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S.M. Logvinkov^a, *O.M. Borysenko*^b, *A.A. Ivashura*^a, *H.M. Shabanova*^b, *V.M. Shumejko*^b,
A.M. Korohodska^b, *N.S. Tsapko*^c

SOLID-STATE EXCHANGE REACTIONS DURING SINTERING OF DISPERSED ALUMINA

^a Simon Kuznets Kharkiv National University of Economics, Kharkiv, Ukraine

^b National Technical University «Kharkiv Polytechnic Institute», Kharkiv, Ukraine

^c Scientific-Research Institution «Ukrainian Scientific-Research Institute of Ecological Problems», Kharkiv, Ukraine

In this article, the mechanism of sintering of Al_2O_3 in the presence of small amounts of Na_2O and CaO was investigated. Based on the results of the electron microscopy, the granulometry and morphological features of the particles of the studied alumina were established. The uniform nature of the distribution of sodium-containing phases was revealed, in contrast to silicon-containing ones, and the dislocation of submicron particles from calcium-containing phases was determined mainly on the basal planes of relatively large corundum particles. It was shown that such an arrangement of calcium-containing phases promotes the formation of a dense layered microstructure during sintering, especially in the presence of β -alumina. The general pattern of the branched mechanism of the reaction phase formation during the sintering of the compositions in the Na_2O – CaO – Al_2O_3 system was illustrated by a diagram explaining the trend of physicochemical processes and the feasibility of using specific types of dispersed alumina for technologies of corundum products and refractory concretes with different contents of aluminous cements.

Keywords: tabular alumina, reactive alumina, calcium aluminates, corundum, β -alumina.

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Introduction

Over the past 30 years, modern types of alumina, in particular, tabular, dispersive, calcined, alkaline and reactive ones have been used in increasing quantities in the world practice for the production of the corundum-based refractories and ceramic composite materials [1–4]. The leading producers of such alumina (primarily Germany and Hungary) ensure stable quality and the expansion of the range of their products, focusing on various fields of application and technological variations in the production of different products or unshaped masses such as slip casting, semi-dry pressing, molding on thermoplastic and thermosetting binders, dry mixtures for refractory concretes, shotcrete masses, self-spreading and vibroformed compositions.

Tabular or lamellar alumina has been produced in Western countries for a long time and the production method differs from the traditional one used by post-Soviet countries. Such alumina got its name due to the availability of a great number of thin, plane-parallel particles with regular hexagonal faceting, peculiar for the polymorphic modification of α - Al_2O_3 (corundum). The noted morphological feature ensures the orientation of the particles by the basal planes perpendicular to the forming force, layering and producing a dense semi-finished product or concrete. Currently, tabular alumina of a various granulometric composition (for example, grade T 60/64) is produced on industrial scale, and due to its high quality, a constant composition, an affordable price category and mandatory delivery times it ensures its wide

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S.M. Logvinkov, O.M. Borysenko, A.A. Ivashura, H.M. Shabanova, V.M. Shumejko, A.M. Korohodska, N.S. Tsapko

application for refractory plants in Ukraine to manufacture various products and unshaped masses for accountable purposes. The phase composition of tabular alumina also contributes thereto and it is mainly represented by corundum, β -alumina (previously considered as a solid solution of Na_2O in Al_2O_3 , later attributed to the $\text{Na}_2\text{O}\cdot 12\text{Al}_2\text{O}_3$ compound, and at present it has been established that it belongs to the $\text{Na}_2\text{O}\cdot 11\text{Al}_2\text{O}_3$ compound) and a small amount of admixture phases with iron and silicon oxides (total up to 0.6–0.7 wt.%), magnesium and calcium oxides being actually unavailable.

Other types of aforementioned alumina are often called dispersed because of their small particle size; these are not produced in granular form and perform auxiliary functions during the manufacture of products or unshaped masses. Dispersing alumina (in particular, ADS-1 and ADW-1, for summer and winter conditions, respectively) are focused on increasing the spreadability and reducing the water demand of concrete mixtures due to the presence of organic superplasticizers of a new generation in their composition (based as a rule on polycarboxylates that are the polyesters of carboxylic acids), as evidenced by the high values of their mass loss during calcination: 19 to 21 wt.% [5]. In addition, the presence of B_2O_3 is identified in their composition that contributes to a low-temperature sintering in parallel with the dehydration of the crystalline hydrate phases of the cement stone, which compensates for the decrease in the strength of concrete in the range of 800 to 1000°C. The oxide composition of these types of alumina contains: SiO_2 (up to 0.5 wt.%), oxides of alkaline earth metals (up to 0.2 wt.%), Na_2O (up to 0.03 wt.%) and very small amounts of Fe_2O_3 (up to 0.01 wt.%). Reactive and calcined types of alumina (in particular, CT 3000 SG, «Salox ALO DN-23» and others [6,7]) are also highly pure in terms of Al_2O_3 content (>99.0 wt.%); these have a low iron content (usually 0.02–0.04 wt.%); the amount of SiO_2 is in the range of 0.04–0.05 wt.%; the content of Na_2O is usually about 0.14 and 0.3 wt.%, respectively; the availability of alkaline earth oxides varies in the range of about 0.03 and 0.4 wt.%, respectively. Other oxides are not indicated in the technical specifications from the supplier, but small amounts of TiO_2 are recorded during the incoming quality control, in particular, in ST 3000 SG alumina these are present in amount of 0.01 wt.%. Obviously, small amounts, including those not indicated in the supplier's certificates, determine the main functionality of such alumina. Accordingly, in the factory conditions, the problems may arise in identifying low amounts of admixtures in the oxide composition and it makes it difficult to predict the

properties of new compositions of refractories and composite ceramics due to the uncertainty of the participation of admixtures in the sintering mechanism.

The purpose of this research was to analyze the mechanism of the Al_2O_3 sintering in the presence of small amounts of Na_2O and CaO based on the results of studies of the «Salox ALO DN-23» alumina.

Materials and methods

Reactive alumina, brand «Salox ALO DN-23», manufactured by «MAL Hungaricum Aluminum Production and Trade Limited Company by Shares» (Hungary) is characterized according to the specifications by the following content (wt.%): Al_2O_3 not less than 99.5; SiO_2 no more than 0.04; Fe_2O_3 no more than 0.04; Na_2O no more than 0.3; and CaO no more than 0.03. Its specific surface area varies in the range of 3.0 to 4.5 m^2/g , the average particle diameter is the range of 1.2 to 1.8 μm , and 90% of the particles are within the diameter size range of 3 to 5 μm .

Studies of alumina were carried out using the methods of the optical microscopy (polarization microscope MIN-8 using standard immersion liquids IZH-1), infrared spectroscopy (Avatar spectrometer), X-ray fluorescence analysis (Expert 3L spectrometer), and scanning electron microscopy (JSM-6390LV with the energy dispersive Aztech Energy spectrometer and X-max 50 detector for the microprobe quantitative determination of chemical elements).

Analysis of the mechanism of alumina sintering included the simulation of the solid-phase exchange reactions [7] in accordance with the subsolidus structure of the state diagram of the $\text{Na}_2\text{O}\text{--}\text{CaO}\text{--}\text{Al}_2\text{O}_3$ system that is the physicochemical basis of phase equilibria in the given system. When simulating the solid-phase exchange reactions, the fundamental Gibbs phase rule was taken into account, according to which five phases can simultaneously coexist in a three-component oxide system under certain external parameters. Accordingly, not only reactions of the «1=3» or «2=2» type (one or two initial phases and three or two phases in the reaction products) were considered, but also those of the «2=3» type (two initial phases and three phases in the interaction products, as well as the inverse type «3=2»).

Results and discussion

The investigated alumina is a white powder of a very high degree of dispersity, clumping into fragile aggregates and sticking to the walls of a plastic sampler. Under the MIN-8 microscope, individual particles larger than 4–5 μm in diameter were not observed; and a certain degree of particle orientation was noted in aggregates (about 10–15 μm). This determines simultaneous or sectorial extinction (when the polarizer

rotates). The average light refraction index is more than 1.734 and it is indicative of the predominance of the polymorphic α - Al_2O_3 modification in the sample.

According to the results of infrared spectroscopy, no maxima typical of the $-\text{OH}$ group were noted, and it is indicative of the unavailability of aluminum hydrates. The spectrum is represented by a flat and monotonically ascending curve with the presence of a single segment of a sharp rise near the limiting frequency of 4000 cm^{-1} that is related to the beginning of the development of a maximum corresponding to the stretching vibrations of the $\text{Al}-\text{O}$ bond in Al_2O_3 . The results of the optical and infrared spectroscopies confirm the characteristics of alumina declared by the manufacturer.

During the X-ray fluorescence analysis, the sensitivity of the device was deliberately abated for the main element (Al) in order to increase the resolution with respect to the admixtures. Therefore, the quantitative elemental composition of the alumina sample is not considered as an adequate result, and the following admixtures were identified in the qualitative composition: silicon, sulfur, copper, and zinc. At the same time, the limited capabilities of the device in detecting light elements, up to magnesium in the periodic table, were also taken into account. The reasons for the absence of Ca and Fe in the alumina composition can be connected with the locality of the analysis and low alumina content. Silicon, according to the specifications for alumina, is declared as SiO_2 and its identification in the sample was expected. The admixture of zinc can be of a targeted nature, since zinc oxide is capable of low-temperature interaction with alumina up to the formation of the zinc spinel ZnAl_2O_4 , garnet.

Such a reaction can initiate low-temperature sintering and maintain volume constancy, since the synthesis of garnet proceeds with an increase in volume. Sulfur can be an admixture introduced into alumina by flue gases during its production. At the same time, the target nature of this admixture cannot be ruled out due to the microadditives of calcium and aluminum sulfates or acid washing to remove undesirable admixtures. In addition, sulfur could enter alumina at the milling stage, since organic salts of sulfonic acids increase the efficiency of this energy-intensive technological operation. Copper, most likely, should be considered as an accidental admixture with an unclear source of entry into alumina and questionable technological functionality. During electron microscopy studies, three regions of the powder sample were selected for the microprobe analysis, in

which a significant number of particles were observed that were differentiated by color from the main substance, i.e., these were enriched with admixtures. Accordingly, quantitative results were obtained with a deviation from the declared content of chemical elements in alumina in terms of oxides (wt.%): Al_2O_3 97.31–98.79; Na_2O 1.08–1.90; and SiO_2 up to 1.61. These deviations do not refute the correctness of the mean values of the content of the noted oxides. The emphasis should also be placed on the relatively uniform distribution of sodium in the alumina sample and, on the contrary, the local and uneven distribution of silicon. Moreover, silicon in alumina is not contained in the form of aluminosilicate phases, but in an individual form, most likely in the form of silica. Figure 1a shows the appearance of the studied alumina dispersed in isopropyl alcohol on an aluminum substrate.

The particles of alumina of a round-polygonal morphology, the particles larger than $5\text{ }\mu\text{m}$ are virtually absent; a predominant number of particles have a diameter less than $2\text{ }\mu\text{m}$. Along the perimeter of the particles, lighter rims are observed due to the effect of misorientation of the electron flux at geometrically imperfect grain boundaries. At a higher resolution (Fig. 1b), sharp-angled particles are also observed, as well as the admixture particles that are different in color and have a size of up to $2\text{ }\mu\text{m}$ (below and to the left of the center in Fig. 1b). Accumulations of such particles were observed in some places of the powder sample (Fig. 1c). Only individual large particles retain the elements of regular crystal faceting, in particular, hexagonal faceting, as the α - Al_2O_3 particle in Fig. 1g with a dihedral angle of 120° . Submicron particles of admixture phases, probably calcium-containing, are observed on the surface of this particle. The dislocation of these phases on the basal planes of lamellar α - Al_2O_3 particles in the presence of β -alumina promotes the formation of a dense structure of the material from the studied alumina during sintering.

The presence of CaO in the studied alumina defines the possibility of direct reaction with corundum even during the alumina production. SA is considered to be the primary product of solid-phase reactions (C, A, and N stand for CaO, Al_2O_3 , and Na_2O , respectively), and other compounds are formed already with its participation, depending on the stoichiometry. Under conditions of excess Al_2O_3 , the general direction of the synthesized calcium aluminates can be displayed as follows: $\text{C}_3\text{A} \rightarrow \text{C}_{12}\text{A}_7 \rightarrow \text{CA} \rightarrow \text{CA}_2 \rightarrow \text{CA}_6$. Due to the incompleteness of the solid-phase reactions, any of the calcium aluminates may be present in the alumina.

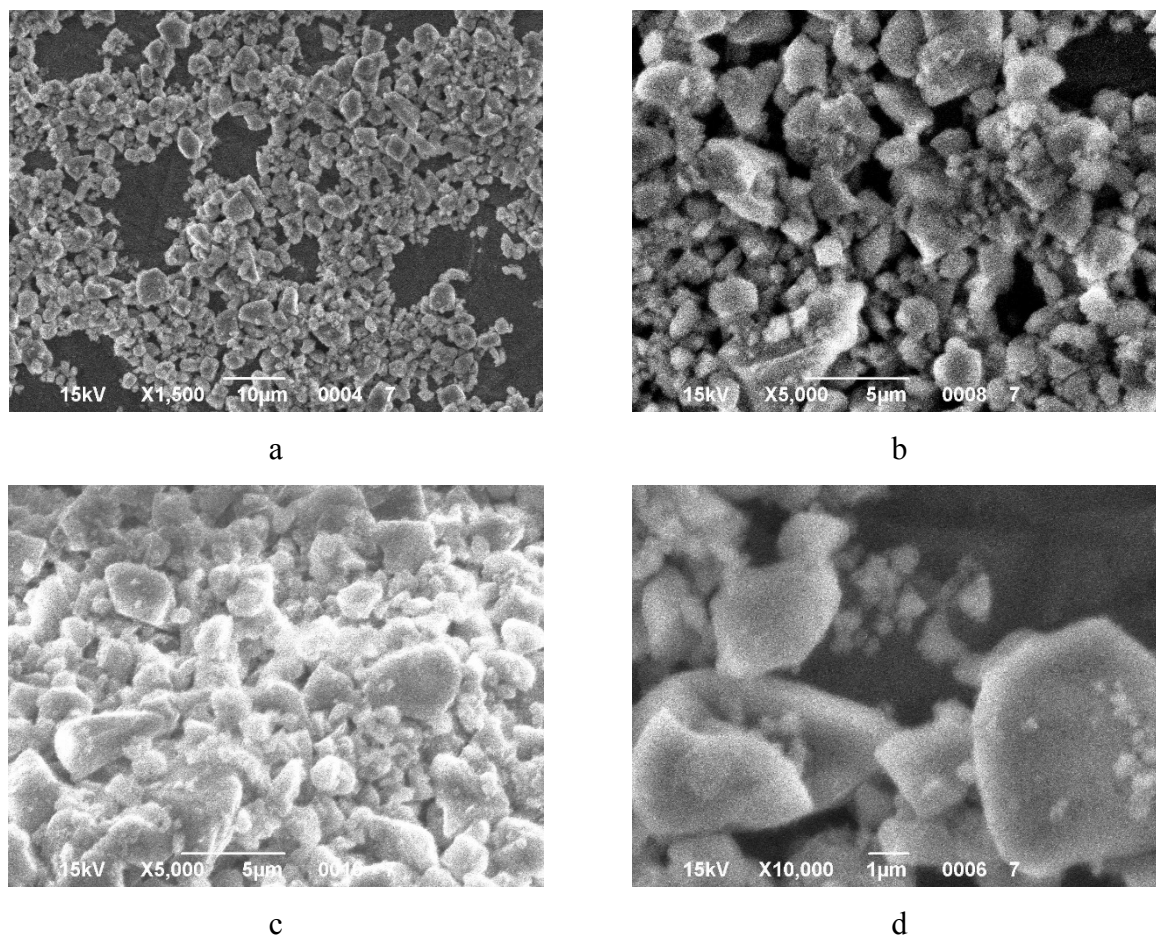
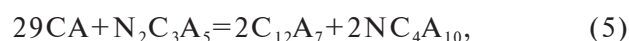
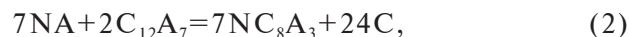


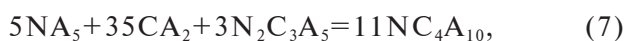
Fig. 1. SEM micrographs of the powder sample of the studied alumina

It should be noted that the presence of sulfates (sulfur was identified by X-ray fluorescence analysis) is possible and the formation of calcium sulfoaluminate, in particular, the synthesis of $\text{Ca}_4\text{Al}_6\text{SO}_{16}$ (or $3(\text{CaO}\cdot\text{Al}_2\text{O}_3)\cdot\text{CaSO}_4$) directly from CaSO_4 and calcium monoaluminate is accompanied by a significant increase in volume (by 13%). This can have a positive effect on the shrinkage compensation during alumina sintering reducing the total open porosity and pore size.

Sodium aluminates with a high content of Na_2O (N_5A , N_{17}A_5 , and N_7A_5) disproportionate at a temperature of up to 980°C and for the solid-phase synthesis it is enough to take into account the reaction with the formation of $\text{NA}\rightarrow\text{NA}_5$ (the so-called β'' -alumina) $\rightarrow\text{NA}_{11}$. These sodium aluminates can be present in the studied alumina, taking into account the fact that NA melts congruently at 1887°C , NA_5 disproportionates at only 30°C below the eutectic (1580°C), and NA_{11} melts incongruently at about 2000°C . At the same time, the joint presence of calcium

and sodium aluminates is not limited to sequential reaction interaction and the mechanism of reaction sintering is significantly branched due to the existence of triple oxide compounds: (I) NC_8A_3 , (II) $\text{N}_2\text{C}_3\text{A}_5$, and (III) NC_4A_{10} . Taking into account the triangulation of the $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3$ in the composition of the studied alumina, the development of solid-phase reactions is thermodynamically beneficial:





All of the above-mentioned compounds are involved in the reactions that cover half of the entire concentration region of the $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3$ system adjacent to the $\text{CaO}-\text{Al}_2\text{O}_3$ side. The general trend of the phase formation during the reaction sintering of the studied alumina will be more visual, if, on the diagram of the subsolidus structure of a part of the $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3$ system, arrows on the connodes indicate the direction from the initial compounds of reactions (1)–(11) to the final ones (Fig. 2).

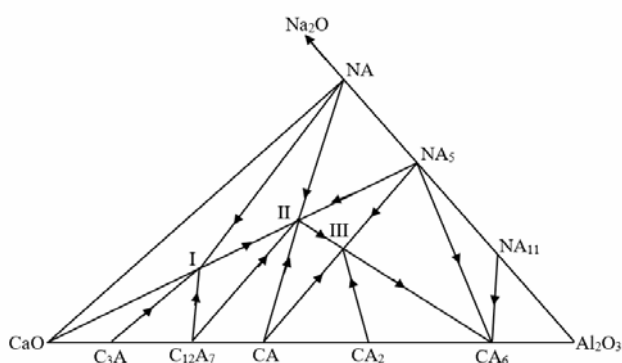


Fig. 2. Trend in phase formation during reaction sintering of alumina

Figure 2 shows that the solid-phase interaction between sodium and calcium aluminates is realized through the sequential formation of ternary compounds: $\text{NC}_8\text{A}_3 \rightarrow \text{N}_2\text{C}_3\text{A}_5 \rightarrow \text{NC}_4\text{A}_{10}$ and ends on the final product, i.e. calcium hexaaluminate that coexists with β -alumina and corundum in the high-alumina concentration region of the $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3$ system. In the analyzed alumina, the solid-phase interaction is limited by the limited content of CaO capable of a significant limitation of refractory properties due to the low temperature of the eutectic (1455°C) between C_3A and C_{12}A_7 .

Obviously, the most effective reaction sintering of such alumina will be observed when it is introduced into the matrix part of corundum and mullite-corundum refractory concretes on aluminous cements,

the main phases of which are calcium mono- and dialuminate. In this case, solid-phase exchange reactions (1)–(11) will occur in fact for any stoichiometry of the components of the matrix part of refractory concretes. Calcined aluminas have no noted limitation in the development of solid-phase interaction and are more adapted for the sintering of the charges for corundum products, cement-free or ultra-low-cement corundum concretes, including those using fractionated tubular alumina.

Conclusions

The analysis of information on modern alumina made it possible to establish commonality and differences in their compositions. Corundum and β -alumina ($\text{Na}_2\text{O} \cdot 11\text{Al}_2\text{O}_3$) form the basis for tabular and all types of dispersed alumina. The differences are determined by the CaO content, first of all, since the main reaction solid-phase interaction during sintering is realized by Al_2O_3 , Na_2O and CaO. Other oxides only supplement the interaction mechanism with the achievement of the effects of the shrinkage compensation, formation of intergranular phases, reduction of total porosity, pore size, etc.

The research of alumina «Salox ALO DN-23» confirmed the performances in accordance with the manufacturer's specifications. At the same time, unspecified admixtures were identified in the studied alumina, the presence of which was analyzed from the standpoint of their functional role during sintering. Based on the results of the electron microscopy, the granulometry and morphological features of the particles of the studied alumina were established. The uniform nature of the distribution of sodium-containing phases was revealed, in contrast to silicon-containing ones. The dislocation of submicron particles from calcium-containing phases was determined mainly on the basal planes of relatively large corundum particles. It was shown that such an arrangement of calcium-containing phases promotes the formation of a dense layered microstructure during sintering, especially in the presence of β -alumina. The latter circumstance was confirmed by the thermodynamic probability of the progress of the solid-phase exchange reactions that are simulated and presented according to the subsolidus structure in the $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3$ system that establishes all two- and three-phase thermodynamically equilibrium combinations of the system.

The general pattern of the branched mechanism of the reaction phase formation during the sintering of the compositions in the $\text{Na}_2\text{O}-\text{CaO}-\text{Al}_2\text{O}_3$ system was illustrated by a diagram explaining the trend of physicochemical processes and the feasibility of using specific types of dispersed alumina for technologies of corundum products and refractory concretes with

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ТВЕРДОФАЗНІ РЕАКЦІЇ ОБМІНУ ПІД ЧАС СПІКАННЯ ДИСПЕРСНИХ ГЛІНОЗЕМІВ

С.М. Логвінков, О.М. Борисенко, А.А. Івашура, Г.М. Шабанова, В.М. Шумейко, О.М. Корогодська, Н.С. Цапко

У статті досліджено механізм спікання Al_2O_3 у присутності малих кількостей Na_2O та CaO . За результатами електронної мікроскопії встановлені гранулометрія та морфологічні особливості частинок досліджуваного глинозему, виявлено рівномірний характер розподілу натрієвмісних фаз, на відміну від кремнієвмісних, а також визначена дислокація субмікронних частинок кальцієвмісних фаз переважно на базальних площинах порівняно великих частинок корунду. Показано, що подібне розташування кальцієвмісних фаз сприяє формуванню щільної шаруватої мікроструктури при спіканні, особливо, у присутності β -глинозему. Загальна закономірність розгалуженого механізму реакційного фазоутворення під час спікання композицій системи $Na_2O - CaO - Al_2O_3$ проілюстрована схемою, що пояснює тренд фізико-хімічних процесів і доцільність застосування конкретних видів дисперсних глиноземів для технологій корундових виробів і вогнетривких цементів.

Ключові слова: табулярний глинозем, реактивний глинозем, алюмінати кальцію, корунд, β -глинозем.

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S.M. Logvinkov^a, O.M. Borysenko^{b,}, A.A. Ivashura^a, H.M. Shabanova^b, V.M. Shumeiko^b, A.M. Korohodska^b, N.S. Tsapko^c*

^a Simon Kuznets Kharkiv National University of Economics, Kharkiv, Ukraine

^b National Technical University «Kharkiv Polytechnic Institute», Kharkiv, Ukraine

^c Scientific-Research Institution «Ukrainian Scientific-Research Institute of Ecological Problems», Kharkiv, Ukraine

* e-mail: onborisenko@ukr.net

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