher sulphate percent removals of sulphate ions from borehole water using a nanofiltration and reverse osmosis pilot membrane, showing sulphate retention rates of 97.0% to 98.7% and 98.4% to 99.0% respectively. This implies that subsequent treatment processes would need to be included in the salt purification process to further reduce levels of sulphate in rock salt.

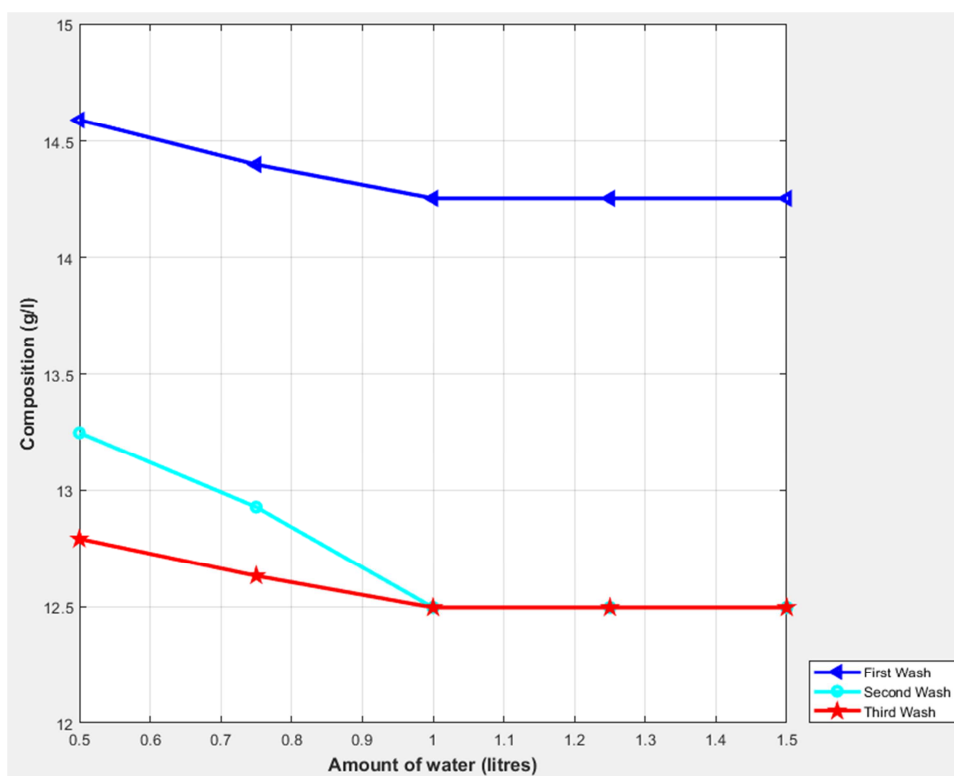


Figure 4. A graph of concentration of sulphate ions against water volumes for different washing times.

4.4. Effect of Amount of Temperature on Magnesium and Calcium Ions in Lake Katwe Rock Salt

Figure 5 shows the results obtained for the concentration of Magnesium and Calcium ions after washing Lake Katwe rock salt with water at different temperatures.

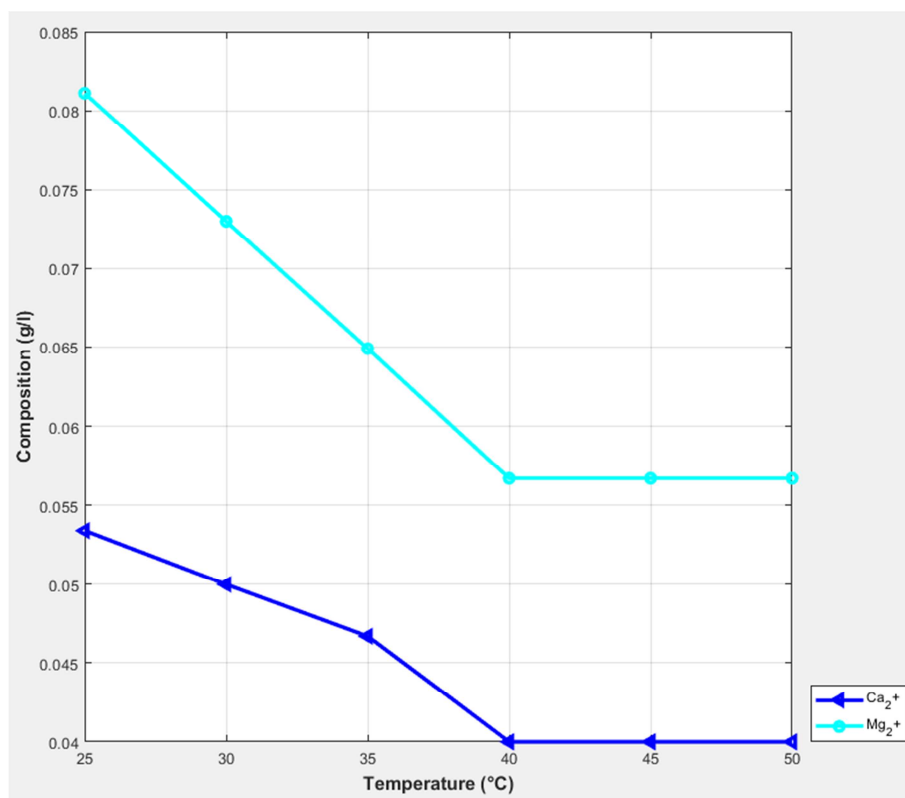


Figure 5. Composition of calcium and magnesium ions at increasing temperatures.

From the graph, the increase in temperature of water decreases the composition of both Magnesium and Calcium ions in Lake Katwe rock salt and the concentrations become constant at 40°C. Further increase in temperature above 40°C does not affect the change in composition of Magnesium and Calcium ions in the rock salt hence it remains at 0.057 g/l and 0.04 g/l respectively. The results indicate that increasing the temperature of the water up to 40°C can effectively reduce the concentration of these ions. However, beyond this point, increasing the temperature further does not result in further reduction of magnesium and calcium ions in the rock salt. However, C. Chen [25] removed magnesium ions from manganese electrolytes using low temperatures of -5°C using

low temperature crystallization. This implies that while increasing the temperature up to 40°C may be effective in reducing the concentration of these ions in some cases, other methods such as low temperature crystallization can also be suitable for certain types of calcium and magnesium ion removal.

4.5. Effect of Amount of Temperature on Sulphate Ions in Lake Katwe Rock Salt

Figure 6 shows the results obtained for the concentration of Sulphate ions after washing Lake Katwe rock salt with different water at different temperatures.

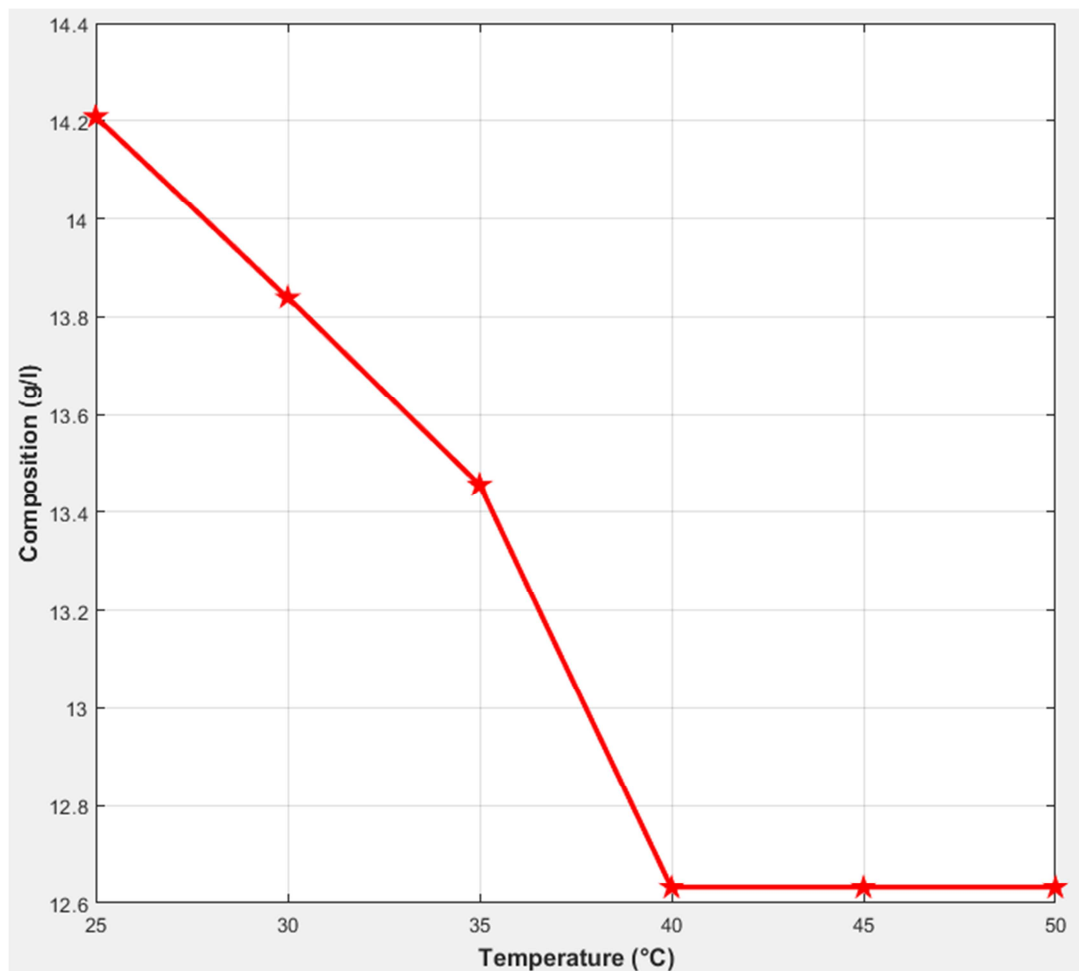


Figure 6. Composition of sulphate ions with increasing temperature.

An increase in temperature of the water used for washing decreases the composition of Sulphate ions in Lake Katwe rock salt and it becomes constant at 40°C. This shows that further increase in temperature above 40°C does not affect the change in composition of sulphate ions in the rock salt hence it remains constant at 12.63 g/l. The results indicate that increasing the temperature of the water up to 40°C can effectively reduce the concentration of these ions. However, beyond this point, increasing the temperature further does not result in further reduction of sulphate ions in the rock salt.

This temperature is similar to average brine evaporation temperature recorded by H. Kasedde [5], who used solar energy to extract salt from Lake Katwe and noted an average brine temperature of 30.94°C.

5. Conclusion

The study provides valuable insights into the effect of varying water volumes and temperature on the levels of calcium, magnesium, and sulphate ions in rock salt samples

from Lake Katwe. The results obtained suggest that a temperature of 40°C and salt-water ratio of 1:2 are the optimal parameters for maximum reduction of calcium, magnesium, and sulphate impurities in rock salt. These findings offer practical and affordable solution to the mining communities at Lake Katwe on how to improve the percentage purity of their rock salt. Additionally, the study provides novel industry-tailored data for future prospective salt processing and chlor-alkali industrial plants near the Lake. The research conducted in this study fills a knowledge gap in the field and serves as a basis for future research on the purification of rock salt from other areas contaminated with similar chemical impurities. Based on the findings of this study, there are several directions for future research that could help to improve our understanding of the purification of Lake Katwe rock salt. It would be beneficial to investigate the effect of different washing solutions on the concentration of magnesium, calcium, and sulphate ions in the rock salt. For example, using solutions with different pH values or different types of chelating agents could potentially lead to more effective removal of these ions. Secondly, it would be important to explore the effectiveness of different types of post-treatment processes to further reduce the concentration of sulphate ions in the rock salt. Thirdly, it would be interesting to investigate the effect of other parameters such as the agitation speed and duration of washing on the concentration of ions in the rock salt. By optimizing these parameters, it may be possible to further improve the efficiency of the purification process.

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