



Effect of Temperature on Mineral Precipitation Sequence of Lake Katwe Brine during Evaporation

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Authors' contributions

This work was carried out in collaboration among all authors. Author JDL initiated the idea, designed the study and wrote the first draft of the manuscript. Author HK co-initiated the idea, developed it, selected the materials and methods for the study. Author JBK assisted in data analysis as well as giving technical guidance during the study. All authors read and approved the final manuscript.

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ABSTRACT

The effect of temperature on the mineral precipitation sequence during evaporation of the natural brine from lake Katwe was studied. Laboratory isothermal evaporation experiments at temperatures (30, 40, 50, 60 and 70°C) using the original Katwe surface brine were undertaken. PHREEQC, a solubility software was used to predict the crystallization paths at the different study temperatures. The mineralogy and morphology of the precipitates were determined by the XRD and SEM techniques, respectively. The precipitation sequence majorly started with sulphates followed by chlorides and lastly carbonates at all temperatures after precipitation of the calcium-magnesium carbonates. Halite emerged the most dominant mineral, with thenardite, trona and glaserite following respectively. Thenardite, glaserite and burkeite precipitation flourished at lower temperatures (30 and 40°C) whereas soda ash precipitation flourished at higher temperatures (60

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and 70 °C). Halite equally precipitated at all temperatures thus not showing a temperature preference in this study. Temperature variation is a key design parameter in the extraction of mineral salts from the Brines of Lake Katwe.

Keywords: Lake Katwe; evaporation temperature; PHREEQC; precipitation.

1. INTRODUCTION

For centuries, salt mining at Lake Katwe has been a source of income to the salt miners due to the readily available domestic salt market [1], [2]. The lake has the largest salt reserves in Uganda [3] and therefore has a huge potential to contribute to the local and national economy of Uganda. At the lake, salt mining is done by both men and women during the dry seasons (February to April and July to September) [4,5]. For the production of salt grades, I and II, brine is channelled from the Salt Lake into mud lined salt pans where it is evaporated until salt is formed. This salt is scraped from these ponds starting with grade I followed by grade II. Salt Grade III commonly known as Mahonde is harvested by men wielding iron bars which they use to hack the Lake bed.

These rudimentary methods produce low quality salt mainly during the dry seasons. This salt is not suitable for food flavouring, neither can it be in the chemical industries and is of low value. The salt production is seasonal, heavily reliant on the availability of solar energy leading to intermittent supply and thus not viable economically. The salt from lake Katwe is of low quality because halite co-precipitates with other salts due to an uncontrolled salt crystallization process. Previous thermodynamic and experimental studies indicate the precipitation of thenardite, halite, glaserite, sylvite, arcanite, trona, soda ash, kaliginite, nahcolite, and thermonatrite upon brine evaporation [6,7]. The aforementioned salts have several applications i.e. halite is used as a flavour enhancer for food, de-icing agent for roads, and in leather tanning; sodium carbonate is used in the manufacture of glass, pulp and paper; arcanite is used in fertilisers and sodium sulphate as a filler in detergent formulations.

These salts are usually manufactured on an industrial scale by fractional crystallization with an acceptable purity of over 90% [8]. Kasedde et al. [7] investigated the mineral recovery sequence at 30°C; the precipitation sequence at other temperatures is not known. The solubility

of the minerals identified in the isothermal evaporation experiment at 30°C might vary with change in temperature which could be used in the design of the separation process scheme. It is upon this background that a temperature range was chosen to examine the effect of temperature on the phase behaviour of the brine.

The design of the salt extraction process is preceded by a thorough understanding of the thermodynamics of the multi-component electrolyte system; identification of the list of minerals which are more likely to precipitate, operating temperature, and the feasible path for the extraction of the required product [9].

This study therefore aims at understanding the lake Katwe brine salt precipitation at different temperatures with the view of identifying optimal conditions for salt production on a commercial scale. The thermodynamic behaviour of the brine will be vital in the design of an effective commercial salt extraction process that is pertinent for optimal exploitation of the salt reserve.

2. MATERIALS AND METHODS

2.1 Isothermal Evaporation Experiments

The rainy season surface brine was sampled from Lake Katwe and stored in 1.5 L plastic bottle at room temperature prior to the evaporation study. The brine had not been saturated since no solid precipitates were observed. Using 10 ml of the original brine from Lake Katwe, the polythermal evaporation experiment was run at five different temperatures (30, 40, 50, 60 & 70 °C). These were chosen because they are easily achievable on a commercial scale with less material selection problems and the evaporation process is faster compared to lower temperatures. Furthermore, the solubilities of the probable minerals are widely published for this temperature range [10,11]. Before evaporation was started, the biological and organic materials were filtered off using a 5 µm membrane. The filtrate was then poured into a 50 ml beaker which was then placed in a Labcon thermostatic water bath at

Table 1. Brine composition [4]

pH	Brine Temp (°C)	Density (g/ml)	EC (us/m)	Major ions (g/l)								
				Na ⁺	K ⁺	Mg ²⁺	Ca ²⁺	Cl ⁻	Br ⁻	SO ₄ ²⁻	HCO ₃ ⁻	CO ₃ ²⁻
9.72	25.2	1.15	14109	69.6	11.6	0.0519	0.0048	44.2	0.461	32.7	18.2	38.8

30°C. A mercury thermometer was immersed in a water bath to ensure that a constant temperature was being maintained. At several intervals, the formed precipitates were filtered off using a 5 µm membrane and then left to dry overnight in open air on an aluminium foil. This procedure was repeated for the other study temperatures (40, 50, 60 & 70°C).

2.2 Mineralogy and Morphologies of the Precipitates

To ascertain the mineralogical composition of the evaporation precipitates, Powder X-ray diffraction was undertaken using a Philips X'Pert – PRO theta-theta PW3050/60 goniometer diffractometer CuKα, (λ = 1.540598 Å) X-ray tube, the diffraction interval was between 2θ–1°–90.5° with a step size of 0.013°. The generator voltage and tube current were 45 kV and 40 mA respectively. The QUALX2.0 search-match identification software that runs with the Crystallography Open Database (COD) powder diffraction database was used to identify the minerals [12]. Also, the morphology of the precipitates was ascertained by a Zeiss Supra 55 VP Field Emission Scanning Electron Microscope (FEGSEM); all samples were carbon coated before SEM analysis in order to make them electrically conductive. The system had an EDAX energy-dispersive X-ray (EDX) spectrometer that allowed an elemental composition analysis with a detection limit of approximately 0.5%. The evaporation experiment, XRD and SEM investigations were done at the Sefako Makgatho Health Sciences University Electron Microscopy Unit, South Africa.

2.3 Thermodynamic Modelling

In the present study, the precipitation sequence and the phase assemblage during evaporation of the lake brine at different temperatures was calculated using the PHREEQC Interactive geochemical software, version 3.4.0 [13]. Brine Evaporation was modelled with the Pitzer database due to the hyper-salinity [14] of Lake Katwe brine. Before the simulation, the Pitzer database was revised to suit the current research. Thermodynamic data of soda ash was

incorporated in the Pitzer database. Furthermore, the analytical expressions that determine the equilibrium constants at varying temperatures were also added for those phases (burkeite, trona, nahcolite, natron, glaserite, and magnesite) in which they were absent. All the aforementioned data was got from the Specific Ion Interaction Theory Database (SIT). Evaporation was simulated in steps, 51 moles of water in 102 steps was removed (92% of the initial brine was evaporated). The sequence of the modelled mineral precipitation at the study temperatures during the evaporation was noted. The average brine composition (rainy and dry seasons) of the major ions in Lake Katwe brine as shown in Table 1 was used as PHREEQC input [4].

3. RESULTS AND DISCUSSION

3.1 Characterization of the Precipitates

3.1.1 Morphological composition of the precipitates

Fig. 1 shows the various microstructures of the precipitates observed during brine evaporation. The SEM micrographs reveal distinctive microstructures of the dominant mineral phases that precipitate out of the brine. At all the study temperatures, precipitation of Halite is observed. The waxy massive form (N) which is due to the hygroscopic nature of the salt [15,16] and the euhedral cubic crystals (M) [17] are typical halite morphologies. Massive forms of halite are also observed as coatings composed of anhedral crystals (O) [16] on top of salt (rice) cake normally a fitting description for thenardite crystals [18,19]. The aforementioned is evidence of mineral stratification and co-precipitation; the latter needs to be minimised if pure salts for industrial use are to be produced. Trona is observed as blade-like [4] and columnar [19] crystals (G), on top of thenardite (salt cake) (P) and sandwiching Halite crystals ((D), (I) and (J)). Hexagonal prismatic crystals of hanksite are also observed as one of the precipitating mineral phases (E) [4,19]. Similar microstructures were identified by Kasedde and co-workers [7] in an isothermal evaporation study.

As temperature increases, the mineral phase stratifications become more distinct unlike at lower temperatures where phases are more intermixed. This is attributed to the increasing difference between the solubilities of the

constituent minerals; as the temperature increases, the difference in the saturation with respect to the mother solution of the different minerals increases as well.

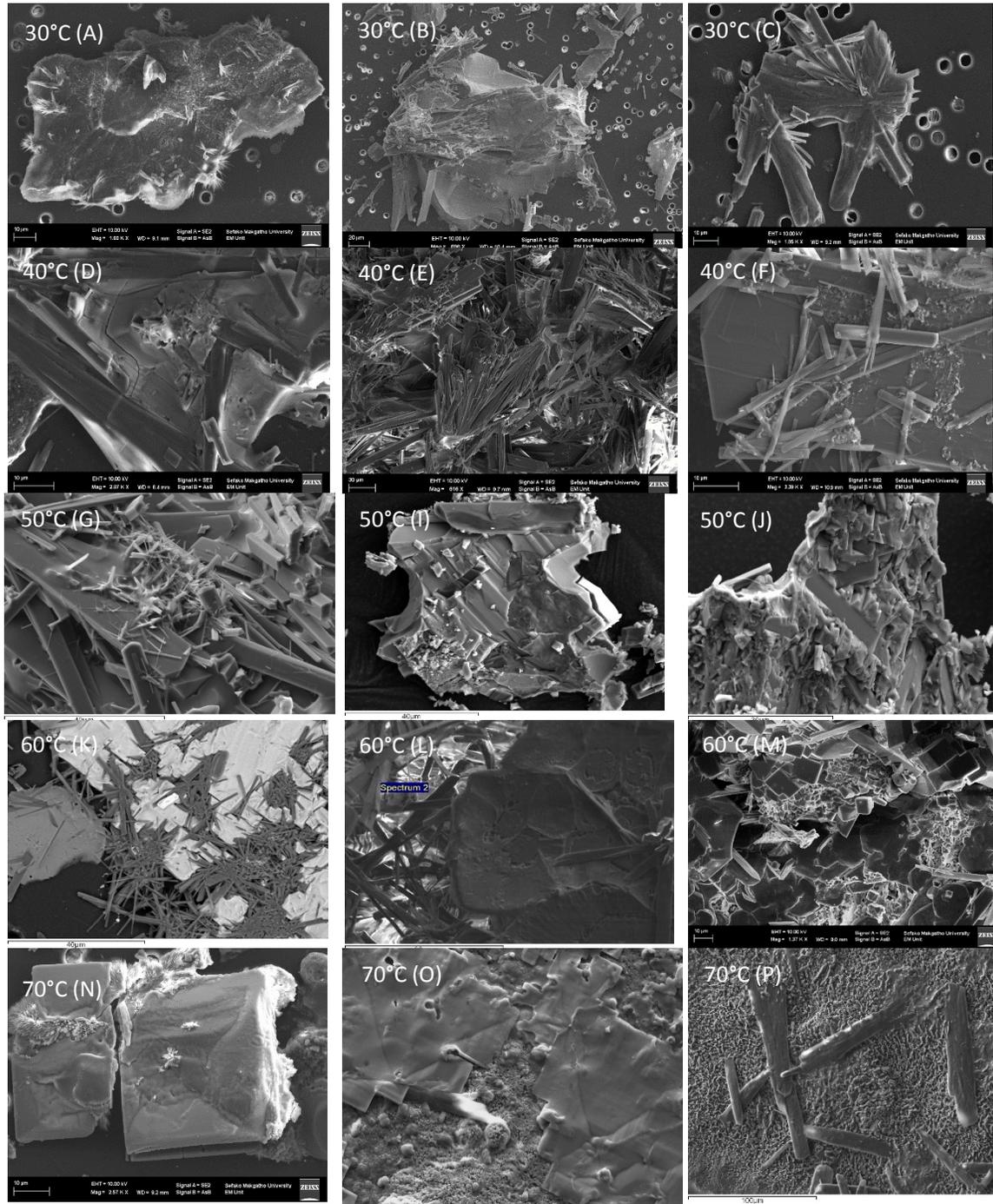


Fig. 1. SEM micrographs of the precipitated minerals from the evaporation experiments

3.1.2 Mineralogical composition the precipitates

A typical XRD measurement showing several mineral characteristic peaks is shown in Fig. 2. Several XRD patterns were used to identify the minerals in this study at the different study temperatures. Generally, the XRD pattern for the precipitates shows that, carbonates and sulphates of calcium and magnesium (calcite, tychite, and dolomite) dominate the first precipitates with increasing brine concentration due to evaporation as predicted by Arad and Morton [20]. This is attributed to their low solubilities within the study temperature range.

The subsequent precipitates are dominated by the Na-K-Cl-SO₄-CO₃-HCO₃-H₂O system derived minerals due to their abundance in the brine [21]. These majorly include thenardite, halite, burkeite, thermonatrite, nahcolite, glaserite, trona, natrite, hanksite and natron. The sulphate minerals dominate the first precipitates, followed by the chlorides, and lastly the carbonates; these at all times coprecipitate with other minerals and thus no pure mineral precipitate is observed. This can be attributed to the precipitation overlap of the different mineral phases and the characteristics of the involved cations Na-K being very similar.

Arad and Morton [20] and Kasedde et al. [4] identified northupite as major evaporite precipitating from the brine but not observed in this study. The reason for this could be that surface brine was used yet northupite forms at about 20 m below the surface after several thousand years [20].

Tables 2-6 show the mineral composition of the precipitates at the different study temperatures. Four samples are analysed at each temperature

and these were collected as soon as crystals appeared. At all the study temperatures, halite emerges as the most abundant mineral as the phase is observed in almost all the samples. This is also evident by the dominance of its microstructures in Fig. 1.

At 30°C, apart from trona, thenardite is the most abundant followed by halite and then natrite. halite, burkeite, glaserite and then hanksite is the order of prominence at 40°C with thenardite appearing as a trace. The existence of the double sulphate salts at this temperature explains the small amounts of thenardite. The sodium carbonate and bicarbonate minerals dominate the precipitates at 50°C with traces of arcanite and thenardite. For 60°C as well, the sodium carbonate and bicarbonate minerals dominate the precipitates with the sulphates and chlorides of sodium precipitating as hanksite. Natrite and hanksite are observed to be the most dominant phases at 70°C.

3.2 Thermodynamic Modelling

At 30°C, 40°C, and 50° evaporation temperatures, the major salt precipitation sequence starts with thenardite followed by trona, halite and lastly glaserite (Figs. 3-5). This is in agreement with the previous isothermal evaporation study at 30°C conducted by Kasedde et al. [7] and Lwanyaga et al. [22]. However in this study, sylvite does not saturate with the brine and therefore doesn't precipitate unlike in the previous phase developments study by Kirabira and co-workers [6]. It is worth noting that as the evaporation temperature increases from 30°C to 50°C, the difference in concentration factors at which glaserite and trona precipitate increases as well.

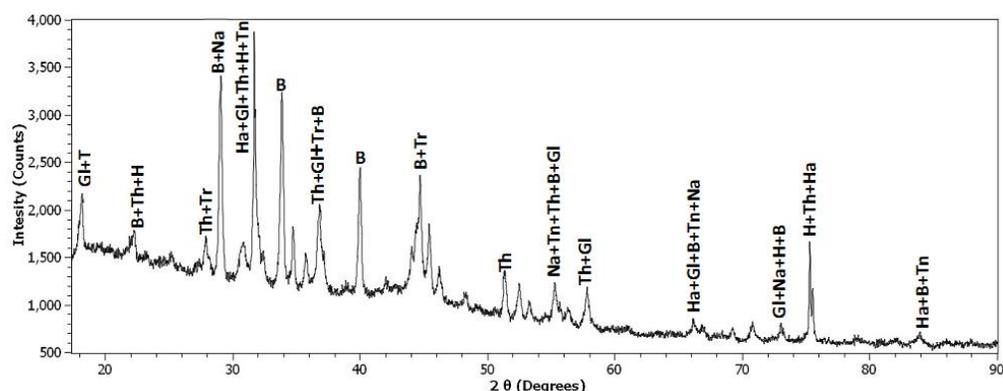


Fig. 2. XRD Pattern of mineral precipitates (GI-Glaserite, Th-Thenardite, Ha-Halite, Tr-Trona, B-Burkeite, Na-Natron, Tn-Thermonatrite, H-Hanksite)

Table 2. Mineralogical composition of the precipitates at 30°C

Mineral Phase	30°C				
	Chemical Formula	Time Interval			
		6 Hrs	12 Hrs	15 Hrs	16 Hrs
Calcite	CaCO ₃	X	X	X	
Anhydrite	CaSO ₄			X	X
Magnesite	MgCO ₃	X	X		
Magnesium Sulphate	MgSO ₄	X		X	
Dolomite	CaCO ₃ ·MgCO ₃				X
Gaylussite	Na ₂ Ca(CO ₃) ₂ ·5H ₂ O				
Tychite	2MgCO ₃ ·2Na ₂ CO ₃ ·Na ₂ SO ₄	X			
Thenardite	Na ₂ SO ₄		X		X
Burkeite	Na ₂ CO ₃ ·2Na ₂ SO ₄	X	X	X	X
Hanksite	9Na ₂ SO ₄ ·2Na ₂ CO ₃ ·KCl				X
Trona	Na ₂ CO ₃ ·NaHCO ₃ ·2H ₂ O				X
Nahcolite	NaHCO ₃				
Thermonatrite	Na ₂ CO ₃ ·H ₂ O		X	X	
Natron	Na ₂ CO ₃ ·10H ₂ O				
Natrite	Na ₂ CO ₃				X
Halite	NaCl	X	X	X	X
Nitratine	NaNO ₃	X	X	X	
Glauberite	Na ₂ SO ₄ ·CaSO ₄				
Glaserite	K ₃ Na(SO ₄) ₂	X		X	X
Arcanite	K ₂ SO ₄				
Sylvite	KCl			X	X

Table 3. Mineralogical composition of the precipitates at 40°C

Mineral Phase	40°C				
	Chemical Formula	Time Interval			
		3 Hrs	4.4 Hrs	5.5 Hrs	6 Hrs
Calcite	CaCO ₃	X	X		
Anhydrite	CaSO ₄				
Magnesite	MgCO ₃	X	X	X	
Magnesium Sulphate	MgSO ₄	X			
Dolomite	CaCO ₃ ·MgCO ₃			X	X
Gaylussite	Na ₂ Ca(CO ₃) ₂ ·5H ₂ O				
Tychite	2MgCO ₃ ·2Na ₂ CO ₃ ·Na ₂ SO ₄				
Thenardite	Na ₂ SO ₄	X		X	
Burkeite	Na ₂ CO ₃ ·2Na ₂ SO ₄			X	X
Hanksite	9Na ₂ SO ₄ ·2Na ₂ CO ₃ ·KCl				X
Trona	Na ₂ CO ₃ ·NaHCO ₃ ·2H ₂ O	X			
Nahcolite	NaHCO ₃				
Thermonatrite	Na ₂ CO ₃ ·H ₂ O			X	
Natron	Na ₂ CO ₃ ·10H ₂ O	X			
Natrite	Na ₂ CO ₃				
Halite	NaCl	X	X	X	X
Nitratine	NaNO ₃				
Glauberite	Na ₂ SO ₄ ·CaSO ₄			X	X
Glaserite	K ₃ Na(SO ₄) ₂	X		X	
Arcanite	K ₂ SO ₄	X			
Sylvite	KCl	X		X	X

When the temperature is increased to 60°C, glaserite does not saturate with the solution and therefore does not precipitate (Fig. 6).

Further increase in temperature to 70°C, results into the precipitation of only thenardite and halite (Fig. 7). At all the study

temperatures, thenardite precipitates first and later with increasing evaporation dissolves into the solution. The Saturation Index (SI) of soda ash, a mineral of interest in this study,

increases with increasing evaporation temperature (Figs. 3-7). This observation predicts precipitation of this mineral at temperatures higher than 70°C.

Table 4. Mineralogical composition of the precipitates at 50°C

Mineral Phase	50°C				
	Chemical Formula	Time Interval			
		1.5 Hrs	3 Hrs	3.5 Hrs	4 Hrs
Calcite	CaCO ₃		X	X	X
Anhydrite	CaSO ₄	X			X
Magnesite	MgCO ₃	X	X	X	
Magnesium Sulphate	MgSO ₄				
Dolomite	CaCO ₃ ·MgCO ₃	X		X	X
Gaylussite	Na ₂ Ca(CO ₃) ₂ ·5H ₂ O			X	
Tychite	2MgCO ₃ ·2Na ₂ CO ₃ ·Na ₂ SO ₄				
Thenardite	Na ₂ SO ₄		X		X
Burkeite	Na ₂ CO ₃ ·2Na ₂ SO ₄			X	X
Hanksite	9Na ₂ SO ₄ ·2Na ₂ CO ₃ ·KCl			X	X
Trona	Na ₂ CO ₃ ·NaHCO ₃ ·2H ₂ O	X	X	X	
Nahcolite	NaHCO ₃				X
Thermonatrite	Na ₂ CO ₃ ·H ₂ O	X		X	X
Natron	Na ₂ CO ₃ ·10H ₂ O				
Natrite	Na ₂ CO ₃			X	
Halite	NaCl	X	X	X	X
Nitratine	NaNO ₃	X	X	X	
Glauberite	Na ₂ SO ₄ ·CaSO ₄			X	
Glaserite	K ₃ Na(SO ₄) ₂				X
Arcanite	K ₂ SO ₄				
Sylvite	KCl		X		X

Table 5. Mineralogical composition of the precipitates at 60°C

Mineral Phase	60°C				
	Chemical Formula	Time Interval			
		30 Min	1 Hr	1.5 Hrs	2 Hrs
Calcite	CaCO ₃	X		X	
Anhydrite	CaSO ₄				X
Magnesite	MgCO ₃	X	X	X	
Magnesium Sulphate	MgSO ₄				
Dolomite	CaCO ₃ ·MgCO ₃		X	X	X
Gaylussite	Na ₂ Ca(CO ₃) ₂ ·5H ₂ O				
Tychite	2MgCO ₃ ·2Na ₂ CO ₃ ·Na ₂ SO ₄				
Thenardite	Na ₂ SO ₄				
Burkeite	Na ₂ CO ₃ ·2Na ₂ SO ₄				X
Hanksite	9Na ₂ SO ₄ ·2Na ₂ CO ₃ ·KCl				X
Trona	Na ₂ CO ₃ ·NaHCO ₃ ·2H ₂ O			X	
Nahcolite	NaHCO ₃				
Thermonatrite	Na ₂ CO ₃ ·H ₂ O				
Natron	Na ₂ CO ₃ ·10H ₂ O				X
Natrite	Na ₂ CO ₃			X	
Halite	NaCl	X	X		X
Nitratine	NaNO ₃	X	X		
Glauberite	Na ₂ SO ₄ ·CaSO ₄				
Glaserite	K ₃ Na(SO ₄) ₂				
Arcanite	K ₂ SO ₄			X	
Sylvite	KCl	X		X	X

Table 6. Mineralogical composition of the precipitates at 70°C

Mineral Phase	Chemical Formula	70°C			
		Time Interval			
		11 Min	45 Min	60 Min	68 Min
Calcite	CaCO ₃	X	X	X	
Anhydrite	CaSO ₄			X	X
Magnesite	MgCO ₃	X	X	X	X
Magnesium Sulphate	MgSO ₄	X			
Dolomite	CaCO ₃ ·MgCO ₃	X	X	X	X
Gaylussite	Na ₂ Ca(CO ₃) ₂ ·5H ₂ O				X
Tychite	2MgCO ₃ ·2Na ₂ CO ₃ ·Na ₂ SO ₄				
Thenardite	Na ₂ SO ₄	X			X
Burkeite	Na ₂ CO ₃ ·2Na ₂ SO ₄				
Hanksite	9Na ₂ SO ₄ ·2Na ₂ CO ₃ ·KCl				
Trona	Na ₂ CO ₃ ·NaHCO ₃ ·2H ₂ O		X		
Nahcolite	NaHCO ₃				
Thermonatrite	Na ₂ CO ₃ ·H ₂ O	X			
Natron	Na ₂ CO ₃ ·10H ₂ O				
Natrite	Na ₂ CO ₃				X
Halite	NaCl	X	X	X	X
Nitratine	NaNO ₃	X		X	
Glauberite	Na ₂ SO ₄ ·CaSO ₄				X
Glaserite	K ₃ Na(SO ₄) ₂	X			
Arcanite	K ₂ SO ₄				
Sylvite	KCl	X	X	X	X

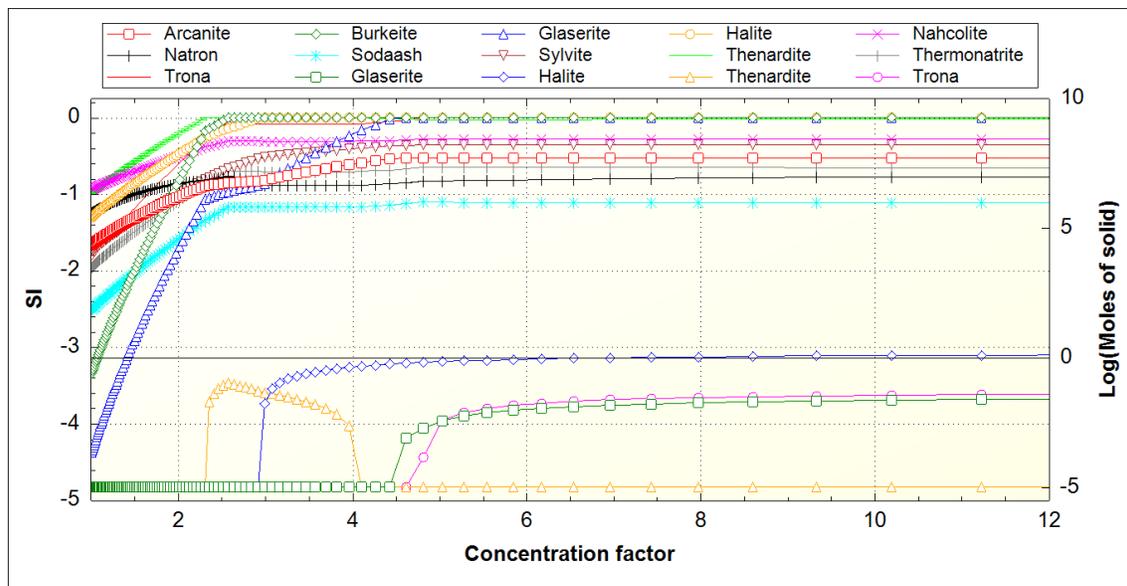


Fig. 3. Saturation Indices and moles of solid posted during evaporation at 30°C

The concentration factor at which both thenardite and trona precipitate reduces with increasing temperature whereas for halite and glaserite, the concentration factor at which they precipitate out of the solution increases with increasing temperature. The model further predicts that for

the same concentration factors, the amount of precipitate for thenardite and trona increases with increasing temperature whereas for both halite and glaserite, the amount reduces with increasing temperature. These trends are due to the solubility of halite slightly increasing with

increasing temperature whereas that of thenardite reduces with increasing temperature within the study temperature range.

From the model results as seen in Figs. 3-7, halite posted the highest amount of precipitate, followed by thenardite, trona and lastly glaserite. This is consistent with previous studies [4,20,22] which ascertained that, at the salt lake, brine are

comprised of more sodium, chloride, Carbonate, sulphate, bicarbonate, & potassium salts. From Figs. 3-7, as temperature increases, the precipitation sequence becomes more distinct and hence reducing the chances of co-precipitation. This means that increase in temperature moves the phase boundaries and thus avoids a large number of salts being present in a small region of the phase diagram.

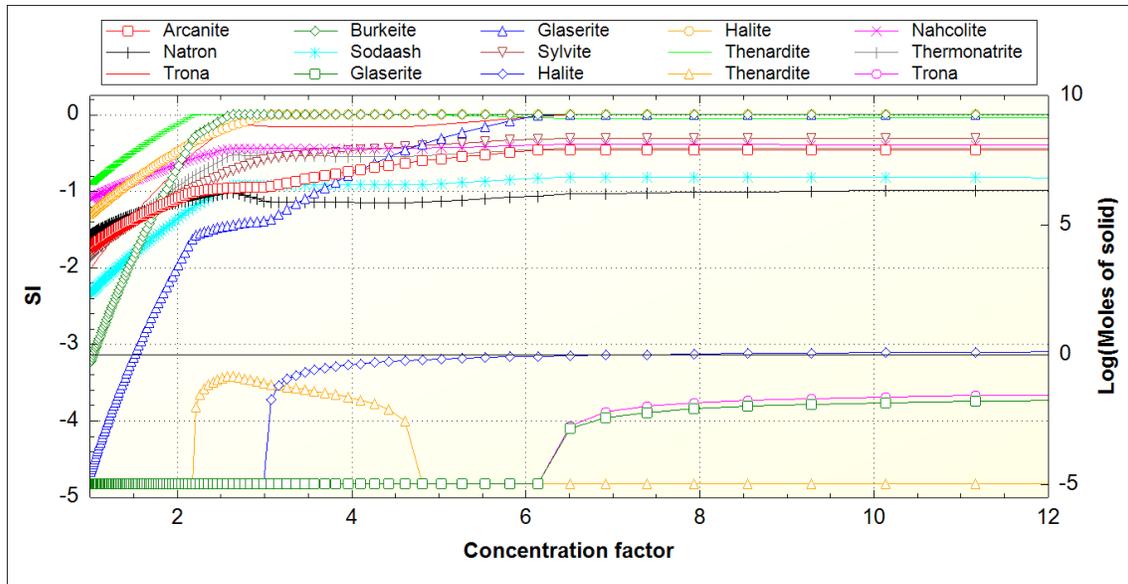


Fig. 4. Saturation indices and moles of solid posted during evaporation at 40°C

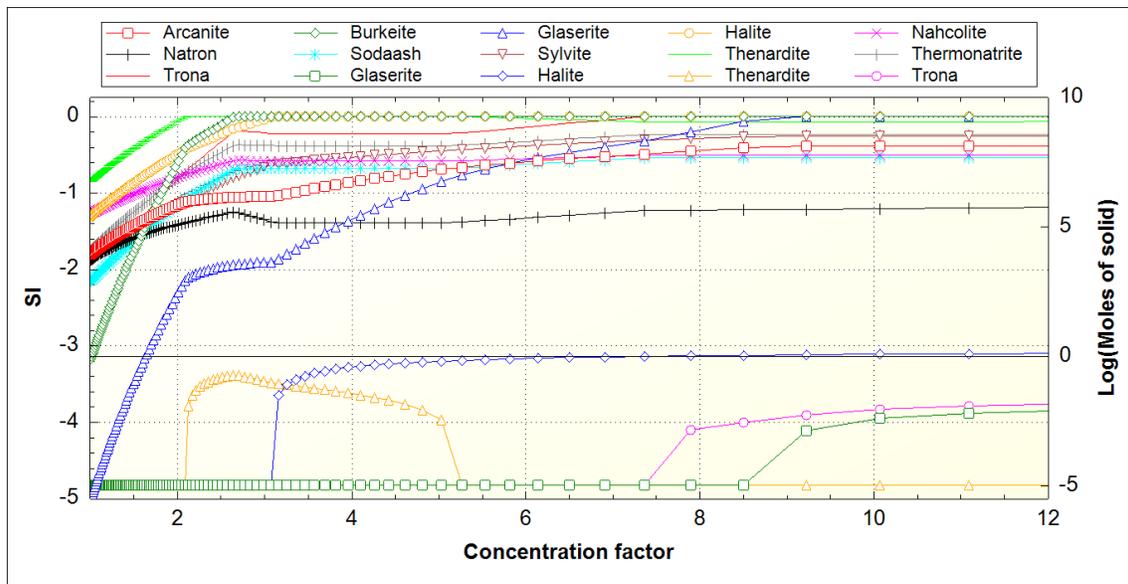


Fig. 5. Saturation indices and moles of solid posted during evaporation at 50°C

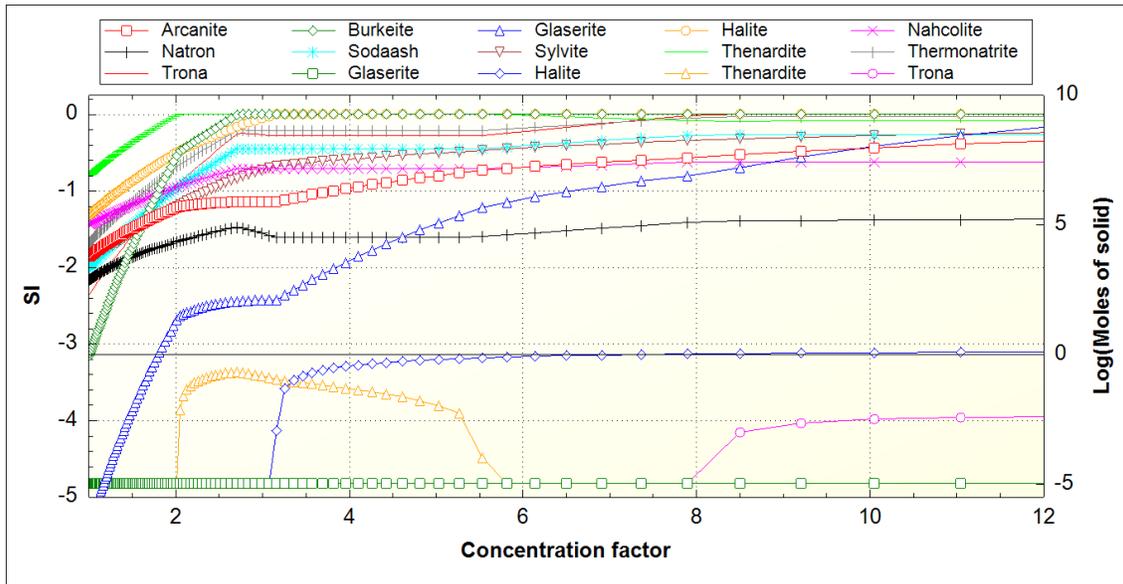


Fig. 6. Saturation Indices and Moles of solid posted during evaporation at 60°C

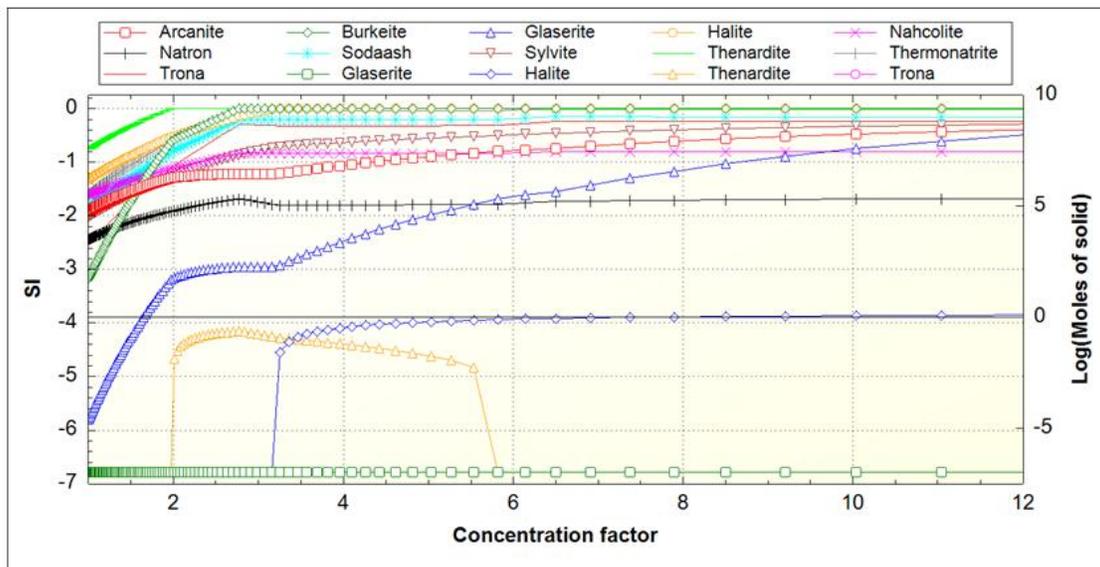


Fig. 7. Saturation Indices and Moles of solid posted during evaporation at 70°C

There are noticeable differences between the actual precipitated salts from the evaporation experiment and the PHREEQC results. The XRD and SEM results confirmed existence of more double salts and hydrates compared to the PHREEQC model. This variation is due to the fact that the PHREEQC model mainly considers the thermodynamic aspects and not the kinetics, yet, the actual setting considers the

kinetics thus some minerals might precipitate from the model and not observed by XRD and vice versa.

4. CONCLUSION

In this work, the effect of temperature on the precipitation and crystallization sequence on the Lake Katwe brine during evaporation was investigated. Increase in temperature fostered

the evaporation rate and therefore it took less time to precipitate minerals at higher temperatures than at lower ones.

XRD and SEM results confirmed that trona was the dominant mineral which is not consistent with the PHREEQC model results. The model does not post hanksite as a precipitate which could reduce on the amount of the halite precipitating. The variation in temperature, did not have an effect on the precipitation sequence, the sequence consistently remained sulphates, chlorides and lastly carbonates. Significant coprecipitation/crystallization overlap was observed, reducing with increasing temperature; dominant minerals were doped by other minerals in lower concentrations.

Higher temperatures in the system favour natrite precipitation and slightly halite precipitation and lower temperatures favour thenardite and burkeite precipitation. This is consistent with the theory that predicts thenardite being more soluble at lower temperatures whereas the solubility of halite is almost constant within the study temperature range.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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