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TYPES AND MAIN CHARACTERISTICS OF ALUMINUM-CONTAINING COAGULANTS

Makhmudov Mukhtor Jamolovich [1]**,**
Haydarov Bekzodjon Abdumalik o'g'li [1]

[1] Bukhara Engineering and Technology Institute, Bukhara, Uzbekistan

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Abstract

Today, various coagulants are used in the world to purify various industrial waters produced in the oil and gas processing industry. This leads to an increased demand for coagulants. This article provides detailed instructions on how to obtain modern coagulants with preserved aluminum, their main characteristics and types.

Keywords: *oil, gas, technical waters, coagulant, aluminum, aluminum sulfate, aluminum oxide*

Aluminum – containing coagulant – $Al_2(SO_4)$ is widely used in the process of water purification. To a lesser extent, aluminum chloride is used, as well as sodium aluminate.

Currently, purified aluminum sulfate in production conditions is mainly obtained from aluminum hydroxide. This method is the simplest and most rational, it allows you to obtain a high-quality product with a low content of iron oxide.

Aluminum hydroxide is an intermediate product in the production of alumina, which is obtained by the Bayer alkaline method mainly from high-quality bauxite. Alumina is produced in smaller quantities from nephelines by sintering them with limestone, as well as from alunites by an alkaline reduction method (Khalilov S. Kh., Makhmudov M. Zh., 2022).

The production of aluminum sulfate consists of two main technological stages – decomposition of hydroxide with sulfuric acid and crystallization of the resulting product. The interaction of aluminum hydroxide with sulfuric acid is described by the reaction:

$$2Al(OH)_3 + 3H_2SO_4 + Q_q = Al_2(SO_4)_3 + 6H_2O + Q_q$$

In (Edelstein A. S., Cammarata R. C., 1998; Ostrovsky N. M., 2007), the solubility of aluminum hydroxide freshly deposited and obtained at alumina plants in dilute sulfuric acid solutions was studied, depending on particle size, impurity content, storage time, and methods for preparing hydroxides. It was found that fine-grained aluminum hydroxide is better soluble in sulfuric acid than coarse-grained. Long-term storage of hydroxide reduces the rate of its dissolution in sulfuric acid. Amorphous hydroxide dissolves faster than crystalline hydroxide.

Aluminum sulfate can be obtained by decomposition of aluminum hydroxide with a stoichiometric amount of 45–50% sulfuric acid at 110–120 °C for 40 minutes. By lowering the acid dose to 95–90%, a product with a higher content of aluminum oxide is obtained with satisfactory crystallizability (Chen S., Yuan, Z., Hanigan D., Westerhoff P., Zhao H., Ni J., 2018).

Aluminum sulfate is produced in periodic or continuous devices with a capacity of 10–150 thousand tons of product per year.

In (Du B. D., He T. T., Liu G. L., Chen W., Wang Y. M., Wang, W., Chen, D.M., 2018; Chuang S. H., Chang T. C., Ouyang C. F., and Leu J. M., 2007) the decomposition of high-iron bauxite with sulfuric acid was studied. Bauxite was characterized by the following chemical composition (in %): Al_2O_3–40.8; Fe_2O_3–27.0; SiO_2–8.7; TiO_2–2.2; CaO – 0.75; MgO – 0.2; Na_2O+K_2O – 0.5; pp.p. – 0.7. With an increase in temperature from 20 to 98 °C and the concentration of sulfuric acid from 10 to 60% extraction of aluminum and iron oxides into the solution increases, reaching 78.9 and 71%, respectively, at 98 °C and 1hour exposure. The degree of extraction of these components at boiling point depends on the duration of the process: with the decomposition of 60% H_2SO_4 bauxite, with an increase in the duration of the process from 5 to 90 minutes, the extraction of Al_2O_3 increases from 67.6 to 94%, and Fe_2O_3 – from 57.3 to 93.1%. boehmite bauxites are worse opened with sulfuric acid. Exposure to 10 n sulfuric acid for 1 hour leads to complete dissolution of gibbsite, partial (by 30%) – kaolinite and insignificant – boehmite. After firing at 550 °C and subsequent decomposition of bauxite for 3 hours, 90% Al_2O_3 is extracted into the solution.

In (Makhmudov M. Zh., Yomgurov S. A., 2023) it is proposed to decompose bauxite with dilute sulfuric acid at 135 °C in a steel reactor lined with acid-resistant tiles.

Initially, 93% H_2SO_4 is fed into the solution, it is diluted with washing waters from sludge washing and heated to a boil, after which bauxite is introduced in small portions to avoid foaming and $Al_2(SO_4)$ is obtained.

The process of obtaining aluminum sulfate from low-quality bauxites is described. Another source of aluminum-containing coagulant is kaolin clays.

In (Dzhamolovich M. M., Ugli Y. S., 2024) kaolins are difficult to open with sulfuric acid. Despite the large number of works devoted to the study of thermochemical transformations of kaolin, there is still no consensus on the mechanism of the process. Based on a complex of studies using physico-chemical methods, including X-ray phase analysis and IR spectroscopy, the mechanism of thermochemical transformations of kaolinite can be represented by the following reactions:

$$Al_2Si_2(OH)_4 \xrightarrow{450\text{-}550^{\circ}C} Al_2O_3 * 2Si_2O_2 + 2H_2O;$$

$$Al_2O_3 * 2SiO_2(OH)_4 \xrightarrow{830\text{-}850^{\circ}C} j\text{-}Al_2O_3 + 2SiO_2;$$

$$3(j\text{-}Al_2O_3) + 2SiO_2 \xrightarrow{900^{\circ}C} 3Al_2O_3 * 2SiO_2.$$

Aluminum alum is also used as coagulants, but they are used less often than aluminum sulfate, which is due to their high cost. In addition, alkali metal sulfates, which are part of aluminum alum, saline the purified water, reducing coagulating properties. Therefore, ammonium-, potassium- or sodium-aluminum alum is used, which has good coagulating properties, as well as more affordable and cheaper ones. Ammonia alum is used for the treatment of chlorinated water (Makhmudov M., Nematov H., Rizayev S., 2023).

An aluminum-containing coagulant can also be obtained from nepheline. In this case, it is advantageous to obtain alum, which is a more effective coagulant.

Nepheline reacts with sulfuric acid at room temperature with the release of a large amount of heat by reaction:

$$(Na, K) / AlSiO_4 / + 2H_2SO_4 + nH_2O \rightarrow (Na, K) / Al(SO_4)_2 * 12H_2O + SiO_2 + (n-12)H_2O\uparrow$$

The sulfatization process is greatly influenced by the concentration of sulfuric acid and its dose. The higher the concentration of sulfuric acid, the higher the temperature the reaction mass is heated due to the released heat of the reaction. At the same time, more water is released into the vapor phase and alum is formed with a lower content of crystallization water. As a result, anhydrous alum can be formed – double sodium-potassium aluminum sulfates. The choice of the acid concentration is primarily determined by the possibility of separating the silica sludge from the sulfuric acid solution (Ostrovsky N. M., 2007; Chen S., Yuan, Z., Hanigan D., Westerhoff P., Zhao H., Ni J., 2018).

In (Makhmudov M., Nematov H., Rizayev S., 2023), a method for obtaining $Al_2(SO_4)$ was developed from nepheline concentrate by a two-stage method. The product is decomposed in the first stage with nitric acid to produce potassium and sodium nitrates, and in the second stage the solid residue is treated with sulfuric acid to produce aluminum sulfate.

Until recently, the technology for producing aluminum dihydroxosulfate $Al_2(SO_4)_2(OH)$ has not been developed$\cdot 11H_2O$, therefore it was rarely used as a coagulant. However, extensive studies are currently being conducted at the Institute of Colloidal Chemistry and Water Chemistry of the Academy of Sciences of Ukraine, which show the effectiveness of this salt as a coagulant for wastewater and drinking water treatment (Makhmudov M.Zh., Salomov S. S., Savriev M. S., 2021). Aluminum dihydroxosulfate has a number of advantages over aluminum sulfate: it has a higher flocculating ability at low temperatures, requires less alkaline reserve and is effective in a wider range of pH values of the treated water (Aristov Yu.I., 2008).

Aluminum-containing coagulants also include chlorine-containing reagents. Aluminum chlorides and hydroxochlorides – $18Al_2(OH)_nC_{16\text{-}n}$ are most often used in water purification from chlorine-containing aluminum compounds. Currently, aluminum pentahydroxochloride – $Al_2(OH)_5Cl$, which has effective coagulating properties, is also widely produced on an industrial scale and is widely used for water purification. Aluminum pentahydroxochloride contributes to the intensive formation of flakes and precipitation of coagulated suspensions. When cleaning waters with a low content of suspended particles and salts, the consumption of this coagulant is significantly lower compared to classical aluminum sulfate. When using aluminum pentahydroxochloride, the range of optimal pH values of the treated waters is significantly expanded.

Aluminum pentahydroxochloride is characterized by lower acidity, so it is effective in purifying waters with small alkaline reserves. In comparison with aluminum sulfate, an equivalent amount of aluminum pentahydroxochloride reduces the alkalinity of water by six times when interacting with calcium bicarbonate. Aluminum pentahydroxochloride contains fewer chlorine ions, therefore, compared with aluminum sulfate, therefore, when used in purified water, there are fewer salts. In addition, the content of residual aluminum in the treated water is noticeably reduced (Patent. 2264589; Desai R., 1992).

One of the effective coagulants is aluminum pentahydroxochloride (Taran, V.N., 2004), which is characterized by higher amounts of water-soluble aluminum compared to other coagulants, and for its solutions there is no need to use anticorrosive protection and stainless steels to preserve pipelines and equipment. This coagulant has a long shelf life (Taran, V.N., 2004).

Chlorine technology is the most advantageous for processing hard-to-open and low-grade aluminum-containing ores, since the technological process provides for the production of aluminum chloride, which can be used as a coagulant (Buluchevsky E. A., 2011).

In (Kuvshinnikov I. M., 1987; Buluchevsky E. A., 2011), technologies have been developed for the production of anhydrous aluminum chloride from various aluminum-containing raw materials. The monograph by B. G. Rabovsky and A. A. Furman (Reshetnikov S. I., 2017) systematizes practical experience and theoretical knowledge in this field.

For oxide raw materials (natural aluminum-containing ores or alumina), chlorination is carried out only with a reducing agent. The reducing agent in this case is coal, clay–like reducing agents are also used – phosgene, generator gas, carbon monoxide, from solid ones – coal pitch and coke. In the presence of these reducing agents, the chlorination process proceeds at temperatures from 1000 to 1100 °C.

Some researchers, in addition to chlorine, suggest the use of carbon tetrachloride, sulfur chloride, hydrogen chloride, etc. as chlorinating agents. It is noted that alumina reacts with hydrogen chloride at a higher temperature, and the reaction itself is reversible. When a small amount of coal is added to the reaction mixture, increased formation of aluminum chloride is noted. When mixing sulfur chloride and chlorine, there is an increase in both reducing and chlorinating properties. Therefore, a lower temperature is necessary for this reaction to occur. The complete interaction of carbon tetrachloride with alumina occurs at a temperature of 390 °C, while $AlCl_3$ is formed.

For alumina, chlorinating agents in the presence of aluminum ores or carbon can also be iron, sodium and calcium chlorides.

In the monograph (Taran, V.N., 2004), several methods for aluminum chlorination are considered, in particular, chlorination in a fluidized bed (the fluidized bed is created by inert materials, for example, sand, or inert gases).

Industrial technological schemes for the production of aluminum chloride, which were developed by Soviet, German and American specialists, are also presented here.

In production conditions, the production of aluminum chloride consists of several stages. At the first stage, concentrated carbon monoxide is produced in special generators in which coal coke is gasified with oxygen. In the second stage, kaolin briquettes are prepared, dried and calcined, the main task of the second stage is the chlorination of kaolin briquettes, which is carried out in the presence of carbon monoxide with chlorine gas in a continuous shaft furnace.

Aluminum chlorides are obtained by the basic reaction:

$$Al_2O_3 + 3Cl_2 + 3CO \longrightarrow 2AlCl_3 + 3CO_2$$

Silicon oxide also reacts with chlorine by reaction:

$$Si_2O_3 + 2Cl_2 + 2CO \longrightarrow SiCl_4 + 2CO_2$$

As shown in (Sircar S., Rao M. B., Golden T. C., 1996), when aluminum and silicon oxides interact with chlorine, the phenomenon of mutual catalysis is noted, when the rate of chlorination of mixtures is higher than when chlorinating individual oxides. According to (Wei, N., Zhang, Z., Liu, D., Wu, Y., Wang, J., Wang, Q., 2015; Xiao, F., Guo, Y., Yang, R., Li, J., 2019), silica and alumina are chlorinated in the temperature range from 550 to 800 °C in the same ratios as in the feedstock.

The literature provides contradictory data on the interaction of chlorine with oxides at higher temperatures. Thus, Kocharovsky (Xiao, F., Ma, J., Yi, P., Huang, J.-C.H., 2008), investigating the chlorination of Polish clays, found that at temperatures above 900 °C, the yield of $AlCl_3$ decreases. Meanwhile, according to Kocharovskys data, confirmed by long-term practice, the yield of $SiCl_4$ in the temperature range from 1000 to 1200 °C reaches minimum values.

These contradictions can be explained by the fact that in (Gritsenko A. I., 1999) the results were obtained in a shaft furnace of continuous countercurrent action, that is, under conditions of a secondary reaction:

$$2Al_2O_3 + 3SiCl_4 \longrightarrow 3SiO_2 + 4AlCl_3$$

In (Natural gas. 2006; Yakushev V. S. 2009; Nagata I., 1966), the results of a study on the chlorination of various types of aluminum-containing raw materials are presented. The authors (Wei, N., Zhang, Z., Liu, D., Wu, Y., Wang, J., Wang, Q., 2015; Gritsenko A. I., 1999) studied the chlorination process of the North Voronezh bauxites, which was carried out in a fluidized bed. It is shown that all oxides included in the bauxite composition are chlorinated in the fluidized bed. The most easily chlorinated are iron and titanium oxides. Optimal chlorination conditions were found, at which the degree of chlorination of iron and titanium oxides reaches 98%, aluminum oxide – 65%, and silicon oxide – 20–35% (Sloan E. D., Koh C. A., 2008).

In (I. A. Zolotarsky, L. I. Voennov, L. Yu. Zudilina, L. A. Isupova, R. A. Zotov, D. A. Medvedev, D. A. Stepanov, A. V. Livanova, E. P. Meshcheryakov, I. A. Kurzina. 2017), a technique was developed for the selective chlorination of bauxites in a fluidized bed to produce aluminum oxide in the temperature range from 800 to 900 °C with a mixture of chlorine and silicon tetrachloride. Chlorination of powdered bauxite in a fluidized bed with 1% carbon makes it possible to remove 65% of iron from it at a temperature of 800 °C, while losing 8% of aluminum (for Uglian bauxite). The chlorination of Prosyanovo kaolin of Ukhta and Severoonezhsky bauxites in the filter layer was studied.

The authors (Reshetnikov S. I., Livanova A. V., Meshcheryakov E. P., Kurzina I. A., Isupova L. A. 2017; Minakova T., 2007) studied the process of chlorination of the mineral part of the Ekibastuz carboniferous rocks. It is shown that during chlorination of these rocks, the yield of iron and silicon oxides is 98–100%, and the yield of aluminum oxide is 91–96%. It is proposed to carry out preliminary hydrochloric acid treatment to pre-release this raw material from silica, or to suppress the process of chlorination of SiO_2 with silicon tetrachloride, to separate silicon and aluminum oxides.

In (Moulton, B. & Zaworotko, M.J., 2001), a series of studies on hydrochloric acid chlorination of carbonaceous rocks of the Ekibastuz deposits was carried out. The authors showed that under certain chlorination conditions, iron oxides can be successfully distilled by 7%, while other oxides of the raw material are not significantly affected.

In (Buleiko V. M., Vovchuk G. A., Grigoriev B. A., Istomin V. A., 2014), a technology for processing kaolin clays from a deposit in Georgia (USA) by carbochlorination was developed. In (Istomin V. A., Fedolov D. M., 2013), a method for processing highly siliceous alumina-containing ores to produce iron oxides was developed, including heat treatment of ore for 30–60 minutes at a temperature from 400 to 500 °C in an atmosphere of chlorine gas. It has been shown that in the presence of natural gas, the yield of iron oxides increases.

A method of purification of aluminum ores from iron has been developed, consisting in reductively sulfidated roasting, chlorination and sublimation of iron chloride (Kalacheva L. P., Fedorova A. F. 2016).

The authors (Maxmudov M. J., Ne'matov X.I., 2023) present a developed method for selective low-temperature reduction of a mixture of iron and aluminum chlorides to metals. It is recommended to carry out the process in an inert gas stream containing hydrocarbons, for example, methane, as well as iron and aluminum chlorides.

The chloride reduction process is recommended to be carried out on the surface of iron, which increases the selectivity of reduction and significantly reduces the temperature of the process: $AlCl_3$ – up to 450 °C, $FeCl_3$ – up to 350 °C, the authors recommend using silicon or manganese instead of jelly, however, on their surfaces the reduction of aluminum chloride occurs at higher temperatures, 950 and 800 °C, respectively.

H. Safiev et al. (Maxmudov M. J., Ne'matov X.I., 2023; Llewellyn, P. L. et al., 2008) studied the decomposition of nepheline syenites from the Turpi deposit of the Republic of Tajikistan by chlorination to produce oxides, and also considered the main thermodynamic characteristics of the processes occurring during chlorination of the main minerals of the nepheline syenite rock (nepheline – $Na_{0,78}K_{0,22}AlSiO_4$, albite – $NaAlSi_3O_8$, microcline – $KAlSi_3O_8$, calcite – $CaCO_3$, hematite – Fe_2O_3 and goethite is FeO(OH). This method is based on the use of coals, carbon monoxide, methane and phosgene and methane as reducing agents. The following probable ways of processes have been identified:

$$M + X + C \longleftrightarrow EX + CO_2,$$

$$M + X + CO \longleftrightarrow EX + CO_2,$$

$$M + X + COCl_2 \longleftrightarrow EX + CO_2,$$

$$M + X + CH \longleftrightarrow EX + CO_2 + H_2O.$$

where: *X is chlorine gas; M is the corresponding mineral; EH is a mixture of chlorides of the corresponding elements in the composition of minerals*

Based on the comments, it can be assumed that feldspar and hematite are chlorinated more effectively when using coal as a reducing agent, and when chlorinating calcite, it is more rational to use methane as a reducing agent. Chlorination of minerals in the presence of a reducing agent – carbon monoxide in the studied temperature range is thermodynamically impossible, and when using phosgene as a reducing agent, all minerals except goethite must be chlorinated equally vigorously.

The conducted studies (Maxmudov M. J., Ne'matov X.I., 2023; Artemova I. I., Kandaurov S.Yu., Bachalov I. S. et al., 2010) have shown the possibility of selective decomposition of Turpi nepheline syenites by chlorination, to obtain a mixture of chlorides of a given composition, depending on the goal. When chlorinating nepheline syenites in the presence of coal, the authors of (Maxmudov M. J., Ne'matov X.I., 2023) achieved sufficiently high degrees of conversion of aluminum oxide to chloride, but at the same time oxides of silicon, calcium, potassium, sodium and iron also pass into chlorides.

The authors of (Vuong D. H., Zhang H. -Q., Sarica C., and Li M., 2009) have developed a method for chlorination of nepheline syenites in a methane atmosphere, consisting of two stages. At the first stage, iron-containing minerals are chlorinated under certain conditions to degrease raw materials, and the second stage consists of chlorination of aluminum-containing minerals. In the second stage, a mixture of chlorides is obtained, which is a good electrolyte for the production of aluminum and chloride melts.

There are several options for conducting the process of chlorine decomposition of these minerals with different initial and final products. Calculations of the temperatures of the beginning of the reaction (T_n), changes in the Gibbs energy (ΔG) and the logarithm of the equilibrium constant (lgK) were carried out using a computer program (Llewellyn, P. L. et al., 2008).

In (Maxmudov M. J., Ne'matov X.I. 2023), the chemical foundations of the chlorine method of processing aluminosilicate ores of Tajikistan were studied, rational conditions for the chlorine process were proposed, and basic technological schemes for the production of aluminum-containing coagulants were developed.

Thus, the high ability of chlorine to enter into chemical reactions, as well as the ability of chlorides to interact with other compounds, make it possible to isolate valuable components from processed raw materials, including aluminum chloride, a coagulant for water purification.

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Contact: makhmudov.mukhtor@inbox.ru