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Metallurgy

Influence of silica on the crystallization of sodium hydroaluminate

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ABSTRACT

To extract aluminium from highly concentrated aluminate solutions of alumina-containing raw materials, decomposition is performed through the crystallization of sodium hydroaluminate. This paper presents the results of a study on the stability of aluminate solutions. To calculate the probable yield of Al_2O_3 in the solid phase, it is necessary to determine the crystallization rate of sodium hydroaluminate according to the composition of the initial solutions and the conditions of the process. The influences of SiO_2 content on the decomposition of aluminate solutions with Na_2O_k concentrations of 540 g/dm^3 and 590 g/dm^3 with and without inoculum were studied. In addition, the influence of silica on the appearance of precipitating crystals of sodium hydroaluminate was considered. A sharp increase in the stability of the aluminate solutions at rest in the presence of silica was observed. Different crystallization conditions for sodium hydroaluminate have been investigated. Silica slows the crystallization process of sodium hydroaluminate, affecting both the nucleation and crystal growth rates. The effect of SiO_2 on the rate of decomposition is similar to the reduction in the degree of supersaturation.

Keywords: Alumina solution, silica, sodium hydroaluminate.

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Introduction

The sintering and Bayer methods are used to process alumina-containing raw materials used in industry [[1], [2], [3], [4], [5]]. Processing by the hydrochemical alkaline method results in high-modulus aluminate solutions with caustic modulus (α_k) values of 6–12. The properties of aluminate solutions significantly depend on the technological process [[6], [7], [8], [9][10]]. Two methods are used for aluminium extraction from the obtained aluminate solutions: decomposition and carbonization [[11], [12], [13], [14]].

Aluminium hydroxide precipitation is performed by the spontaneous decomposition of supersaturated solutions.

When $\text{Al}(\text{OH})_3$ is separated by carbonization, the solution is treated with CO_2 -containing gases.

When opening high-siliceous aluminosilicate rocks with hydroalkali technology, high-modulus aluminate solutions are obtained. In addition, spontaneous decomposition of these solutions with aluminium hydroxide extraction is impossible, and carbonization is economically unfeasible due to the transfer of expensive caustic alkali into carbonate products. Therefore, for aluminium extraction from highly concentrated aluminate solutions, a decomposition method based on the crystallization of sodium hydroaluminate is used. Then, the obtained product is dissolved in industrial water to obtain aluminium hydroxide by decomposition.

The crystallization process is a bottleneck of the hydrochemical method. The obtained solid phases are finely dispersed, contain a large amount of entrained mother liquor and are poorly filtered; additionally, the resulting solution has a high caustic modulus (1.8-2), which adversely affects the subsequent conversion–decomposition process [11].

To calculate the probable yield of Al_2O_3 in the solid phase, it is necessary to determine the crystallization rate of sodium hydroaluminate according to the composition of the initial solutions and the conditions of the process. Of great importance for the crystallization of sodium aluminate is the presence of silica in the solution.

Silicon is the main impurity in bauxite, and is also one of the most harmful impurities in the process of the alumina production using alkaline method such as the conventional Bayer process [15]. The presence of even small amounts of silica in Oaluminate solutions sharply inhibits the crystallization of sodium hydroaluminate. To realize its recycling, the alkali-silicate solution needs to be desiliconized [16]. After leaching by a hydrochemical method, the solutions contain a maximum of $10 \text{ g/dm}^3 \text{ SiO}_2$, from which only a small part of the Al_2O_3 can be separated into a solid phase. Moreover, the crystallization process proceeds at a very low rate. Therefore, it is necessary to purify the solution from silica.

There is a known method [17] in which some silica is removed from high-modulus aluminate solutions by decreasing the solubility of sodium aluminosilicate in relatively low-concentration Na_2O_c solutions. For this purpose, the solutions are diluted to $300 \text{ g/dm}^3 \text{ Na}_2\text{O}$ and stirred at 90°C for 2–3 h. The silica in this case precipitates in the form of sodium aluminosilicate. Therefore, the silicon modulus of the μ_{Si} solution (mass ratio of $\text{Al}_2\text{O}_3/\text{SiO}_2$) increases from 7–10 to 50–100. A similar method combining desiliconization with sludge separation was recommended in the literature [18]. In the process of bauxite sintering, CaO is added to bind SiO_2 and obtain 2CaO-SiO_2 [[19], [20]]. CaO is not used during the hydrochemical processing of high-modulus bauxite with a silicon modulus exceeding 7, since the SiO_2 content is low enough, and the losses are insignificant [21].

The deep desiliconization of aluminate solutions with silicon moduli (μ_{Si}) of 8–12 can be performed by binding silica to hydrogenates in the polythermal regime. Studies on the conditions allowing the decomposition of unsilicified aluminate solutions have not been carried out.

The distribution of Na, Al, and Si between the solid and liquid phases depends on the chemical compositions of the silicate and aluminate solutions [22].

The influence of SiO_2 on the rate of decomposition of aluminate solutions and the formation of hydroaluminate crystals under different conditions has not been sufficiently clarified, and the mechanism of silica action in this process remains unclear. This work aims at studying the influence of silica on the production of sodium hydroaluminate.

Materials and methods

The kaolin sample from Alexeevskoe deposit was used in this study. The sample was determined to contain Al_2O_3 35.6%; SiO_2 43.2% and other components 21.2%, the silicon modulus was 0.6. The kaolin sample was submitted to autoclave leaching using $240 \text{ g/dm}^3 \text{ Na}_2\text{O}$ for 90 min at 240°C . Obtained solution was evaporated to a concentration of 540 g/dm^3 of Na_2O in total; 115.5 g/dm^3 of Al_2O_3 ; and 0.609 g/dm^3 of SiO_2 with a μ_{Si} of 7.5–8.5. Silica was added as an alkaline sodium silicate solution containing 550 g/dm^3 of $\text{Na}_2\text{O}_{\text{cu}}$ and 200 g/dm^3 of SiO_2 . The solution with added silicate solution was heated until it boiled and then cooled to the experimental temperature.

Leaching was carried out in a thermostat with a stirrer.

The resulting precipitates were X-ray amorphous, and the phase composition could not be identified by X-ray diffraction (XRD) analysis. Consequently, we indirectly determined their compositions from the values of μ_{Si} and α_k (caustic modulus).

Experimental part

The decomposition kinetics of solutions with a Na_2O content of 540 g/dm^3 ($\alpha_k=8$) in the presence of different amounts of silica are shown in Figure 1. These experiments were carried out without inoculum since it was possible to trace the influence of silica on nucleation in this case. Stirring was performed at a speed of 15 rpm.

The obtained data show that small amounts of silica (from 0.3 to 0.6 g/dm^3) affect the initial period of crystallization. In this case, the average rate of the process decreases sharply, especially at the maximum point, but the content of SiO_2 in solution practically does not change. The effect of 0.8 g/dm^3 of SiO_2 ($\mu_{\text{Si}} = 136$) is more noticeable than that of a

smaller amount. At a SiO_2 content of 1.7 g/dm^3 ($\mu_{\text{Si}} = 64$, curve 4), the rate decreases throughout the process. After 5 h of stirring, the Al_2O_3 content in the solution is 21.4 g/dm^3 instead of 14.5 g/dm^3 at an SiO_2 content of 0.3 g/dm^3 . However, this amount of SiO_2 has a negligible effect on the alumina yield (80–86%). With the addition of 2.6 g/dm^3 of SiO_2 ($\mu_{\text{Si}} = 40$), an induction period appears, and the maximum rate of the is approximately halved; however, after 3 h, approximately 70% of the Al_2O_3 is released into the solid phase. Thus, even without inoculum, sodium hydroaluminate can be isolated from solutions containing 540 g/dm^3 of Na_2O with a silicon modulus of 40 at a satisfactory rate. It is known [23] that for solutions containing 500 g/dm^3 of Na_2O_k ($\alpha_k=7$) at $\mu_{\text{Si}} = 50$ in the presence of an inoculum for 7 h equivalent to either 2.5% of the solution weight or 33% of the solution Al_2O_3 content precipitates only 50% of the Al_2O_3 , and only at $\mu_{\text{Si}} = 100$ is the yield 73%.

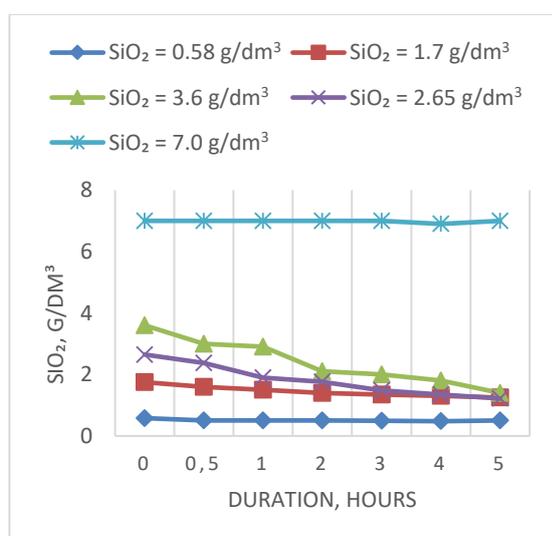
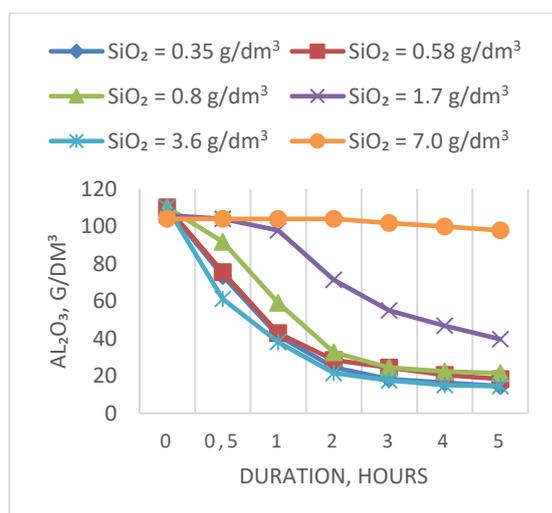


Figure 1 - Kinetic curves of the decomposition of aluminate solutions with a Na_2O_k concentration of 540 g/dm^3 depending on the SiO_2 content

A higher Al_2O_3 content ($\mu_{\text{Si}} = 30$) in the solution with a Na_2O_k concentration of 540 g/dm^3 considerably slows the process. Even with stirring for 3 d (10 g/dm^3 of SiO_2), the concentration of Al_2O_3 in the solution is 48 g/dm^3 . The SiO_2 content in the solution first gradually and then sharply decreases in the first hour of stirring. The silicon modulus of the solutions with low SiO_2 contents ($0.3\text{--}0.9 \text{ g/dm}^3$) gradually decreases, and at $2.6\text{--}3.6 \text{ g/dm}^3$, the silicon modulus peaks, which agrees with the data in the present work. The final μ_{Si} at an SiO_2 content of 0.6 g/dm^3 in the initial solution is low and equals approximately 17 at 1.7 g/dm^3 . The silicon modulus of the mother solution is low because silica is practically not removed from it, and a small amount of aluminium oxide is present. For the silica-rich solutions, the μ_{Si} after 5 h of stirring is 25–27.

The effect of silica on the average rate of decomposition of the solutions is similar to the decrease in the degree of supersaturation [24]. The solubility of aluminosilicate in aluminate solutions increases with increasing Na_2O_k concentration, while the solubility of sodium hydroaluminate decreases. Under these conditions, we attempt to isolate aluminate from high-silica aluminate solutions.

While decomposing aluminate solutions with a Na_2O_k content of 590 g/dm^3 at $\alpha_k = 8$ and a temperature of $45 \text{ }^\circ\text{C}$ (Figure 2), the presence of 2.3 g/dm^3 of SiO_2 ($\mu_{\text{Si}} = 47$) does not substantially affect the crystallization rate of sodium hydroaluminate. At a SiO_2 content of 4.6 g/dm^3 ($\mu_{\text{Si}} = 27$), decomposition proceeds at a noticeably reduced rate, but after 5 h of stirring, 77% of the Al_2O_3 is released into the solid phase. In the presence of 6.5 g/dm^3 of SiO_2 in the solution, 70% of the Al_2O_3 precipitates during this time interval. Thus, the decomposition process proceeds at a satisfactory rate, even when the silicon modulus of the solution is 20.

The SiO_2 content decreases during the first 2 h of stirring and remains almost unchanged afterward. The silica modulus of the solution gradually decreases by a factor of 2 after 5 h.

Most of the silica (>50%) remains in the solution, and its amount does not decrease when stirred with sodium hydroaluminate precipitate; this result is in good agreement with previously obtained data [3].

Precipitation from solutions with 540 g/dm^3 of Na_2O_k (stirring at 15 rpm) has a caustic modulus in the range of 1.8–2.0. For solutions with a Na_2O_k content of 590 g/dm^3 , the caustic modulus is 1.7–1.9. In the latter case, after stirring at $80\text{--}90 \text{ }^\circ\text{C}$ for 2 h and cooling to $45 \text{ }^\circ\text{C}$, precipitates with a low alkali content ($\alpha_k = 1.5\text{--}1.6$) are obtained.

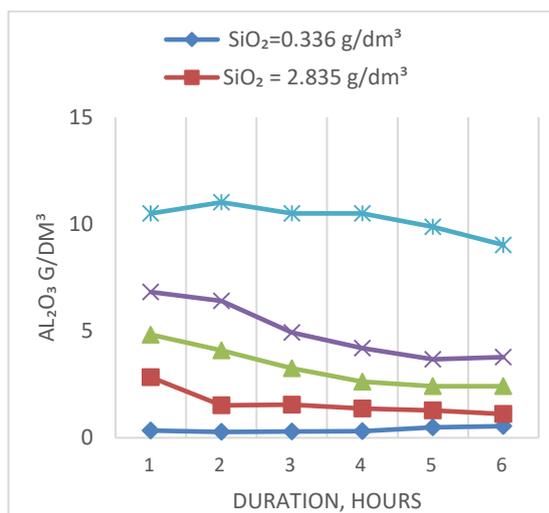


Figure 2 - Kinetic curves of decomposition of aluminate solutions with Na₂O = 590 g/dm³ in the presence of different amounts of silica

Microscopic examination of the solid phases shows that sodium hydroaluminat is represented by large crystals (200–1000 microns), and the crystals are not faceted. This morphology complicates the filtration process (Figures 3 and 4).

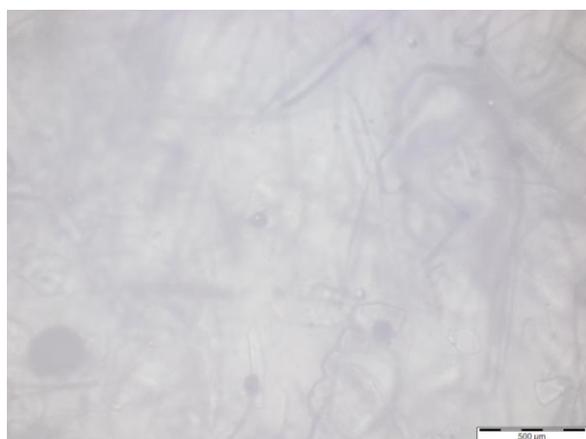


Figure 3 - Sodium hydroaluminat precipitate, 40×

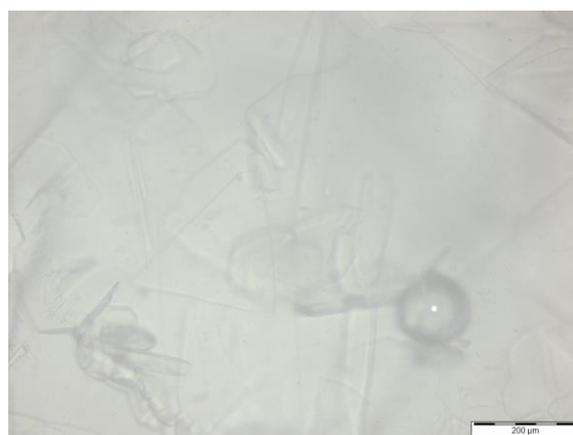


Figure 4 - Precipitate of sodium hydroaluminat, 100×

If the influence of silica on the crystallization of sodium hydroaluminat is reduced to adsorption, it is likely that introducing additional inoculum into the solution can remove SiO₂, thus accelerating the crystallization process [24].

According to the literature [25], when using a dispersed inoculum, the degree of maximum decomposition is 73.9% after 7 h.

The results of studying the decomposition of aluminate solutions containing silica in the presence of inoculum are given below. The size of the initial aggregates ranges from 200 to 1000 microns.

The solutions are stirred with the inoculum at a speed of 85 rpm. The obtained data are shown in Figure 5. In the presence of inoculum equivalent to 16% of the Al₂O₃ content in an aluminate solution containing 10 g/L of SiO₂ ($\mu_{Si} = 10$), no induction period is observed on the kinetic curves. After 1 h, approximately 15% of the Al₂O₃ is released, after which the crystallization is very slow (3% Al₂O₃ in 4 h). When 16% of the inoculum is added to the solution after stirring for 1–2 h, the curve again shows a jump. After 4 h, 35% of the Al₂O₃ is released. Thus, each new portion of inoculum (16%) contributes to the crystallization of 18% Al₂O₃; specifically, to isolate 70% of sodium hydroaluminat, approximately 70% of the inoculum is needed.

A similar pattern is observed in the presence of 5.0 and 3.3 g/L of SiO₂ ($\mu_{Si} = 20$ and 32). The alumina yields after are 60 and 75% in the presence of 5.0 and 3.3 g/L of SiO₂, respectively, which are significantly greater than those previously obtained in the presence of 37.5% inoculum (probably coarse crystalline). These data show the principal possibility of sodium hydroaluminat extraction from nondispersed solutions. However, the precipitates formed in this case are finely dispersed and contain a large amount of alkali ($\alpha_k = 2-3$).

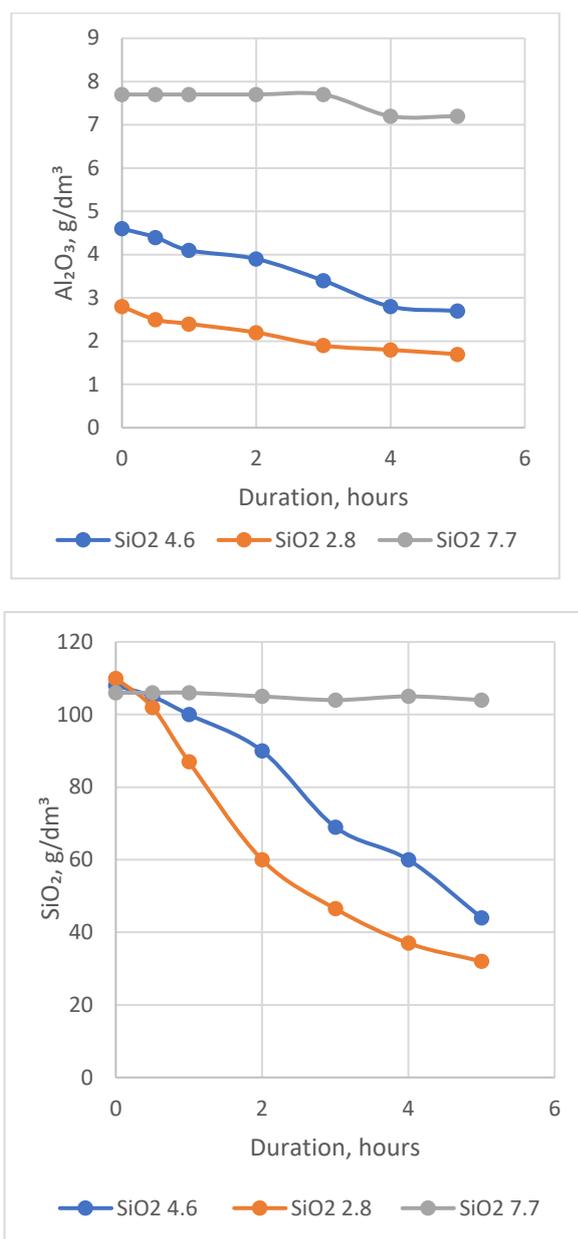


Figure 5 - Kinetic curves of aluminate solution decomposition in the presence of inoculum

Microscopic examination of many precipitates shows that in the presence of 2–3 g/L of SiO₂, monosodium hydroaluminate crystals grow in the form of thin plates with pelletized tops. The dispersibility of the precipitates is related to the conditions of production, depending on the concentrations of Na₂O and Al₂O₃, the temperature, and the method and speed of mixing, which should be the same as those in pure solutions to ensure that the difference in α_k is nonsignificant. Sometimes, without inoculum, relatively large crystals grow with increasing SiO₂ content in solution. Small crystalline precipitates (1–3 μ m) are difficult to study by the crystal–optical method. Therefore, for this purpose, large crystals of sodium hydroaluminate (reaching

200 μ) are obtained by stirring the solutions in rotating nickel autoclaves at temperatures of 45 and 85°. The Na₂O_k contents of the solutions are 532 and 620 g/L, and the caustic moduli are 4, 8 and 12. Silica is added at a concentration of 10 g/L. The appearance of hydroaluminate crystals obtained in the presence of silica is very different from that of crystals precipitated from pure solutions. Thus, thin, rounded flakes form from solutions with $\alpha_k = 8$ –12 (532 and 620 g/L of Na₂O) at 45° (Figure 6). The cross sections are shaped like elongated lenticels with high interference colours. From solutions with $\alpha_k = 4$ and Na₂O concentrations of 532 and 620 g/L (45 and 85 °C), various oval and rounded aggregates are formed (Figure 6 (c)). At $\alpha_k = 12$ (620 g/L) and $\alpha_k = 8$ (530 g/L), round lamellar crystals grow, with similar small formations appearing on the surface (Figure 6 (2)). Without silica, 8-cornered laminae form under these conditions. From a solution with $\alpha_k = 8$ (620 g/L) at 85 °C, crystals (Figure 6 (e)) precipitate (Figure 6 (2)), which have a pronounced stepped surface, sometimes in the form of typical helical dislocations. From pure solutions, well-bounded 12-angular plates are formed in this process. When aluminate solutions with $\alpha_k = 8$ and a Na₂O concentration of 620 g/L (45 °C) decompose, clear crystals precipitate instead of forming plates, as shown in Figure 6 (e). No sodium aluminosilicate crystals are observed in the precipitate. There is a tendency for the light refractive indices of sodium hydroaluminate to decrease. For precipitates obtained at 45°, mainly the ray of ordinary light (No; 0.005–0.007 decreases). Crystals formed in the presence of silica at 85° have a ray of extraordinary light (Ne) with a lower value than that of pure precipitates.

In the presence of 7.8 g/L of SiO₂ during 5 h of contact, the content of Al₂O₃ in the solution almost does not change, and despite the high silica content, the latter is not deposited on the surface of the seed in appreciable amounts.

At a SiO₂ content of 4.6–2.8 g/L in the initial solutions, the crystallization process proceeds at a relatively high rate. The amount of silica in the solution gradually decreases.

If sodium hydroaluminate does not crystallize, the silica is not deposited on the inoculum in appreciable quantities. In general, hydroaluminate and silica are deposited together, which indicates is a relationship between Al₂O₃ and SiO₂ in the solution.

Results and discussion

The effect of silica on the stability of aluminate solutions and the appearance of precipitating sodium hydroaluminate crystals is noted. The stability of aluminate solutions at rest in the presence of silica increases dramatically.

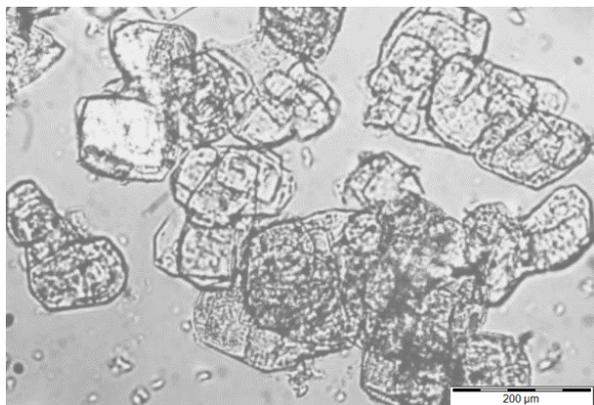


Figure 6 - Sodium hydroaluminate precipitate in the presence of SiO_2 , 100 \times

When standing solutions with a Na_2O content of 500–520 g/L ($\alpha_k = 8$) in the presence of 0.2 g/L of SiO_2 , sodium hydroaluminate precipitates after 1–2 d. At $\text{SiO}_2 = 3$ g/L, the solution does not decompose within 7 d, and at 7–10 g/L, the solution is stable for approximately one month. With the mechanical stirring of these solutions, an induction period occurs, and the amount of SiO_2 in the solution increases. During the crystallization of sodium aluminate in the presence of a large amount of inoculum with different dispersity degrees, no preferential deposition of silica can be observed. In general, the sodium aluminate is released into the solid phase only during the simultaneous crystallization of sodium hydroaluminate. With the introduction of the active inoculum, a new portion of the hydroaluminate crystallizes with the silica. These phenomena lead to the concept that the effect of SiO_2 is a consequence of the poisoning of active centres on the seed crystals and acts on the state of sodium hydroaluminate in solution. However, the effect of silica is not limited to the formation of sodium aluminosilicate because it binds a relatively small part of dissolved aluminium, thus slightly decreasing the degree of supersaturation of solutions by 0.2–0.5 at $C_p - S_n / S_n = 5.5-7$.

The increase in the degree of saturation at an unchanged SiO_2 content in solution is similar to the decrease in silica concentration. In solutions with concentrations of Na_2O (590 g/L), the solubility of aluminosilicate increases, and as a result, its influence on the crystallization rate of sodium hydroaluminate decreases. The interaction of sodium silicate with surface molecules of aluminate and the adsorption of aluminosilicate from the solution, occurs at the sites with the highest surface energy, which include the tops of crystals.

Further deposition of hydroaluminate molecules occurs mainly on the faces; thus, the crystals have a rounded shape. The formed crystals have a mosaic structure, and the entrained aluminosilicate is distributed in the form of single molecules or small groupings on the surface of the cells since X-ray analysis does not reveal any change in the interplanar distances. Moreover, free aluminosilicate is not detected in the sediments.

The stability of aluminate solutions at rest in the presence of silica increases dramatically. For standing solutions with a Na_2O content of 500–520 g/L ($\alpha_k = 8$) in the presence of 0.2 g/L of SiO_2 , sodium hydroaluminate precipitates in 1–2 d. At $\text{SiO}_2 = 3$ g/L, the solution does not decompose within 7 d, and at 7–10 g/L, the solution is stable for approximately one month.

Conclusions

Different crystallization conditions for sodium hydroaluminate have been investigated. Silica slows the crystallization process of sodium hydroaluminate, affecting both the nucleation and crystal growth rates. The effect of SiO_2 on the rate of decomposition is similar to the reduction in the degree of supersaturation.

At a Na_2O concentration = 540 g/L ($\alpha_k = 8$), the crystallization of sodium hydroaluminate even without inoculum proceeds at a satisfactory rate (70% Al_2O_3 in 5 h) at $\mu_{\text{Si}} = 40$. When the Na_2O content increases to 590 g/L, the solutions with $\mu_{\text{Si}} = 19$ decompose quickly. Moreover, approximately half of the SiO_2 remains in the liquid phase.

The possibility of crystallization of sodium hydroaluminate from nonsilica aluminate solutions (SiO_2 content reaching 10 g/L, Na_2O content of 500–530 g/L, and $\alpha_k = 8$) by introducing a large amount of inoculum has been established.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

review and editing. **G. Ruzakhunova:** formal analysis, **S. Tugambay:** formal analysis.

Author Contributions CRediT: **R. Abdulvaliyev:**

Conceptualization, writing original draft preparation
S. Gladyshev: Methodology, writing – review and editing
N. Akhmediyeva: Data curation, writing –

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Кремнийдің натрий гидроалюминатының кристалдануына әсері

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Сараптамадан өтті: 3 мамыр 2024
Қабылданды: 27 мамыр 2024

ТҮЙІНДЕМЕ

Жоғары концентрлі алюминат ерітінділерінен алюминий алу үшін құрамында алюминий оксиді бар шикізатты өңдеу кезінде натрий гидроалюминатының кристалдануымен ыдырау әдісі қолданылады. Al_2O_3 -тің қатты фазаға ықтимал шығымдылығын есептеу үшін бастапқы ерітінділердің құрамына және процестің шарттарына байланысты натрий гидроалюминатының кристалдану жылдамдығын білу қажет. Ерітіндіде кремнеземнің болуы натрий алюминатының кристалдану процесі үшін үлкен маңызға ие. Мақалада алюминат ерітінділерінің тұрақтылығын зерттеу нәтижелері берілген. SiO_2 мөлшерінің Na_2O_k концентрациясы 540 г/дм^3 және 590 г/дм^3 болатын алюминат ерітінділерінің бұрын дайындалған натрий гидроалюминат түріндегі (затравканы) қосып және қоспай алюминат ерітінділерінің ыдырауына әсері зерттелді. Кремнеземнің түзілетін натрий гидроалюминатының кристалдарының сыртқы түріне әсері қарастырылды. Алюминат ерітінділерінің кремнеземнің қатысында тыныштықтағы тұрақтылығының күрт жоғарылауы анықталды. Натрий гидроалюминатының кристалдануының әртүрлі шарттары зерттелді. Кремний диоксиді натрий гидроалюминатының кристалдану процесін бәсеңдететіні, эмбрион түзілуіне де, кристалдардың өсу жылдамдығына да әсер ететіні көрсетілген. SiO_2 -нің ыдырау жылдамдығына әсері қанығу дәрежесінің төмендеуіне ұқсас. Кремнийсізделмеген алюминат ерітінділерінен натрий гидроалюминатының кристалдануының негізгі мүмкіндігі көбірек (затравка) енгізу арқылы анықталды.

Түйін сөздер: алюминат ерітіндісі, кремний диоксиді, натрий гидроалюминаты.

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Влияние кремния на кристаллизацию гидроалюмината натрия

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АННОТАЦИЯ

При переработке глиноземсодержащего сырья для извлечения алюминия из высококонцентрированных алюминатных растворов применяется метод разложения с кристаллизацией гидроалюмината натрия. Чтобы рассчитать вероятный выход Al_2O_3 в твердую фазу, необходимо знать скорость кристаллизации гидроалюмината натрия в зависимости от состава исходных растворов и условий проведения процесса. Большое значение для процесса кристаллизации алюмината натрия имеет наличие в растворе кремнезема. В статье представлены результаты исследования устойчивости алюминатных растворов. Было изучено влияние содержания SiO_2 на разложение алюминатных растворов с концентрацией $Na_2O_{к} 540 \text{ г/дм}^3$ и 590 г/дм^3 с добавлением затравки в виде предварительно полученного гидроалюмината натрия и без него. Было рассмотрено влияние кремнезема на облик выпадающих кристаллов гидроалюмината натрия. Установлено резкое повышение устойчивости алюминатных растворов в покое в присутствии кремнезема. Были исследованы различные условия кристаллизации гидроалюмината натрия. Показано, что кремнезем замедляет процесс кристаллизации гидроалюмината натрия, действуя и на зародышеобразование и скорость роста кристаллов. Действие SiO_2 на скорость разложения аналогично снижению степени пересыщения. Установлена принципиальная возможность кристаллизации гидроалюмината натрия из необескременных алюминатных растворов путем введения большего количества затравки.

Ключевые слова: алюминатный раствор, кремнезем, гидроалюминат натрия.

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