

## Preparation of Various Salts from Deposits of Egypt

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In the present study an investigation of the mineral constituents of evaporates formed in the south- west part of western desert in Egypt has been made. The evaporate samples were analyzed by different techniques. These techniques include chemical analyses, X-ray diffraction patterns and atomic absorption spectra. Simple and application methods were suggested for the preparation of magnesium, aluminium, sodium and potassium salts from these evaporates.

**Key Words: Preparation, Salts, Evaporites, Egypt.**

### INTRODUCTION

Several geological investigations have been carried out on the different deposits spreaded all over several parts in the western desert of Egypt. The present studies are concerned with exploration and characterization of deposits located at El-Baharyia Oasis<sup>1</sup>, Wadi Natroun<sup>2</sup>, Dir El-Biraqat<sup>3</sup>, Qarun lake brine<sup>4</sup>, Qattara Depression<sup>5</sup> and Bir El-Shab<sup>6</sup>.

The present investigation was carried out in order to throw the light on the exploitation of the deposits found in the area located between Bir El-Shab and Bir Misaha<sup>6</sup> for production of different salts of aluminium, magnesium, sodium and potassium, following simple and applicable methods that can be used in future in a large scale.

### EXPERIMENTAL

The chemicals used in the present investigation were of AnalR grade and obtained from Merck Company (Darmstadt, Germany).

The salt samples were supplied by the Mineral Resources Authority of Egypt. Representative samples, 100 kg each, were collected from the area located between Bir El-Shab and Bir Misaha (340 km south-southwest of Kharga and 370 km southwest of Aswan). The samples were homogenized by crushing to a particle size of 0.1 mm using a jaw crusher mill.

**Preparation of water extract solution:** The samples under investigation were digested twice in 100 mL of distilled water while boiling for 0.5 h for dissolution of soluble salts, followed by filtration to remove the

insoluble product. The summation of the two filtrates was completed to 500 mL, in a standard measuring flask. Suitable aliquots of combined filtrate were utilized for determination of water soluble species of iron, aluminium, calcium and magnesium according to the methods mentioned elsewhere. The percentage of extraction of different oxides was calculated by dividing the extracted weight by their theoretical amounts found in the evaporate deposit.

The contents of iron oxide, aluminium oxide, calcium oxide and magnesium oxide were determined according to the methods described elsewhere<sup>7,8</sup>.

The X-ray diffraction patterns (XRD) of evaporate deposits were carried out using a Philips diffractometer (type PW 1051) employing Ni-filtered  $\text{CuK}\alpha$  radiation. The X-ray tube was operated at 36 KV and 16 mA. The diffraction angle was scanned at a rate of  $2\theta$  per min. The pH of the aqueous phases was measured with digital pH meter model 7065 (Electronic measurement Ltd., Kent UK). The contents of sodium and potassium species in the evaporate ore was determined using a Varian spectrometer 220.

## RESULTS AND DISCUSSION

The XRD analysis of the evaporate deposits located at the studied zone showed that the crystalline constituents of the deposits are composed of,  $\text{CaCl}_2$  (I),  $\text{CaSO}_3$  (II),  $\text{CaSO}_4$  (III),  $\text{Fe}_2\text{O}_3$  (IV),  $\text{FeSO}_4$  (V),  $\text{KCaCl}_3$  (VI),  $\text{K}_2\text{Ca}_5(\text{SO}_4)_6 \cdot \text{H}_2\text{O}$  (VII),  $\text{KCl}$  (VIII),  $\text{KMgCl}_3 \cdot 6\text{H}_2\text{O}$  (IX),  $\text{MgSO}_3 \cdot 6\text{H}_2\text{O}$  (X),  $\text{MgSO}_4$  (XI),  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (XII),  $\text{NaAl}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  (XII),  $\text{NaAl}(\text{SO}_4)(\text{OH})_2$  (XIII),  $\text{NaAl}_3(\text{SO}_4)_2(\text{OH})_6$  (XIV),  $\text{Na}_8(\text{Al}_6\text{Si}_6\text{O}_{24})(\text{SO}_4) \cdot 3\text{H}_2\text{O}$  (XV),  $\text{NaCl}$  (XVI),  $\text{Na}_2\text{Mg}(\text{SO}_4)_2 \cdot 5\text{H}_2\text{O}$  (XVII),  $\text{Na}_2\text{SO}_3$  (XVIII) and  $\text{Na}_2\text{SO}_4$  (XIX).

The previous compounds may be formed as a result of interaction between soil constituents and underground water, which may resemble the sea water. This means that the interaction may be taken place between kaolin group  $[\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4]$ , montmorillonite group  $(0.5\text{Ca},\text{Na})_{0.067}(\text{Al}_{3.33}\text{Mg}_{0.67})\text{Si}_8\text{O}_{20}(\text{OH})_4 \cdot n\text{H}_2\text{O}$ , illite group (Hydromicas)  $(\text{K}_{1-1.5}\text{Al}_4(\text{Si}_{7-6.5}\text{Al}_{1-1.5})\text{O}_{20}(\text{OH})_4)^9$  and calcium carbonate and constituent of underground water which composed of  $\text{KCl}$ ,  $\text{MgCl}_2$ ,  $\text{NaCl}$ ,  $\text{MgSO}_4$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{K}_2\text{CO}_3$  and  $\text{CaCO}_3$ <sup>10</sup>. The interaction process may lead to formation of  $\text{Na}_2\text{Al}_2\text{O}_4$  and  $\text{Na}_4\text{SiO}_4$ . The last two species interact with each other *in situ* leading to formation of vishnevite  $(\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}) \cdot 3\text{H}_2\text{O}$ <sup>11</sup>.

The formation of both  $\text{Na}_2\text{Al}_2\text{O}_4$  and  $\text{Na}_4\text{SiO}_4$  may be ascribed to the probable interaction of  $\text{Na}_2\text{CO}_3$  (formed as a result of interaction of the last compound and  $\text{MgCl}_2$ ) with the silicious compounds formed the soil.

The presence of sodium and calcium sulfites may be ascribed to formation sulfur dioxide, which interacts with the carbonates of calcium

and sodium. Sulfur dioxide could be generated in case of reaction of some sulfide compounds with oxygen in presence of alkaline solution.

The double salts such as  $\text{Na}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 5\text{H}_2\text{O}$  and  $\text{KCl} \cdot \text{MgCl}_2$  may be formed during the process of evaporation of salty water, which contains their ingredients, since these salts are found in the underground water.

The presence of iron oxide  $\text{Fe}_2\text{O}_3$  (IV) gives an indication that the pH of the resulted salt solution was higher than 3.0 and lower than 4.8 which is considered as the suitable pH for precipitation of  $\text{Al}^{3+}$  ions. This hypothesis was confirmed from the presence of hydroxysulfate species of soda alum (XIII) and (XIV) and absence of aluminium oxide and/or hydroxide. This hypothesis was also confirmed from the experimental finding which indicates that the percentage of extraction of aluminium from the deposits does not increase in case of extraction with sulfuric or hydrochloric acids instead of water.

**Chemical analysis of water soluble salts:** The chemical analysis of water soluble and dried solid evaporites (Table-1) showed the production of that the corresponding salts which find various industrial and pharmaceutical applications.

TABLE-1  
CHEMICAL ANALYSIS OF EVAPORITE ORE IN WATER AND  $\text{H}_2\text{SO}_4$

Oxides	$\text{H}_2\text{O}$ (%)	$\text{H}_2\text{SO}_4$ (%)
$\text{Al}_2\text{O}_3$	3.7	3.80
$\text{MgO}$	7.5	7.90
$\text{CaO}$	3.3	3.60
$\text{Fe}_2\text{O}_3$	0.6	0.63
$\text{K}_2\text{O}$	1.4	1.40
$\text{Na}_2\text{O}$	9.5	9.50

#### Preparation of different salts

**1st step:** The pH value of the aqueous extract solution of the evaporite was raised gradually with NaOH to reach the values 3.0, 5.0, 8.5, 10.5 to precipitate the hydroxides of  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Mg}^{2+}$ , respectively. The resulted hydroxides can be changed to the corresponding salts by reaction with the desired acid such as sulfuric acid.

**2nd step:** The resulted basic filtrate was neutralized with sulfuric acid to produce an additional amount of sodium sulfate to that already present in the evaporite. The resulted filtrate which contain NaCl, KCl,  $\text{Na}_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$  can be used as a source of production of previous salts.

#### Separation of the chlorides and sulfates of sodium and potassium

These salts can be separated according to several steps, the first of them is concerned with the separation of sulfates of potassium and sodium

from the corresponding chlorides. The method of separation can be verified by following the method reported by Bassett<sup>12</sup> in the form of a patent. According to this method both sulfates were separated after addition of sodium chloride to the solution followed by concentration and separation of both salts. The resulted salts were recrystallized from water to ensure the formation of dehydrated sodium sulfate. The mixture of both sulfates was then heated at 35°C for melting of hydrated sodium sulfate followed by its separation of the corresponding potassium salt.

The second step of separation was focused on separation of KCl of NaCl. This separation can be verified by the method reported by Assarsson<sup>13</sup> in the form of a patent. The process depends on the addition of solid calcium chloride and formation of double salt composed CaCl<sub>2</sub>·KCl, which precipitated by heating above 37°C, while the liquor contains NaCl alone. The precipitate of the double salt was then stirred with a dilute solution of the double salt, below 37°C, where upon KCl can be separated as a solid.

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