$FeCl_3$-$MgCl_2$-$CaCl_2$-KCl-NaCl-HCl-H_2O carried out by the authors of [5].

In the crystallization of ACH ($AlCl_3 \cdot 6H_2O$) was carried out with gaseous hydrogen chloride (HCl) obtained as a result of the interaction of NaCl with concentrated (94%) sulfuric acid according to the reaction: $H_2SO_4 + 2NaCl = 2HCl\uparrow + Na_2SO_4$ [7]. The authors found that within 15 minutes of the crystallization process, the solution is saturated with HCl vapours, then the first crystals of ACH appear. After 30 minutes of the beginning of the experiment, the authors observed a sharp increase in the number of crystals in the solution. After an hour, the formation of crystals slowed down and practically stopped. The authors' data obtained from studying the effect of temperature on the crystallization of ACH has a great interest. It was found that with an increase in the temperature of the process, the content of the main impurities decreases sharply: chromium by 3.5 times, iron by 2.1 times. The content of Mg and Na is almost halved; the proportion of other impurities does not exceed 10^{-2}%.

An important aspect of the ACH crystallization process is washing the crystals obtained from the residues of the hydrochloric acid solution. The results of works [[6], [7]] on the study of washing aluminium chloride hexahydrate with hydrochloric acid of various concentrations (20, 25, 30, 35.5%) showed good agreement with each other. In experiments with an acid concentration of up to 30%, partial dissolution of the obtained crystals was observed. When using a more concentrated acid (> 30%), the moisture content of the crystals was 25%. As an alternative, an organic reagent, acetone, was chosen, using which the reverse dissolution of ACH was not observed. Humidity was in the range of 3.5-4.5%.

To optimize the crystallization process, it is necessary to have information about the influence of various factors [[8], [9], [10], [11], [12]], which include four parameters that are important for controlling the crystallization of ACH: (1) - the concentration of aluminium chloride in the initial solution for crystallization, (2) - the consumption of gaseous chloride hydrogen, (3) - temperature and (4) - concentration of impurities in the initial solution. Parameters (1), (2) and (3) have a strong influence on the formation of crystals. The crystallization temperature below 60 °C reduces the purity of the crystals, and its increase does not significantly affect the growth of ACH crystals. Nevertheless, the result of parameter (4), the combined effects are also significant since some impurities, in particular phosphorus and magnesium, are concentrated in crystals at the early stages of crystal growth.

The analysis of the results of published works shows the fundamental possibility of crystallising ACH from hydrochloric acid solution with further alumina production by its thermal decomposition. Comparative analysis of the effects of well-known studies, both in terms of the mechanism of the crystallisation processes of ACH and in terms of optimal parameters, shows good agreement with each other. Minor deviations in the quality of the obtained products can be explained by the presence of impurities in the initial solutions and various equipment and techniques for their implementation.

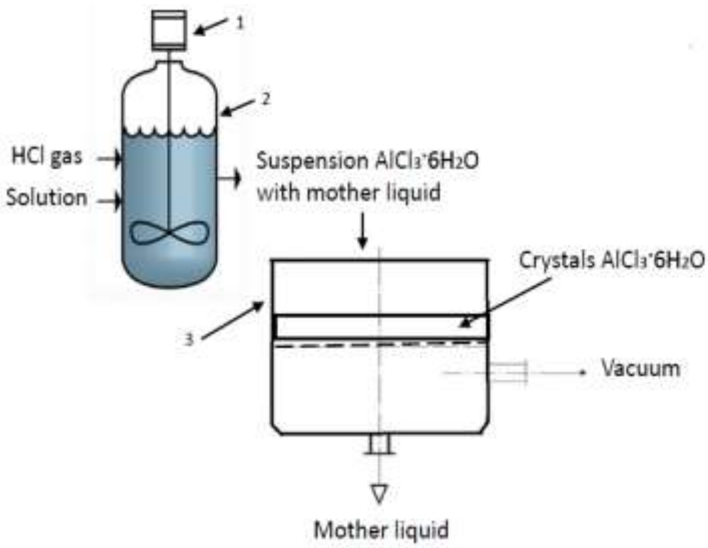
The purpose of this work is to study the effect of the $AlCl_3$ content in the initial solution, the consumption of gaseous HCl, and the behavior of impurities on the crystallization of $AlCl_3 \cdot 6H_2O$ from aluminium-containing salt solutions obtained after leaching the cinder of chlorinating ash roasting from thermal power plants in Kazakhstan [[13], [14]].

Research methods

To study the crystallization process, synthetic solutions were used with the following composition: $AlCl_3$ - 11-15%, $CaCl_2$ - 12-16%, $TiCl_4$ - 0.2-0.3%, HCl - 3-5% and others typical for solutions obtained after leaching the cinder with hydrochloric acid and their filtration. The density of solutions is 1.25-1.29 *g/cm*^{3}.

The experimental technique was as follows. A crystallizer vessel was charged with 1 *l* of an aluminium chloride solution obtained after leaching and filtration. The concentration of aluminium chloride in the solution varied from 10 to 15%. After pouring the solution into the crystallizer, the solution was stirred at a stirrer speed of 250 *rpm*. The process temperature was maintained at 60 °C. Next, hydrogen chloride gas was fed into the crystallizer at a 0.5 *l/min* until its concentration reached 26%. The total duration of the process was 1 *hour*. The obtained crystals of aluminium chloride was separated from the mother liquor by filtration and washed with 26% HCl solution. Then the crystals were dried at a temperature of 80-100 °C.

The mother liquors were sent for the extraction of non-ferrous metals containing rare-earth metals from them. The products obtained in the process of crystallization - crystals of aluminium chloride, mother liquors and solutions after washing the crystals with ACH, were subjected to elemental analysis for the content of aluminium, non-ferrous, rare-earth metals, as well as impurities - phosphorus, iron, sodium, potassium, calcium, magnesium, titanium, barium, etc.



1 - automatic stirrer; 2 - reactor; 3 - suction filter.

Figure 1 - Schematic diagram of a laboratory setup for crystallization of ACH

Results and discussion

A schematic diagram of a laboratory setup for crystallizing aluminium chloride from a solution is shown in Fig. 1.

In crystallization, two products were obtained - mother liquor and crystals of ACH ($AlCl_3·6H_2O$).

The mother liquor composition obtained during the crystallization of ACH from an aluminium chloride solution is shown in Table 1.

Table 1 – Composition of mother liquor obtained during crystallization

Components	Composition	
	g/l	*%*
$CaCl_2$	527.21	40.55
$AlCl_3$	11.32	0.87
$FeCl_3$	37.22	2.86
$MgCl_2$	31.48	2.42
$TiCl_4$	2.37	0.18
KCl	0.42	0.03
NaCl	5.65	0.43
H	85.25	6.82
O	611.88	48.95
Cu	0.013	10.25 ppm
Ni	0.167	128.8 ppm

The consumption of hydrochloric acid determines the yield of ACH crystals. The dependence of the aluminium chloride content in the solution on the increase in the concentration of HCl, constructed from the results of the experiments (Fig. 2), shows a close relationship between these values. The content of aluminium chloride in the solution decreases with an increase in the concentration of hydrochloric acid in the solution.

The obtained results of experimental studies were subjected to mathematical processing with the data of work [5], obtained in similar conditions of the crystallization process. As a result of mathematical processing, a regression equation was obtained that predicts the content of aluminium chloride in solution (y) depending on the concentration of HCl in solution (x), which has the following form:

$$y = 23.162 – 0.675x, \quad r = 0.83 \qquad (1)$$

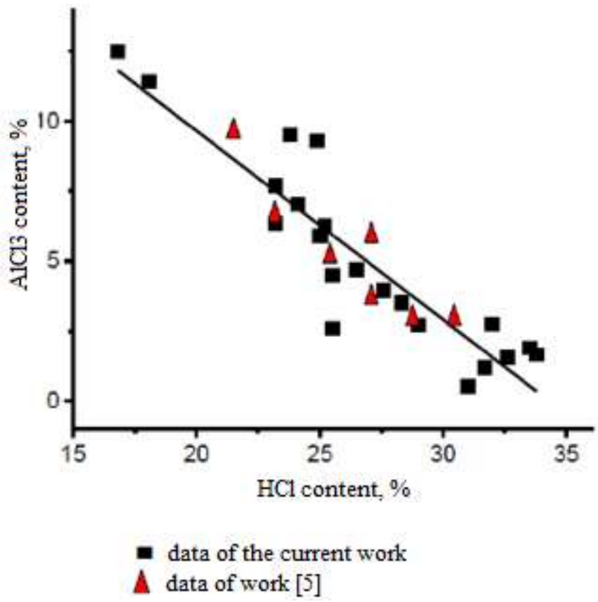


Figure 2 - Dependence of the content of aluminium chloride on the concentration of HCl in solution

Based on the experimental data on the change in the content of aluminium chloride in the solution depending on the concentration of HCl (Fig. 2), the extraction of aluminium from the solution during crystallization was calculated for each experiment. A graphical representation of the dependence of the extraction of aluminium from the solution on the HCl concentration is shown in Fig. 3.

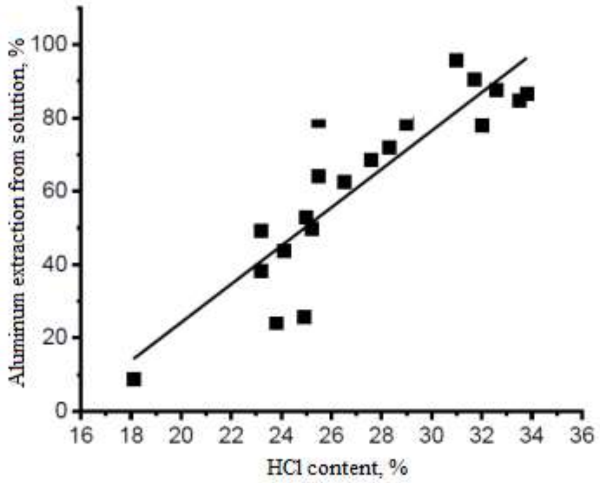


Figure 3 - Dependence of the extraction of aluminium from the solution on the concentration of HCl in the solution

The extraction of aluminium from the solution increases with an increase in the consumption of hydrochloric acid during crystallization. The highest extraction of aluminium from the solution (~ 95%) is achieved when the HCl concentration in the solution is 32%.

As a result of the mathematical processing of the experimental data shown in Fig. 3 (total array - 21 experiments), a regression equation was constructed, making it possible to predict the extraction of aluminium from the solution depending on the concentration of HCl.

The resulting equation has the following form:

$$\xi = -80.379 + 17.939\times[HCl], \quad r = 0.78 \quad (2)$$

where: ξ - extraction of aluminum from solution, %;

[HCl] - concentration of hydrochloric acid in solution, %;

r - the correlation coefficient.

Based on the quantitative ratios of crystallization products - mother liquor and isolated ACH crystals and the results on the content of impurity metals in them, the distribution of impurity metals between the crystallization products was established. The calculation results for the distribution of impurity metals (average values based on the results of several experiments) are shown in Table 2.

Table 2 – Distribution of metal impurities between products during crystallization of ACH

Components	**Metal distribution, %**		
	In mother liquor	Into the solution after washing	Into ACH crystals
Al	2	3	95
Ca	92	7	1
Mg	91	6	3
Fe	92	7	1
Ti	90	9	1
Na	91	7	2
P_2O_5	89	8	3
Ba	15	6	79
Cu	97	3	–
Zn	99	1	–
Ni	98	2	–
Sc	97	3	–
Y	97	3	–

It was found that all metal impurities, except for barium, almost wholly pass into the mother liquor. In solutions after washing with hydrochloric acid, their concentrations are insignificant.

The effect of the solution's acidity on its barium content is shown in Fig. 4.

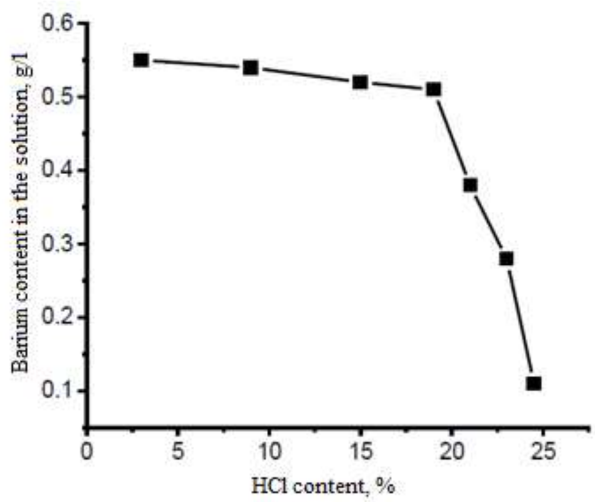


Figure 4 - Dependence of the barium content in the mother liquor on the HCl concentration in the solution

As shown in Fig. 4, an increase in the concentration of hydrochloric acid up to 20% does not affect the barium content in the mother liquor: the barium concentration in the solution remains practically constant ~ 0.55 *g/l*. An increase in the concentration of hydrochloric acid over 20 % leads to a sharp decrease in the barium content. When the concentration of hydrochloric acid in the solution is 26 %, the barium content in the solution reaches its minimum equal to 0.1 *g/l*.

Several impurity metals, even insignificant (except for barium), are concentrated in the obtained crystals of $AlCl_3·6H_2O$ (Table 2). To increase the purity of the obtained crystals, they were subjected to multiple washing with HCl solution (31%), the main meaning was as follows. An initial sample of $AlCl_3·6H_2O$ crystals in 200 *g* was mixed with 400 *ml* of 30% HCl solution and washed at room temperature. The stirring time was 20 *minutes*. After a specified time, the resulting mixture was filtered. The solution was used to wash the next set of crystals. The solution and washed crystals were analyzed for the content of aluminium, iron, calcium, and metal impurities. The operation was repeated five times.

The results of analyzes of the solutions obtained after each washing of the $AlCl_3·6H_2O$ crystals with hydrochloric acid are shown in Table 3.

Table 3 – Compositions of solutions obtained after each wash

Washing	$AlCl_3$, *g/l*	$CaCl_2$, *g/l*	Acidity, pH
Initial solution	-	-	10.1
1	40	6	9.0
2	75	10	7.8
3	100	18	7.0
4	130	23	5.9
5	170	23	5.5

It was found that repeated washing of crystals with a solution of used hydrochloric acid leads to a decrease in the acidity of the washing solution from 10 to 5.5. As a result, partial dissolution of ACH crystals in washing acid with a significant transition of aluminium and calcium into the solution is observed, which is seen in the graphical dependence shown in fig. 5.

The final melt obtained after five times washing of the ACH crystals is sent to the cinder leaching.

The compositions of the crystals obtained after each washing with hydrochloric acid are shown in Table 4.

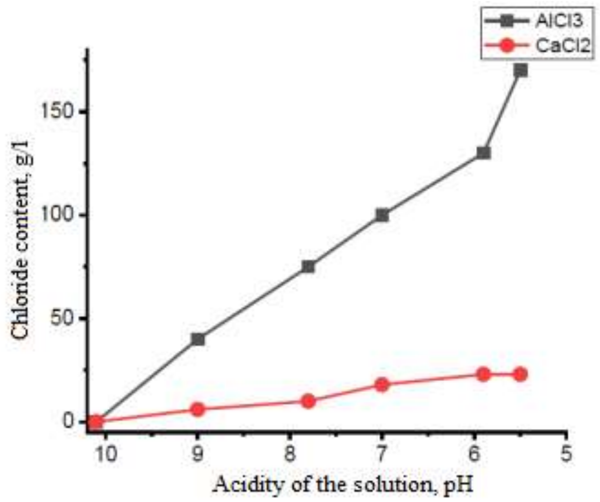


Figure 5 - Dependence of the content of $AlCl_3$ and $CaCl_2$ in the solution on the acidity of the solution with repeated washing of crystals with acid

Table 4 – Compositions of the obtained crystals of $AlCl_3·6H_2O$

Washing	Content of metal impurities, ppm					
	Ca	Fe	Mg	Si	Ti	Na
1	6.5	6.2	2.0	45	0.1	1.0
2	6.0	5.0	1.8	2.0	n.d.	1.0
3	5.0	3.5	1.7	n.d.	n.d.	0.7
4	5.3	3.8	2.1	n.d.	n.d.	0.7
5	4.2	2.8	2.0	n.d.	n.d.	0.5
n.d. – not defined						

The final average composition of $AlCl_3·6H_2O$ crystals obtained after repeated washing with hydrochloric acid contained, ppm: 3-5 Ca, 3-6 Fe, 1-3 Mg, 0.1-0.5 Ti, 1-3 Na, 20-30 P_2O_5. The moisture content of the crystals is 4-5%; the particle size is 400-900 microns.

Based on the carried-out studies and the obtained results, the following optimal parameters for the crystallization of ACH from an aluminium chloride solution were selected:

– crystallization temperature of ACH with gaseous HCl – 60 ºC;

– HCl gas consumption – 0,5 *l/min*;

– concentration of HCl in solution – 26-30 %;

– process duration – 60 *min*;

– ACH crystal washing – repeatable, with hydrochloric acid (32 % HCl).

The crystals of ACH obtained after the crystallization process are sent to a further operation of its thermal decomposition to alumina suitable to produce commercial aluminium.

Conclusions

1. It is shown that it is possible to obtain ACH crystals ($AlCl_3 \cdot 6H_2O$) from aluminium chloride solutions in one stage. The behavior of impurity metals during the crystallization of an aluminium chloride solution has been studied. The distribution of metal impurities between the products of the crystallization process has been established. It was shown that all metal impurities, except barium, pass into the mother liquor during crystallization up to 98%.

2. It was found that repeated washing of ACH crystals with a solution of hydrochloric acid (32% HCl) increases the extraction of aluminium from the solution into crystals up to 96%. It has been shown that a decrease in the acidity of the washing solution from pH = 10 to pH = 5.5 ensures the generation of ACH crystals with a minimum content of impurity metals, ppm: 3-5 Ca; 3-6 Fe; 1-3 Mg; 0.1-0.5 Ti; 1-3 Na; 20-30 P_2O_5. The moisture content of the crystals obtained is 4-5%, and the particle size is 400-900 microns. The recovery of ACH from the solution was 95%.

3. The optimal parameters of the crystallization process of aluminium chloride hexahydrate have been determined: T = 60 ºC, HCl concentration in solution - 26-30%, HCl gas consumption = 0.5 *l/min*, duration 1 *hour*.

Conflict of interests. On behalf of all authors, the corresponding author declares that there is no conflict of interest.

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$AlCl_3$ – MeClx – HCl – H_2O жүйесіндегі $AlCl_3 \cdot 6H_2O$ кристалдану ерекшеліктері

[1] Досмұхамедов Н.К., [1] Жолдасбай Е.Е., [2] Каплан В.А., [1] Даруеш Г.С., [1] Арғын А.Ә.

[1] Satbayev University, Алматы, Қазақстан
[2] Вейцман атындағы Ғылыми институт, Реховот, Израиль

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Қабылданды: *31 желтоқсан 2021*

ТҮЙІНДЕМЕ

Тұз қышқылды ерітінділерден алюминий хлориді гексагидратының кристалдану заңдылықтарын зерттеу үшін зертханалық қондырғы жасалды. Бастапқы ерітіндідегі $AlCl_3$ құрамының, газ тәріздес HCl шығынының және Қазақстан ЖЭС күлдерін хлорлап күйдіру нәтижесінде алынған құрамында алюминий бар күйіндіні тұзды ерітінділермен шаймалау арқылы алынған қоспалардың ерітінділерінің $AlCl_3 \cdot 6H_2O$ кристалдануына әсері зерттелді. Алюминий хлориді ерітіндісінің кристалдану процесінде қоспа-металдарының бөліну ерекшелігі зерттелді және олардың кристалдану процесінің өнімдері арасында бөлініп таралуы анықталды. Ерітіндідегі алюминий хлоридінің мөлшері тұз қышқылының шығыны жоғарылағанда төмендейтіні көрсетілді. $AlCl_3 \cdot 6H_2O$ кристалдану жағдайында барийден басқа барлық қоспалар 98%-ға дейін аналық (кристалдану мен бөліну процестерінен кейінгі қалдық сұйықтық) ерітіндіге өтетіні анықталды. Алынған $AlCl_3 \cdot 6H_2O$ кристалдарындағы барий мен басқа қоспаларды азайту үшін кристалдарды тұз қышқылымен (32% HCl) бірнеше рет жуу ұсынылады. Жуу ерітіндісінің қышқылдығын pH=10-нан pH=5,5-ке дейін төмендеткенде қоспа-металдары минималды түрде АГХ кристалдарына шығарылады, ppm: 3-5 Ca; 3-6 Fe; 1-3 Mg; 0,1-0,5 Ti; 1-3 Na; 20-30 P_2O_5. Алынған кристалдардың ылғалдылығы 4-5 %, ірілігі 400-900 микронды құрайды. Математикалық өңдеу нәтижесінде тұз қышқылының шығынына тәуелді ерітіндідегі алюминий хлоридінің мөлшерін және оның кристалл гидратына бөлінуін барабар болжайтын регрессия теңдеулері құрылды. Кристалдану процесінің оңтайлы параметрлері анықталды: Т=60 ºС, ерітіндідегі HCl концентрациясы 26-30%, газ шығыны HCl=0,5 л/мин, ұзақтығы, τ=1 сағ.

Түйін сөздер: кристалдану, алюминий хлориді гексагидраты, ерітінді, тұз қышқылы, қоспалар, жуу, қышқылдық, экстракция.

Авторлар туралы ақпарат:

Досмухамедов Нурлан Калиевич	*Т.ғ.к., профессор, Satbayev University, 050013, Алматы, Сатпаев көш. 22, Қазақстан. ORCID ID: 0000-0002-1210-4363. E-mail: nurdos@bk.ru*
Жолдасбай Ержан Есенбайұлы	*Докторант, Satbayev University, 050013, Алматы, Сатпаев көш. 22, Қазақстан. ORCID ID: 0000-0002-9925-4435. E-mail: zhte@mail.ru*
Каплан Валерий Аронович	*Т.ғ.к., ғылыми кеңесші, Вейцман атындағы Ғылыми институты, Реховот, Израиль. ORCID ID: 0000-0002-0866-3023*
Даруеш Ғаламат Султанбекұлы	*Докторант, Satbayev University, 050013, Алматы, Сатпаев көш. 22, Қазақстан. ORCID ID: 0000-0001-6739-1569. E-mail: gdaruesh@mail.ru*
Арғын Айдар Әбділмәлікұлы	*Докторант, Satbayev University, 050013, Алматы, Сатпаев көш. 22, Қазақстан. ORCID ID: 0000-0001-5001-4687. E-mail: aidarargyn@gmail.com.*

Особенности кристаллизации $AlCl_3 \cdot 6H_2O$ в системе $AlCl_3$ – MeClx – HCl – H_2O

[1] Досмухамедов Н.К., [1] Жолдасбай Е.Е., [2] Каплан В.А., [1] Даруеш Г.С., [1] Аргын А.А.

[1] Satbayev University, Алматы, Казахстан
[2] Научный институт имени Вейцмана, Реховот, Израиль

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АННОТАЦИЯ

Разработана лабораторная установка для исследования закономерностей кристаллизации гексагидрата хлорида алюминия из солянокислых растворов. Изучено влияние содержания $AlCl_3$ в исходном растворе, расхода газообразного HCl и поведения примесей на кристаллизацию $AlCl_3 \cdot 6H_2O$ из алюминийсодержащих соляных растворов выщелачивания огарка, полученного в результате хлорирующего обжига золы ТЭЦ Казахстана. Изучено поведение металлов-примесей в процессе кристаллизации раствора хлорида алюминия и установлено их распределение между продуктами процесса кристаллизации. Показано, что содержание хлорида алюминия в растворе снижается с увеличением расхода соляной кислоты. Установлено, что в условиях кристаллизации $AlCl_3 \cdot 6H_2O$ все примеси, за исключением бария, на 98 % переходят в маточный раствор. Для снижения бария и других примесей в получаемых кристаллах $AlCl_3 \cdot 6H_2O$ предложено проведение многоразовой промывки кристаллов соляной кислотой (32 % HCl). Показано, что снижение кислотности промывного раствора с pH=10 до pH=5,5 обеспечивает выделение кристаллов ГХА с минимальным содержанием металлов-примесей, ppm: 3-5 Ca; 3-6 Fe; 1-3 Mg; 0,1-0,5 Ti; 1-3 Na; 20-30 P_2O_5. Влажность полученных кристаллов составляет 4-5 %, крупность – 400-900 микрон. В результате математической обработки построены уравнения регрессии, адекватно прогнозирующие содержание хлорида алюминия в растворе и его извлечение в кристаллогидрат в зависимости от расхода соляной кислоты. Установлены оптимальные параметры процесса кристаллизации: Т=60 ºС, концентрация HCl в растворе – 26-30 %, расход газа HCl=0,5 *л/мин*, продолжительность, τ=1 *ч*.

Ключевые слова: кристаллизация, гексагидрат хлорида алюминия, раствор, соляная кислота, примеси, промывка, кислотность, извлечение.

Информация об авторах:

Досмухамедов Нурлан Калиевич	*К.т.н., профессор, Satbayev University, 050013, Алматы, ул. Сатпаева 22, Казахстан. ORCID ID: 0000-0002-1210-4363. E-mail: nurdos@bk.ru*
Жолдасбай Ержан Есенбайулы	*Докторант, Satbayev University, 050013, Алматы, ул. Сатпаева 22, Казахстан. ORCID ID: 0000-0002-9925-4435. E-mail: zhte@mail.ru*
Каплан Валерий Аронович	*К.т.н., научный консультант, Научный институт имени Вейцмана, Реховот, Израиль. ORCID ID: 0000-0002-0866-3023*
Даруеш Галамат Султанбекулы	*Докторант, Satbayev University, 050013, Алматы, ул. Сатпаева 22. Казахстан. ORCID ID: 0000-0001-6739-1569. E-mail: gdaruesh@mail.ru*
Аргын Айдар Абдилмаликулы	*Докторант, Satbayev University, 050013, Алматы, ул. Сатпаева 22, Казахстан. ORCID ID: 0000-0001-5001-4687. E-mail: aidarargyn@gmail.com*

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