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Studying the Process of Purification of Solutions with Flotation and Hallurgical Potassium Chloride in Obtaining Potassium Hydroxide

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ABSTRACT: To substantiate the process of obtaining potassium hydroxide, studies were carried out on cleaning the initial solution from impurities of oxides of two and trivalent metals, in particular, calcium, magnesium, which negatively affect the electrolysis process. when cleaning white crystalline potassium chloride, the pH of the solutions rises from 7.01 to 11.53, and when using flotation potassium chloride, this indicator changes slightly - from 11.96 to 12.17. X-ray diffraction analysis showed that the precipitates obtained consist mainly of calcium carbonate and magnesium hydroxide.

KEYWORDS: potassium chloride, potassium hydroxide, concentration, electrolysis, process, precipitate, solution, purification, analysis, calcium carbonate, magnesium hydroxide.

I. INTRODUCTION

The world market for potassium hydroxide is estimated at 2.06 million tons, and the United States is the largest supplier of this product [1].

The main applications [2] of potassium hydroxide are acid neutralization, alkali batteries, detergents, drilling dyes, fertilizers, food production, metallurgical production, oil refining, various organic and inorganic substances, paper production, pesticides, pharmaceuticals, pH regulation, potassium carbonate, and other potassium compounds, soaps.

On an industrial scale, potassium hydroxide is obtained [3-6] by electrolysis of potassium chloride. Three variants of electrolysis are possible: electrolysis with a solid asbestos or polymer cathode (diaphragm and membrane production methods), and electrolysis with a liquid mercury cathode (mercury production method).

Mercury cathodes are used many times more often since they make it possible to obtain a purer substance with a minimum number of impurities. However, it is difficult to call this method safe: if you choose a production method from the point of view of safety, it is better to give preference to the membrane one. Despite the complexity of the process, the risk to workers is minimized.

The membrane method appeared at the end of the 20th century, but the mercury method was used much longer - it was first used at the end of the 19th century. However, recently the mercury method is losing its popularity: the use of mercury cathodes is not only unprofitable from the point of view of the economy but also dangerous for the environment.

From an environmental point of view, the membrane method is better: the technological process involves the flow of wastewater into a new cycle, and not their discharge into the sewer. Accordingly, the environment experiences absolutely no harm. The membrane technique allows to solve the following issues: to exclude the stage of liquefaction and evaporation of chlorine, and to exclude gas emissions of chlorine.

Caustic soda is produced in Uzbekistan by electrolysis of an aqueous solution of sodium chloride by a membrane method. In the same way, potassium hydroxide can be obtained from an aqueous solution of potassium chloride purified by flotation and gallurgical methods.

In this regard, we conducted research on the production of potassium hydroxide based on the above.

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II. MATERIALS AND METHODS

For the precipitation of calcium ions, a 30% solution of potassium carbonate was used, and for the precipitation of magnesium, a 32% solution of potassium hydroxide was used.
In the selected sample, the content of OH-, CO2 was determined [7–9], the content of calcium ion was determined by the trilonometric method [10], and the content of chloride ion was determined by the Mohr method [11]. The potassium content was determined by flame photometry [12].
Morphological studies of the sediment surface of the sample were carried out using an SEM - EVO MA 10 scanning electron microscope (Zeiss, Germany). This instrument is designed for microscopic analysis of the structure and surface defects of inorganic materials, including particles, surface microstructures of metals, semiconductors, and thin films. Experiments on a scanning electron microscope were carried out as follows.
The elemental composition was determined using an SEM - EVO MA 10 scanning electron microscope (Zeiss, Germany) by the method of energy-dispersive X-ray spectroscopy (EDS) in a local area, in which it was determined using an energy-dispersive elemental analyzer of the brand - Oxford Instrument - Aztec Energy Advanced X-act SDD. When obtaining data on the elemental composition, electronic photographs with selected local areas, a table of composition, and a graphical spectrum were presented.
Measurements of the phase characteristics of the precipitates obtained were carried out on a Panalytical Empyrean X-ray powder diffractometer. All control over the operation of the equipment is carried out by means of a computer using the Data Collector program, and the X-ray diffraction patterns were analyzed using the High Score program with the PDF 2013 database. The measurements were carried out at room temperature in the range of 2θ angles, in the range from 5° to 90°, in the step-by-step scanning mode with a step of 0.013 degrees and a signal accumulation time at a point of 5 s [13].

III. RESULTS AND DISCUSSION

It is known that during the production of potassium chloride at the Dekhkanabad potash plant, the flotation method produces potassium chloride of the following composition, (wt.%): KCl - 95.0; NaCl-2.16; n.d.–2.38.
For the physicochemical substantiation of the process of obtaining potassium hydroxide, we studied the physicochemical properties of white crystalline and flotation potassium chloride
Figure 1 shows the energy dispersive spectra of the initial potassium chlorides.
When comparing the obtained data of the energy dispersive analysis of the initial chlorides, it was revealed that their elemental composition is very diverse: white crystalline potassium chloride has the following chemical composition (Table 1), (wt %): K - 51.19, 51.56 and 51.48; C1 - 47.59; 47.67 and 47.69; Na, 0.75-0.90; Ca - 0-0.24.
And in the flotation potassium chloride, the content of elements is, (wt.%): K - 47.08, 47.67 and 47.80; C1 - 48.53; 50.43 and 50.48; Na, 1.23, 1.51, and 2.51; Ca, 0.20 and 1.29 (Table 2).
Therefore, to obtain potassium hydroxide by the electrochemical method, it is necessary to study the purification of impurities of oxides of di- and trivalent metals, in particular: calcium, magnesium, which adversely affect the electrolysis process. In this regard, we studied the process of purification of a saturated solution of potassium chloride. The data are presented in table 3.
Table 3 shows that with an increase in the rate of potassium hydroxide and carbonate from 90 to 122%, when cleaning white crystalline potassium chloride, the pH of the solutions increases from 7.01 to 11.53, and when using flotation potassium chloride, this indicator changes slightly - from 11, 96 to 12.17.

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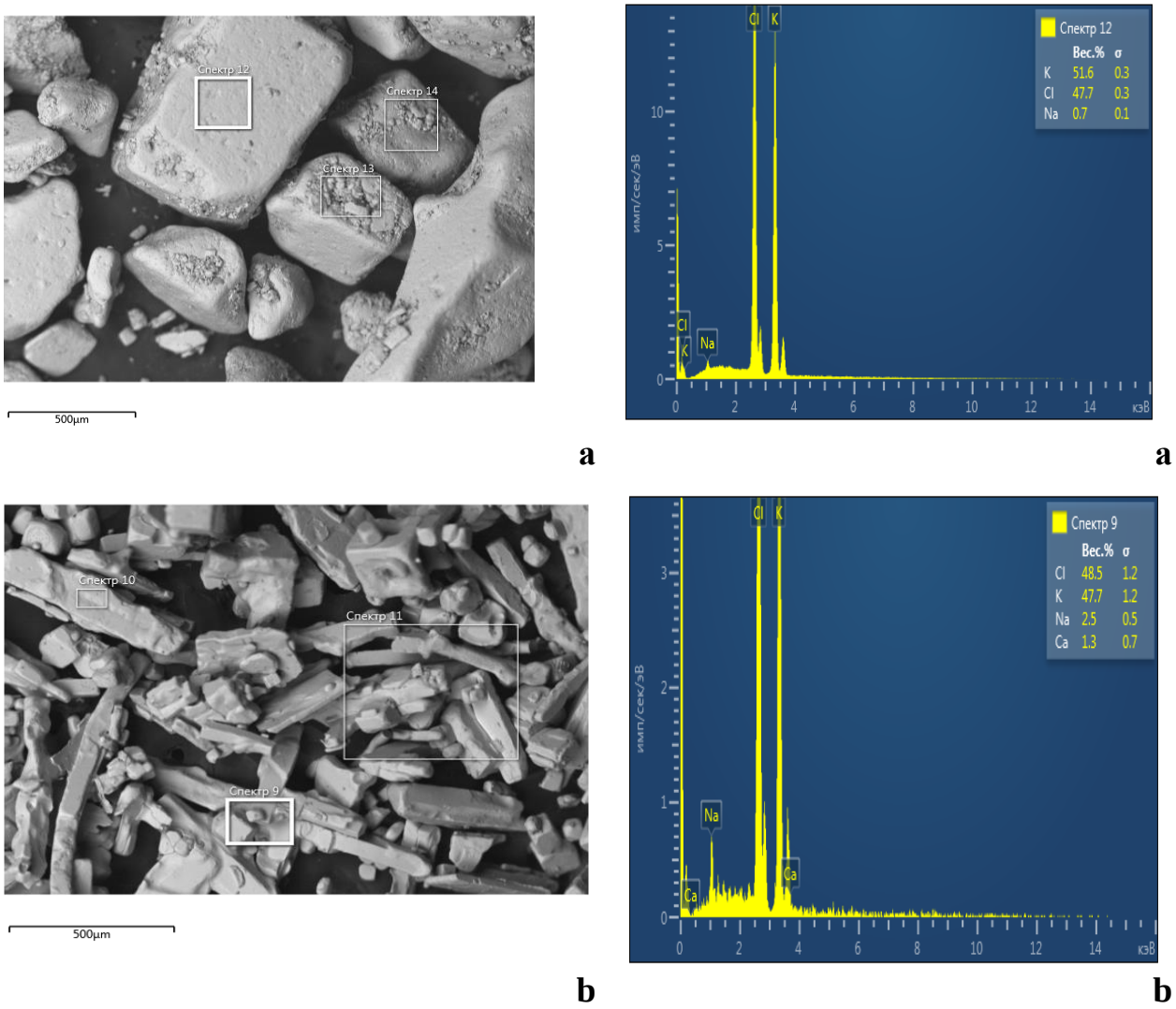

Figure 1. Energy dispersive spectrum of initial potassium chloride samples.
a - white crystalline potassium chloride, b - flotation potassium chloride

Table-1
Content of elements in white crystalline potassium chloride

Spectrum numbers	Content of elements, wt %				
	K	Cl	Na	Ca	Sum:
12	51.56	47.69	0.75	-	100.00
13	51.19	47.67	0,90	0,24	100.00
14	51.48	47.59	0.75	0.18	100.00

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Table-2
The content of elements in potassium chloride obtained by the flotation method

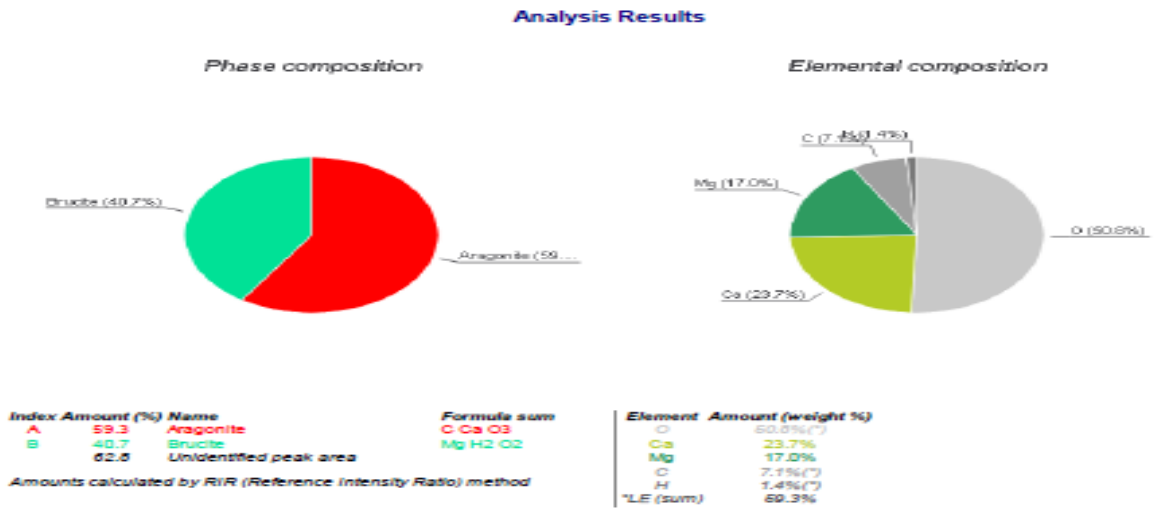
Spectrum numbers	Content of elements, wt %				
	K	Cl	Na	Ca	Sum:
9	47.67	48.53	2.51	1.29	100.00
10	48.08	50.43	1.23	0.26	100.00
11	47.805	50.485	1.51	0,20	100.00

Table-3
Investigation of the influence of the norm of potassium hydroxide and carbonate on the degree of purification of the solution from Ca2+ and Mg2+ ions at a temperature of 50°C

№	Norm	pH	T:Ж	Viscosity, cps (sec)	Density, g/cm3
White crystalline potassium chloride					
1.	90	7,01	1 : 18,2	2,793	1,177
2.	100	11,38	1 : 16,3	2,876	1,179
3.	110	11,50	1 : 15,7	2,907	1,180
4.	122	11,53	1 : 15,4	2,972	1,183
Flotation Potassium Chloride					
5.	90	11,96	1 : 4,43	3,140	1,187
6.	100	12,03	1 : 4,89	3,154	1,189
7.	110	12,12	1 : 5,13	3,209	1,192
8.	122	12,17	1 : 5,42	3,269	1,194

- - mixing time 30 min.

Sample 3



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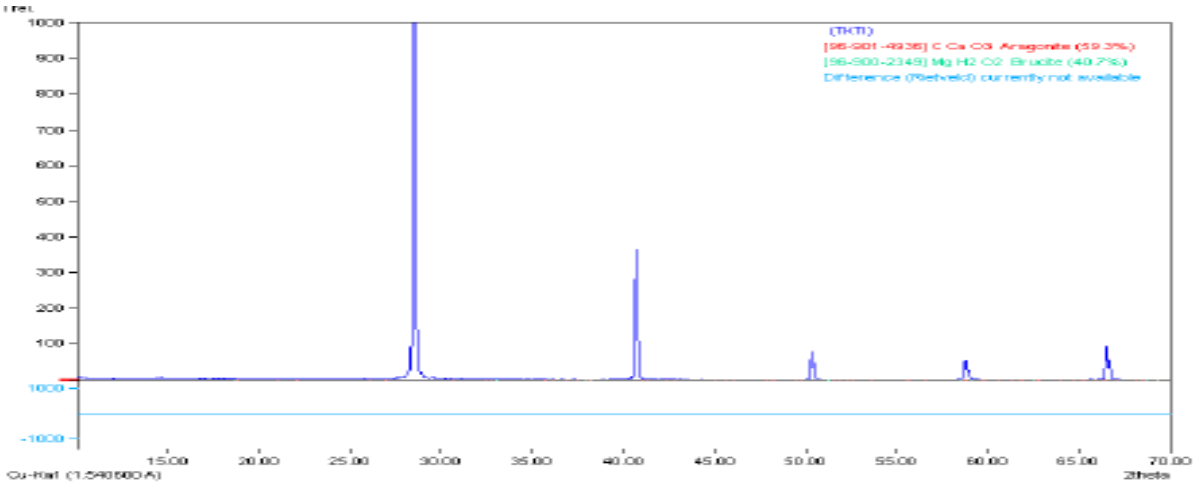

Sample 7

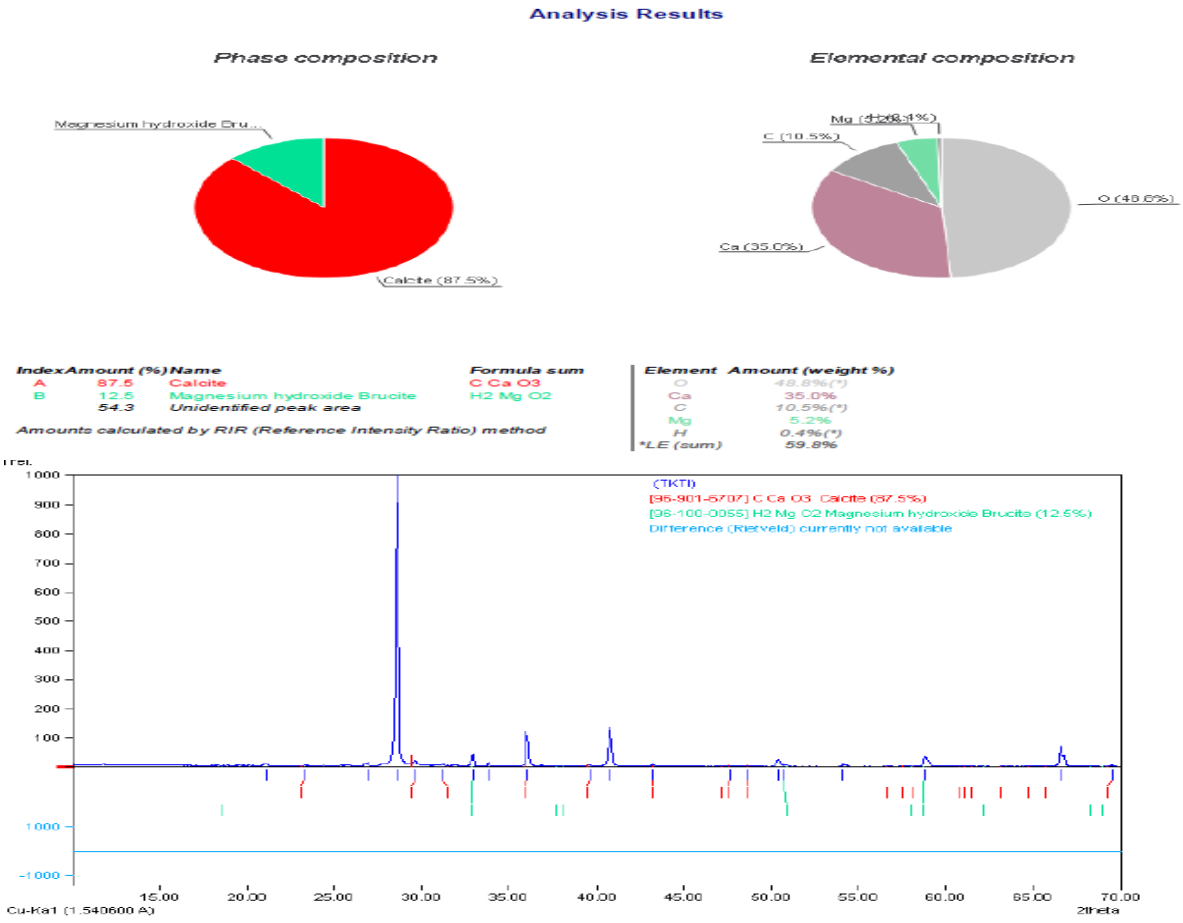

Figure 2 X-ray patterns of the obtained precipitation
The sample numbers correspond to the numbers in Table. 3

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Table-4
The mineralogical composition of the obtained sediments

Sample numbers correspond to the numbers in table 1	Name minerals	Entry number	Formula sum	Crystal system	Unit cell	Содержание, масс% When using the original potassium chloride	
						Flotation at normal	Gallurgical at normal
3.	Calcite	96-901-4936	$CaCO_3$	orthorhombic	a = 4.9713 Åb=8.0311 Åc=5.8141 Å	-	110
	Magnesium hydroxide	96-900-2349	$Mg(OH)_2$	trigonal (hexagonal axes)	a = 3.1264 Åc=4.7315Å		
7.	Calcite	96-901-6707	$CaCO_3$	trigonal (hexagonal axes)	a = 4.9844 Åc=17.0376Å	110	-
	Magnesium hydroxide	96-100-0055	$Mg(OH)_2$	trigonal (hexagonal axes)	a = 3.1420 Åc=4.7660Å		

According to Table 4 and X-ray diffraction analysis (Fig. 2), the precipitates obtained consist mainly of calcium carbonate and magnesium hydroxide.

As the results of the analysis of the pH of the system show, when using the flotation method of purification of potassium chloride, it is necessary to bring the pH of the system to 12.17, in contrast to the gallurgical method, which is associated with a high content of Ca2+ and Mg2+ ions, which requires a greater amount of precipitating alkaline reagents hydroxide and carbonate potassium. With a change in the ratio of solutions, the density and viscosity of the solutions when using white crystalline potassium chloride, the density of the solutions increases, and the viscosity value practically does not change, and when using flotation potassium chloride, the density of the solutions decreases, and the viscosity practically does not change.

IV. CONCLUSION

Thus, as a result of the research, the elemental composition of white crystalline and flotation potassium chloride used to obtain potassium hydroxide was determined, which showed the need for their purification from calcium and magnesium ions. The highest degree of purification of solutions from calcium (81.63%) was achieved at a rate of 122%, and from magnesium - at almost all rates from 100 to 122%.

According to X-ray diffraction data, the precipitates obtained consist mainly of calcium carbonate and magnesium hydroxide.

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