**Utilization of Uzbekistan’s Saponite Mineral for Local Production of Magnesium Chloride and Magnesium Chlorate**

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**Abstract.** Chemical and modern physicochemical analysis methods have revealed the presence of saponite mineral within dolomitic mineral at the vaush deposit, which is situated in the Navbahor district of the Navoi region of Uzbekistan by scientists from Navoi State Mining and Technology University. The chemical and mineralogical content of this mineral was studied. Research results indicated that saponite mineral included an average of 20-23% oxides of calcium and magnesium. The process of treating this saponite mineral with hydrochloric acid for extracting magnesium chloride solution through calcination using calcium chloride salt was presented, as well as the conversion of magnesium chloride into magnesium chlorate.

**Keywords:** sodium hypochlorite, saponite, mineral, magnesium chlorate, calcium chloride, defoliant, caustic soda.

**INTRODUCTION**

Saponite was first determined in Khmelnitskyi Oblast of Ukraine, which has been defined as the most rich saponite deposit in the world. Saponite, or "wash stone", is a silicate, and is classified as part of the montmorillonite group of minerals. Saponite is a light red to dark colored, odorless and tasteless substance. Its formula is (Ca, Na)0.3(Mg, Fe2)3(Si, Al)4O10(OH)2\*4·H2O. Saponite can contain Fe and Cr substitutions for Mg, and may include nikel, zinc, copper, lithium, and other metals [1–3]. Today this mineral with magnesium is utilized in the these areas: treatment of water and industrial wastes; as an absorbant for radioactive materials; as a highly absorbent, catalytic and filter medium; as a dietary additive for livestock; as a micro-elemental fertilizer for agriculture; in the metal casting industry; and as a medicinal agent in medicine and pharmacology. In Uzbekistan there are numerous studies being done today on the saponite mineral [4]. The Kyzylkum oasis of the Republic of Uzbekistan has an abundance of local resources and is one of the countries with the largest diversity of chemical elements. The deposits present in this area significantly to promote the national economy and chemical industry of our Republic. Researchers of the Navoi State Mining and Technology University have determined that saponite mineral is present in dolomite mineral at Vaush deposit, situated in the Navbakhor place of the Navoi region. Therefore, the purpose of this article is to describe the scientific research done on saponite mineral. The article will report on the results received during the recovery of magnesium chloride solutions through interaction of the saponite mineral with magnesium oxide and HCl , and the results of its calcination with calcium chloride. We all know that primary source of magnesium chloride in our Republic is importation of it from Turkmenistan and China using the Republic's foreign currency. This magnesium chloride is synthesized to magnesium chlorate for use as defoliant to strip leaves from cotton cultivated in our country, resulting in a high price for defoliant.

**METHODS**

Using a Retsch RM-200 mobile analytical grinder, several samples of the saponite mineral that had been crushed were then used for experimentation. Using a mechanical laboratory separation tool (model AS-200 BASIC) with a variety of mesh sizes (from 0.01 to 4.0 mm), the crushed saponite mineral was separated into the various samples. These samples were further analyzed by means of a number of different techniques including; flame photometry on a Model 410 device, SEM-EDS analysis, X-ray phase analysis, infrared (IR) spectroscopy on an IRTRACER-100 SHIMADZU FTIR spectrophotometer, thermogravimetry (TGA) using a modern LABSYS EVOS device, X-ray fluorescence, and microscopy.

Prior to this analysis, the chemical content of the saponite mineral was first identified, with the results presented in Table 1.

**TABLE 1.** Chemical Content of Saponite Mineral from Vaush Field

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **The saponite mineral** | **Elemental composition of the mineral saponite, %** | | | | | | | | | | |
| **CaO** | **MgO** | **SiO2** | **Fe2O3** | **MnO** | **Al2O3** | **CuO** | **TiO** | **Na2O** | **K2O** | **Organic compound** |
| **Sample №1**  **(upper layer)** | 32,4 | 23,5 | 22,1 | 8,8 | 0,14 | 5,11 | 0,05 | 1,7 | 0,22 | 3,40 | 2,67 |
| **Sample №2**  **(substrate)** | 31,3 | 23,4 | 21,2 | 9,1 | 0,13 | 5,02 | 0,07 | 1,8 | 0,18 | 3,28 | 4,52 |

An EDS map was then created by utilizing BSE (Backscattered Electrons), as well as EDS (Energy Dispersive Spectroscopy) detectors with a SEM and thereby determining its chemical makeup.

**TABLE 2.** Analysis results from spectrum 50 of the sample

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Element** | **Line** | **SEM-determined concentration** | **k Ratio** | **Wt%** | **Wt% Sigma** | **Standard Name** | **Factory Standard** |
| **C** | K | 1.11 | 0.01101 | 11.17 | 0.56 | C Vit | Yes |
| **O** | K | 10.13 | 0.03412 | 46.08 | 0.41 | SiO2 | Yes |
| **Na** | K | 0.22 | 0.00098 | 0.67 | 0.05 | Albite | Yes |
| **Mg** | K | 3.61 | 0.02397 | 11.24 | 0.14 | MgO | Yes |
| **Al** | K | 0.45 | 0.00316 | 1.41 | 0.05 | Al2O3 | Yes |
| **Si** | K | 0.68 | 0.00544 | 1.94 | 0.05 | SiO2 | Yes |
| **Cl** | K | 1.09 | 0.00946 | 2.62 | 0.08 | NaCl | Yes |
| **K** | K | 0.06 | 0.00061 | 0.16 | 0.03 | KBr | Yes |
| **Ca** | K | 10.58 | 0.09465 | 24.07 | 0.25 | Wollastonite | Yes |
| **Sc** | K | 0.11 | 0.00098 | 0.31 | 0.07 | Sc | Yes |
| **Mo** | L | 0.10 | 0.00111 | 0.33 | 0.10 | Mo | Yes |
| **Total** |  |  |  | 100.00 |  |  |  |

**RESULTS AND DISCUSSION**

The existence of the mineral saponite in the mineral dolomite at the Vaush field, located in the Navbahor area. The Vaush Deposit's chemical mineral composition of saponite mineral was also evaluated through analysis of this mineral's chemical mineral structure. Results indicated that saponite mineral contained approximately 20-23% of calcium magnesium oxide. A scientific study of extracting magnesium-based compounds from this mineral is presented.

In order to determine whether or not the results obtained for the composition and structure of synthetic saponite, are accurate, several experiments were performed. These include IR-spectra, differential thermal analysis, and x-ray diffraction analysis, in addition to an Energy Dispersive Spectroscopy (EDS) scan using a Backscattered Electron Detector (BSE). IR Spectral Analysis was conducted on an IRTRACER-100 (Shimadzu, Japan) Spectrometer in the frequency range from 400-4000 cm-1. The samples were formed into tablets using KBr as a base. As indicated in the above section, the presence of Saponite Si-O is confirmed in the IR Spectra of the mineral in the valence vibration region through three distinct absorption bands located at 798.53, 930.54, and 1000.85 cm-1. This band at 930.54 cm-1 corresponds to the tetrahedral silicon oxygen arrangement which is affected by the size of the cationic elements within the crystal lattice of the mineral, since increasing the amount of magnesia causes an increase in the frequency of Si-O oscillations. Additionally, this linear frequency relationship between 930 and 1000 cm-1 represents the most sensitive area of the saponite to changes in iron-magnesium content. Due to the presence of the OH group, numerous absorption peaks appear in the region of 3630-3903 cm-1 of the IR Spectra of saponite [9,10]. Thermal differential analysis was conducted on a Setaram LabSys Evo Derivatograph in the the temperature range from 20 to 800 °C. The rate of heating was 10°C/minute. The synthetic samphire was used as a standard sample. The mass loss of the test sample of saponite at 800°C was 10.22%. The endothermic peak from 90-160°C corresponds to the release of liquid adsorbed onto the internal surfaces of the intergranular minerals that comprise saponite. At temperatures of 675-800°C, rate of weight loss increased dramatically in the decomposition region of the carbonate minerals, with a mass loss of 9.11%, which resulted primarily from the rapid decomposition of calcite. The results of both the chemical and physical studies of compound and features of saponite mineral weight loss rate support its potential use in the agricultural sector, chemical industry, and other sectors of the national economy. Consequently, studies were initiated to examine the dissolution of saponite in both concentrated and dilute hydrochloric acid to produce chloride salts.

(1)

(2)

(3)

Magnesium chloride was produced through the transfer of the solution into an alkaline environment.

Converting the sodium hypochlorite into a magnesium chloride solution using the magnesium chloride solution that is obtained from the mineral saponite was done as follows. First a laboratory glassware equipped with a stirrer that uses a sensor to monitor the temperature to be maintained was attached to a three-necked flask (the volume of the flask was 500 cm3). The contents of the flask were 20 grams of the magnesium chloride salt and the same quantity of sodium hypochlorite. The flask was equipped with a thermostat to ensure the reaction took place at the desired temperature. Then it was stirred vigorously. The reaction was conducted under the conditions of temperatures of 60°C, 75°C, and 90°C and time intervals of between thirty to ninety minutes; during this time the following chemical reactions occurred:

(4)

(5)

The overall reaction for this procedure may be described using the following generalized chemical reaction:

(6)

An intermediate, magnesium hypochlorite, has been found to form initially; then magnesium chlorate is formed from the addition of further sodium hypochlorite.

As a result, when the conversion was done with no vaporization (Table 2), it was established that conviction factor increased as a function of the temperature. As such, at temperatures of 60°C, 75°C, and 90°C, the conversion ratios for the first 60 minutes were 15.06%, 15.83% and 25.90%, respectively. By 90 minutes, the conversion ratio increased to 17.30%, 18.65%, and 32.85%, respectively. As such, the percent (Mg(ClO3)2 present in the slurry form at the above temperatures after 90 minutes was 12.52%, 13.50%, and 23.77%, respectively. It was also established that the increase in the conversion duration had a negligible effect on the degree of conversion.

**TABLE 3.** The influence of temperature and time on the conversion degree and rate constant for non-volatile conversion processes based on temperature and time

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temperature, (T) oC** | **Duration (τ), minutes** | **Concentration of magnesium chlorate in the liquid phase, %** | **Convection coefficient (Ck), %** | **Rate constant, K·10-2 τ-1** |
| 60 | 30 | 5,87 | 8,2 | 0,253 |
| 45 | 9,54 | 13,1 | 0,262 |
| 60 | 10,91 | 15,07 | 0,257 |
| 90 | 12,51 | 17,2 | 0,262 |
| average 0,257 | | | | |
| 75 | 30 | 7,622 | 10,532 | 0,4571 |
| 45 | 9,789 | 13,541 | 0,4619 |
| 60 | 11,461 | 15,829 | 0,4631 |
| 90 | 13,499 | 18,651 | 0,4632 |
| average 0,462 | | | | |
| 90 | 30 | 6,911 | 9,549 | 0,6620 |
| 45 | 12,771 | 17,651 | 0,6680 |
| 60 | 18,739 | 25,900 | 0,6675 |
| 90 | 23,770 | 32,852 | 0,6715 |
| average 0,668 | | | | |

In comparison to the method of converting via evaporation, the conversion rate increases considerably due to an increased rate of the conversion process as indicated by the values listed in table No 3. Evaporating 19.65% of water from the reaction mixture at 60°C for 90 min results in an increased conversion rate of 30.85%; similarly, evaporating 47.33% of water from the reaction mixture at 75°C for 90 min results in an increased conversion rate of 61.81%. The increased temperature enhances the rate of the conversion process and the rate of water evaporation. For example, at 90°C, after 90 minutes the degree of evaporation and the conversion rate were 79.15% and 75.21%, respectively. The results demonstrate that the formation of a solution (Mg (ClO3)2), during the conversion process was analyzed by means of chemical and physicochemical methods. The presence of ClO3- in the solution was measured by the permanganometric technique; while the amount of Mg2+ , in the solution was determined by means of atomic absorption photometry and complexometric methods with the following results.

Composition of theoretically prepared magnesium chlorate wt. %: Mg2+—14.95 wt.%; ClO3– — 43.77 wt.%; Na+ —15.34 wt.%; Cl– —34.06 wt.%; OH– —1.74 wt.%; H2O —3.5 wt.%. Composition of practically prepared magnesium chlorate wt. %: Mg2+ —13.75 wt.%; ClO3– —39.13 wt.%; Na+ —17.13 wt.%; Cl– —38.07 wt.%;   
OH– —1.74 wt.%; H2O —3.3 wt.%.

IR Spectroscopy is a qualitative technique that can be used to determine the structural identity of a new compound and its type of chemical bonding. IR Spectra of magnesium chlorate and NaCl and their individual components were examined to identify the chemical structures and basic types of chemical bonds of the compound, the starting materials NaClO and MgCl2, and the newly formed compound.

**TABLE 4.** Effect of temperature and water evaporation rate on the liquid-phase composition and conversion degree

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Temperature, (T) oC** | **Time (τ), min.** | **Concentration of Mg(ClO3)2 in the liquid phase, %** | **Conversion rate (Ck), %** | **Degree of water evaporation, %** | **Rate constant,K·10-2 τ-1** |
| 60 | 30  45  60  90 | 8,31  16,69  23,78  30,86 | 7,75  15,62  22,23  28,85 | 7,36  11,84  15,86  19,64 | 0,543  0,557  0,553  0,552 |
| average 0,553 | | | | | |
| 75 | 30  45  60  90 | 23,98  33,29  51,69  61,80 | 22,42  31,10  48,32  57,77 | 17,26  30,19  40,19  47,35 | 2,636  2,670  2,685  2,695 |
| average 2,665 | | | | | |
| 90 | 30  45  60  90 | 27,42  39,25  69,77  75,22 | 25,65  36,67  65,20  70,32 | 36,51  55,86  69,54  79,16 | 9,148  9,164  9,322  9,318 |
| average 9,272 | | | | | |

A large number of bands appear in the 3600-3000 cm-1 region of the IR spectra of sodium hypochlorite due to oscillation frequencies relating to crystalline water. Bands appearing in the 1633 cm-1 region relate to deformation vibration of this group. A group at 1400 cm-1 is possibly attributed to an anti-symmetrical stretching vibration of sodium chloride. An intense band remains in the 935-950 cm-1 region and represents symmetrical stretching vibrations of [ClO3]- ions. Bands characteristic of NaClO are found in the 3630 cm-1 region and bands representing both symmetrical and anti-symmetrical stretching vibrations of [ClO]- ions are found in the 671-700 cm-1 region.

Based upon these IR spectroscopy data, it can be concluded that through the conversion process the [ClO]- ion changes into the [ClO3]- and Cl- (NaCl) ions. These findings were concordant with the outcomes obtained from the IR spectroscopy of the products formed by the conversion process, indicating the presence of magnesium chlorate and sodium chloride.

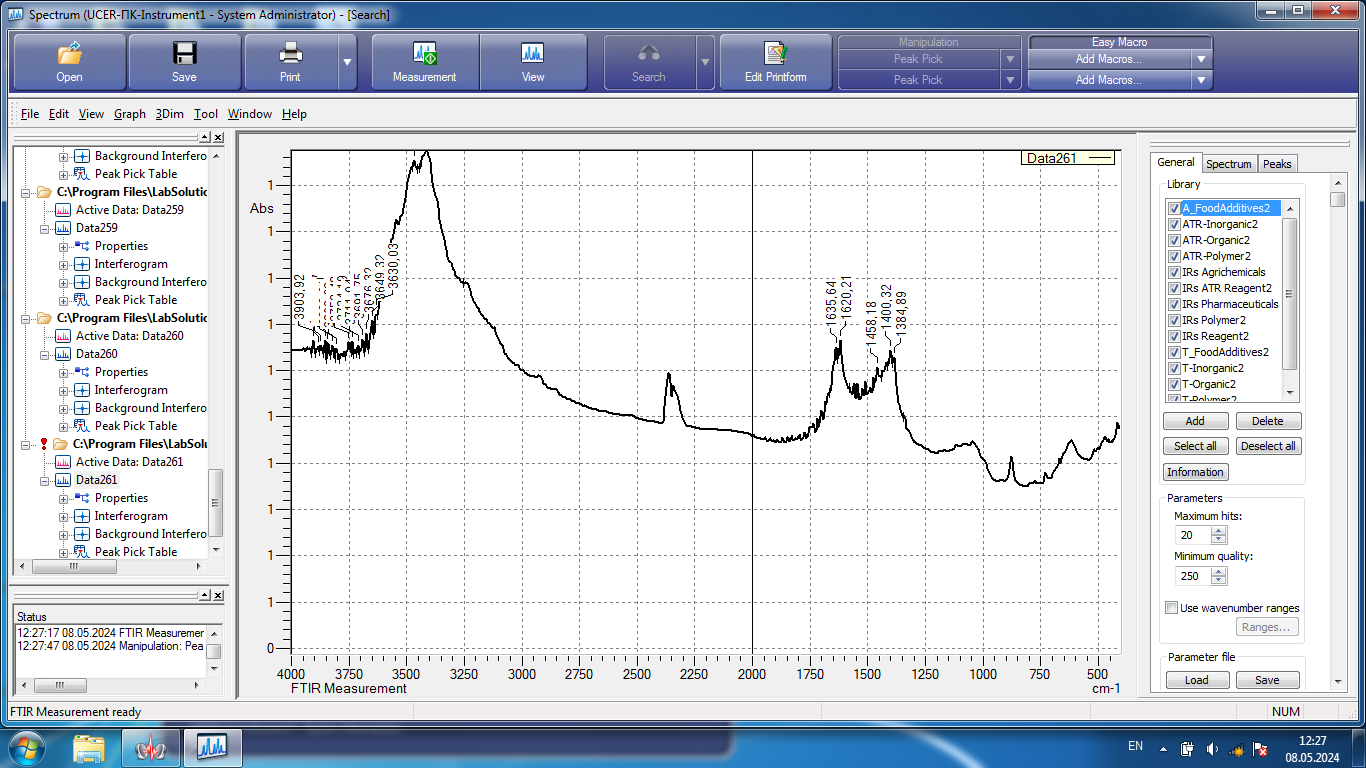
An investigation of the thermal processing of the mineral saponite using CaCl2 salt to form Mg(ClO₃)₂ was performed. This thermal treatment process was done under conditions involving three different weight ratios of saponite to CaCl2 of 50:10, 50:20, and 50:50 at temperatures ranging between 200°C and 600°C.

Calcined Saponite mineral has been treated with calcium chloride (CaCl2) at several different temperatures for sixty minutes. After being heated, the calcined mixture was placed in 500ml of water and allowed to dissolve at room temperature. The solution was filtered into solid and liquid phases, which were then analyzed for their chemical composition and that of the solid phase. The percent composition of the solid phase is reported in the table 5.

As demonstrated by the data in the table, the increase in Mg transferred to the solution from the Saponit mineral due to thermal treatment using CaCl2 increases from 9.42% at a molar ratio of 1:1 to 15.4% at a molar ratio of 1:1.

**TABLE 5.** Outcomes achieved after calcination of saponite mineral with CaCl₂

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Elements | 200° T° (50g+50g) | 300° T° (50g+10g) | 400° T° (50g+20g) | 400° T° (50g+50g) | 500° T° (50g+50g) | 600° T° (50g+50g) |
| Na | - | - | 50,50 % | - | - | - |
| Mg | 12,0 % | 12,7 % | 9,820 % | 12,6 % | 14,4 % | 12,6 % |
| Al | 5,78 % | 6,52 % | 4,05 % | 5,12 % | 6,51 % | 6,52 % |
| Si | 15,5 % | 16,7 % | 8,97 % | 14,7 % | 15,3 % | 10,5 % |
| Ca | 61,0 % | 60,00 % | 25,9 % | 62,6 % | 59,9 % | 64,4 % |
| Fe | 5,74 % | 4,12 % | 1,68 % | 4,94 % | 4,94 % | 6,03 % |

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**FIGURE 1.** FTIR analysis diagram of a blend of saponite and calcium chloride at a ratio of 50:50 g at a temperature of 500 0C. IQ spectrum was obtained in the range of 400-4000 cm-1.

The broad bands centered at 3468 cm−1 and 3406 cm−1 are indicative of hydrogen-bonded structural H2O and hydroxyls that are linked via Mg-OH or Al-OH linkages throughout the crystal lattice of saponite. Bands that appear between 2350 cm−1 are possibly indicative of the presence of carbon dioxide or other gases that are entrapped within the crystal lattice of saponite. However, these bands can also arise from the adsorption of carbonic acid onto the mineral surface. The bands in this range generally do not represent the inherent structure of the clay mineral itself and are indicative of an Exchange of the mineral with the outside environment and/or the existence of contaminants. Bands appearing in the spectrum 1600–1650 cm−1 are generally indicative of water molecule deformation in the interlayer regions of minerals in the smectite family. The presence of adsorbed H2O in saponite results in a features band in this range. The presence of a large, diffuse band at approximately 1404 cm−1 that is representative CO32− ion formation is indicative of some disruption of the saponite lattice. The feature band for saponite appears as a single band in the spectrum 1000–1050 cm−1; the band observed in the spectrum shown above occurs at 1041 cm−1 and represents the stretching vibrations of the Si-O-Si bond in a tetrahedral arrangement. Bands observed in the range 600–900 cm−1 correspond to deformation bands of Al--OH and or Mg--OH units present in octahedral layered saponite. Bands occurring at 460 cm−1 and 420 cm−1 are representative of Si--O bends that are characteristic of silicate minerals, including saponite.

**CONCLUSION**

Recovery of a solution of MgCl2 using the mineral saponite. The chemical and mineralogical characteristics of saponite mineral present in Uzbekistan were established; SEM-EDS-analysis, IQ-spectrum, derivatogram, X-ray phase analysis were used for these purposes. The study showed the obtained results correspond to the data about saponite mineral. The recovery of MgCl2 solution by recycling saponite mineral using hydrochloric acid was also studied. The investigation parameters included: temperature – 80 - 95 °С; HCl concentration - 20%; amount of MgO; HCl norm - 100% and the degree of MgO dissolution - 90.36%. The increase of Mg content in saponite mineral after heat treatment with CaCl2 addition in range 200 - 600 °С were also studied.

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