

## Mineral recovery from Lake Katwe brines using isothermal evaporation

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**Abstract** Lake Katwe is a saline lake within the East African Rift system in Western Uganda, with a rich source of mineral salts. The present work aims at evaluating possibilities of future salt extraction from the lake deposit. An isothermal evaporation experiment was conducted on the lake brines. The precipitated salts were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) methods. Various economic salts such as thenardite, gypsum, mirabilite, burkeite, hanksite, anhydrite, trona, halite, nahcolite, thermonatrite, and soda ash precipitate from the lake brines. The experiments also reveal the sequence of mineral salt precipitation in the order sulfates → chlorides → carbonates.

**Keywords** Lake Katwe; Brine; Isothermal Evaporation; Sulfate salts; Chloride salts; Carbonate salts; XRD; SEM.

### Introduction

Lake Katwe is a closed saline lake on the northern side of fresh water Lake Edward within the western branch of the East African Rift valley system, and about 15 km below the equator in western Uganda. The lake lies at an elevation of about 885 m, with a maximum area of 2.5 km<sup>2</sup>, depth of less than 1.5 m, and measuring 9 km in circumference. The natural salt lake brines are highly alkaline and rich in Na<sup>+</sup>, K<sup>+</sup>, Cl<sup>-</sup>, CO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup>, and HCO<sub>3</sub><sup>-</sup> with lesser amounts of Mg<sup>2+</sup>, Ca<sup>2+</sup>, Br<sup>-</sup>, and F<sup>-</sup>. The surface brines are hydro-chemically of a carbonate type and represent an important source of mineral salts that are of great economic value. The salinity and density of the lake brines varies from 140 to 150 g/L and 1.15 to 1.23 g/mL respectively and depend on seasonal variations (Kasedde *et al.* 2012 submitted). The meteorological conditions in this region are generally semi-arid, with little rainfall, and a great capacity for evaporation. Because of these conditions, traditional solar pond evaporation techniques are currently used to extract salts

from the surface brine resources of the lake. These salt extracts are composed of halite mixed with other impurities.

Since the 1960s, studies have been developed to exploit and utilize the lake's mineral resources. Besides the study of the geological setting of the region and chemistry of the lake brines (Arad and Morton 1969), investigations were extended to studying the feasibility of salt extraction through estimation of the salt reserves (Morton and Old 1968, Dixon and Morton 1970, Morton 1973, UDC 1997), and the mineralogical composition of the evaporites (Nielsen 1999). Further studies involved devising techniques and concepts of improving salt mining and extraction from the lake resources (Kirabira *et al.* 2013) and characterization of the mineral salt raw materials from the salt lake deposit (Kasedde *et al.* 2012 submitted).

In the present investigation, an isothermal evaporation experiment was performed to determine the nature of mineral salts that can be recovered from Lake Katwe brines, and to study the sequence of their precipitation.

The study is essential for evaluating the possibilities for future comprehensive and sustainable utilization of the salt-lake brine resources.

## Materials and methods

### Apparatus and reagents

An FP12 thermostatic water bath (Julabo Labortechnik, GmbH, Seelback, Germany) was used for the isothermal evaporation experiment. The precision of temperature control was  $\pm 1$  °C. The density of the liquid phase was measured by a portable densito-meter (DMA 35 Anton Paar, Graz Österreich, Austria) with an accuracy of  $\pm 0.001$  g/cm<sup>3</sup>. The salinity and electrical conductivity were measured by an electrode probe meter (HANNA instruments HI 98360, Woonsocket, RI, USA) with an accuracy of  $\pm 0.5$  %. The pH value of the liquid phase was determined by a PC Titrator (Mantech). The mineralogy of the precipitated salts at each stage of evaporation was identified by X-ray diffractometry (XRD) using a D2 Phaser benchtop XRD system (Bruker Corporation, Massachusetts, USA) with a copper K $\alpha$  radiation ( $\lambda = 1.5405$  Å) operating at a voltage and current power of 30 kV and 10 mA. A diffraction interval between  $2\theta$ -10°-80° with step increments of 0.01° and a scan speed of 0.5 seconds were used. The morphology of the salts was examined by a Field Emission Gun Scanning Electron Microscope (FEG-SEM) using a LEO 1530 Gemini (Zeiss, Oberkochen, Germany) with settings at a voltage of 15 kV and aperture 60  $\mu$ m. The images were taken with a secondary electron detector. A gold sputter coater (Emitech K550) was used to prepare the mineral salt samples before SEM analysis to make them electrically conductive. Double distilled water having a conductivity of 0.0182 S/m at 25 °C was used in the experiment.

### Experimental methods

The dry season surface brine was sampled from Nambawu salt pans at Lake Katwe in August 2012 and was stored in plastic bottles at room temperature prior to the present study

in October 2012. The brine had not been saturated since no solid precipitates were observed. For the isothermal evaporation experiment, one liter of the natural brine was filled in a glass beaker. The beaker was placed in a thermostatic water bath which was maintained at  $30 \pm 1$  °C. The evaporation conditions were close to those existing at Lake Katwe. The brine was left to evaporate without stirring in a ventilated environment at  $30 \pm 1$  °C with a continuous air flow of 1 m/s. The evolution of the brine evaporation was monitored on a daily basis for newly precipitated solid salts. When a sufficient amount of the solid salts appeared, they were separated from the solution by filtration. The salts were then dried and stored in small plastic sample bags and subsequently characterized with X-ray diffraction and scanning electron microscope techniques. At the same time, a 10 mL brine sample was taken from the liquid phase, diluted with distilled water to a final volume of 50 mL, then measurement of its physical properties were taken. The isothermal evaporation experiment was repeated for each sample until all the brine dried up.

## Results and Discussion

The brine sample used in the evaporation experiment was the original brine from Lake Katwe. Its chemical composition in g/L was 137 Na<sup>+</sup>, 39.1 K<sup>+</sup>, 0.00143 Mg<sup>2+</sup>, 0.005 Ca<sup>2+</sup>, 124 Cl<sup>-</sup>, 43 SO<sub>4</sub><sup>2-</sup>, 3.39 HCO<sub>3</sub><sup>-</sup>, 61 CO<sub>3</sub><sup>2-</sup>, and 0.082 F<sup>-</sup>. In the course of the isothermal evaporation experiment, twelve liquid and twelve solid samples were collected. The physico-chemical parameters of the original brine (sample L<sub>0</sub>) and the mother liquors (sample L<sub>n</sub>, n representing the corresponding evaporation stage) are presented in Table 1.

The evolution of density and conductivity as given in Table 1 is shown graphically in Fig. 1. The original brine (sample L<sub>0</sub>) is undersaturated and hence, upon evaporation of water from the original brine, both density and conductivity increase until the first salt precipitate is harvested at sample L<sub>1</sub>. Thereafter, density

Sample	Evaporation Time(days)	Electrical Conductivity (mS/cm)	pH	Salinity (g/L)	Density (g/cm <sup>3</sup> )
L <sub>0</sub>	0	140.1	10.2	70.0	1.306
L <sub>1</sub>	6	147.3	10.7	73.8	1.315
L <sub>2</sub>	12	138.3	10.7	69.2	1.316
L <sub>3</sub>	15	133.5	10.7	66.6	1.322
L <sub>4</sub>	20	132.3	10.7	66.1	1.326
L <sub>5</sub>	25	134.5	10.8	67.4	1.320
L <sub>6</sub>	29	130.5	10.9	65.3	1.325
L <sub>7</sub>	33	123.6	10.9	62.1	1.336
L <sub>8</sub>	36	116.6	10.8	58.4	1.343
L <sub>9</sub>	40	99.6	11.1	49.8	1.360
L <sub>10</sub>	43	95.2	11.1	47.4	1.361
L <sub>11</sub>	47	93.0	11.2	44.0	1.355
L <sub>12</sub>	50	-	-	-	-

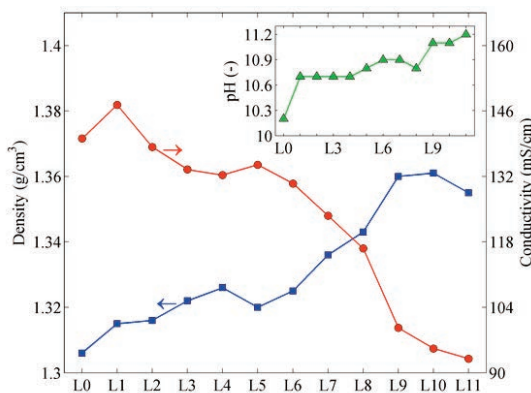
**Table 1** Physico-chemical parameters of the liquid brine samples.

and conductivity follow a different trend: while density tends to increase, conductivity gradually decreases. This indicates a change in the ionic composition of the brine as different ionic species contribute differently to density and conductivity. Furthermore, the local maxima of density, respectively the local minima of conductivity, observed for sample L<sub>4</sub> indicates a change in the precipitation sequence, as confirmed by analysis of the precipitates as outlined below.

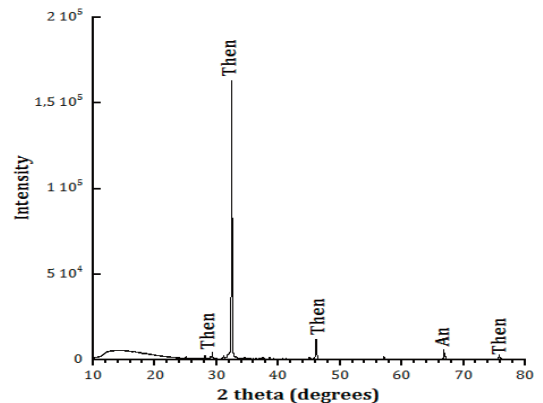
The inset in Fig. 1 shows the evolution of pH during evaporation. After a sharp increase when moving from the original brine to the first sample, the pH assumes a constant value that lasts until sample L<sub>4</sub> after which a further

increase is observed. This second period of increase culminates in a local maximum at sample L<sub>6</sub> and L<sub>7</sub>, followed by a local minimum at sample L<sub>8</sub>. Thereafter, a third increase is observed leading to plateau at sample L<sub>9</sub> and L<sub>10</sub>. Likewise to the local extrema in density and conductivity, the different regions in the evolution of pH relate to the precipitation sequence.

The mineralogical composition of the recovered solids were analyzed by XRD and SEM. These measurements indicate the presence of various solid phases in each sample. A typical XRD measurement showing several characteristic peaks is shown in Fig. 2. Analyzing the XRD spectra allowed for identifying the solid



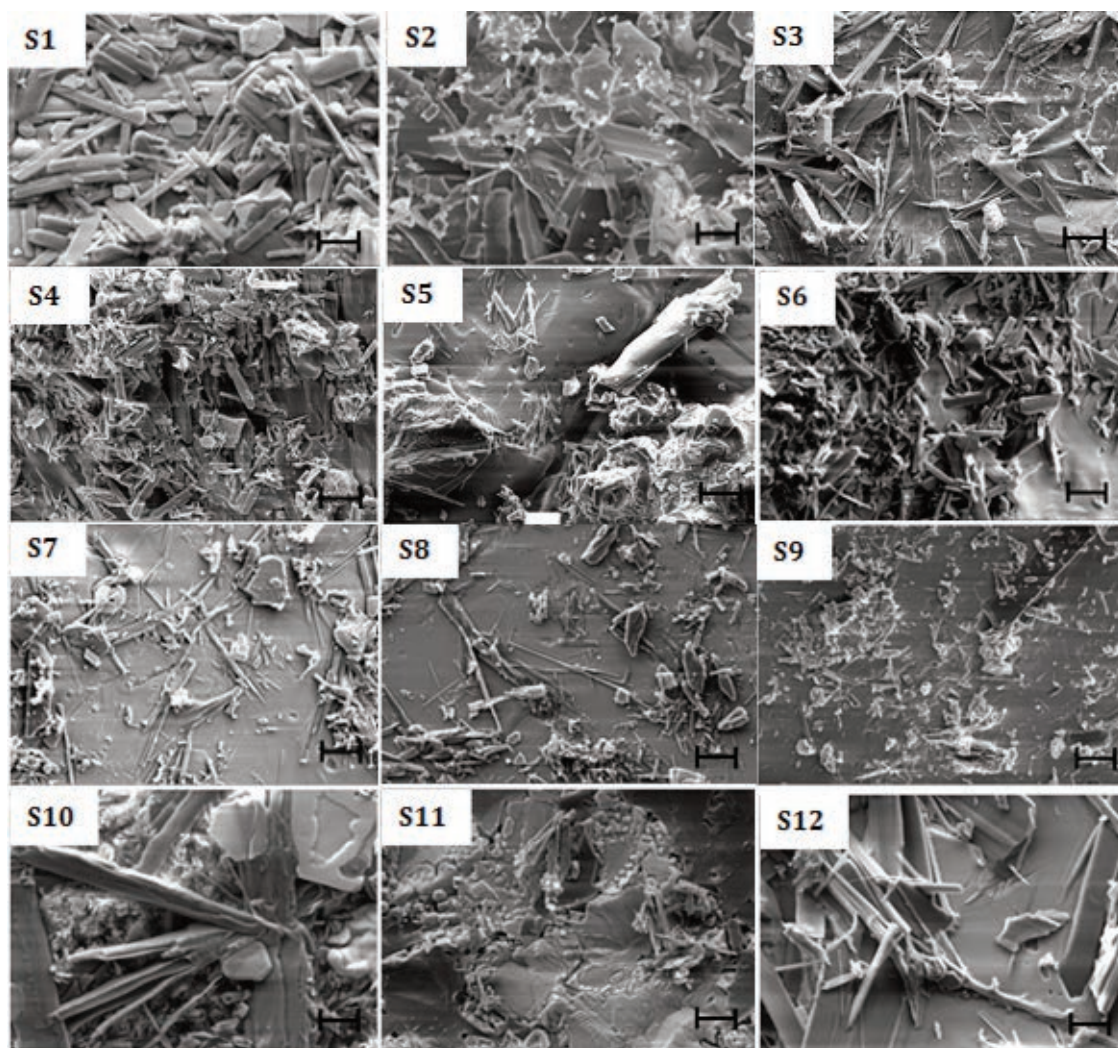
**Fig. 1** Evolution of brine density (square symbols), conductivity (circles), and pH (inset).



**Fig. 2** X-ray diffraction (XRD) results for salt sample S1 (Then-Thenardite, An-Anhydrite)







**Fig. 3** SEM micrographs of the recovered salts ( $S_1$ - $S_{12}$ ) during the isothermal evaporation experiment. Scale bars = 10  $\mu$ m.

large indistinguishable crystals which we relate to nahcolite and trona, respectively.

### Conclusion

From the isothermal evaporation experiment of Lake Katwe brine, the following conclusions are drawn:

Precipitates show a rich variety of different mineral salts, *i.e.* upon evaporation of lake brines thenardite, anhydrite, mirabilite, burkeite, hanksite, gypsum, trona, halite, nahcolite, soda ash, and thermonatrite are formed. Thenardite precipitates during the entire evap-

oration process and its crystals were observed to be present in all samples.

The mineral salts precipitating from the lake brines follow the sequence sulfates  $\rightarrow$  chlorides  $\rightarrow$  carbonates. The evaporation pathway of Lake Katwe brines thus differs from that of modern sea water evaporation sequence.

The salts produced at Lake Katwe by traditional and artisanal techniques are composed of several mineral phases with limited production rates. The results from the present study can provide an important reference in the development of technologies for the extraction

of various pure mineral salts from the natural brines of Lake Katwe. Understanding the sequence of salt precipitation from the brine helps to control its evolution during concentration and hence will lead to an improved operating design scheme of the current extraction process. However, to fully exploit the lake's brine resource, further work is required in studying its thermodynamics and the related phase equilibria.

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