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SOL-GEL SYNTHESIS IN THE $CaO-Al_2O_3$ SYSTEM USING ALUMINA-CONTAINING WASTE

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Abstract
The article determines the optimal synthesis temperatures and methods of using alumina-containing waste in the process of solid-phase formation of crystalline structures of tricalcium aluminate. It has been established that the optimal synthesis temperature by the sol-gel method is 1100 °C and corresponds to the most complete formation of tricalcium aluminate with the smallest particle dispersion, size in the range of 100-700 nm.

Keywords Tricalcium aluminate, sol-gel method, aluminum containing waste, phase transformations, X-ray analysis, crystal structure, γ-alumina

INTRODUCTION

Calcium aluminates are among the most widely studied refractory compounds, included in a number of technical products, such as aluminous cement, Portland cement, some special cements, abrasives, phosphors, etc. [1-2]. They are also widely used in ceramics, binders in refractory castings for the steel industry, detectors, biomaterials and optical devices [3]. They have different crystal structures and are formed during the production of a number of chemical products, in the manufacture of transparent glasses for infrared radiation, called cobol glasses . They have not been found in natural materials, but their formation as intermediate compounds is possible during the formation of igneous rocks [4-5].

Over the past few decades, many methods have been used to synthesize calcium aluminates, including hydrothermal, combustion, Pechini , precipitation, and sol-gel methods [6-10].

In this work, Ca3Al2O4 was synthesized by the sol-gel method. The sol-gel method allows you to form the necessary phase compositions and structure of the material at lower temperatures.

In this regard, the possibility of using the alumina-containing component of the Shurtan gas-chemical complex for the synthesis of tricalcium aluminate by the sol-gel method.

MATERIAL AND METHODS

As the starting component for the study, we used 4-hydrate calcium nitrate ($Ca(NO3)\cdot 4H2O$ “pure grade”) and an alumina-containing spent catalyst, in which the aluminum oxide content is in the range of 94-96 mass % (Table 1), as well as nitric acid and polyvinyl alcohol.

The phase composition of the materials used and the synthesized calcium aluminate powder was determined on a LABX XRD-6100 SHIMADZU diffractograph using CuKα radiation, a Ni filter with a wavelength of 1.5418 Å.

RESULTS AND DISCUSSION

In many gas-chemical industries, the Claus method is used to purify natural gas from hydrogen sulfide; in particular, the Shurtan Gas-Chemical Complex (SGCC) in the Republic of Uzbekistan carries out catalytic oxidation of the latter with atmospheric oxygen on the surface of a high-alumina “bauxite” catalyst with the associated production of “gas” sulfur.

In this case, imported highly porous synthetic granular aluminum hydroxide is used as a catalyst, which, after expiration of its service life, is transferred to a dump as waste. The mass content of Al2O3 in this waste is 82-90 mass %, and after calcination at a temperature of 900 oC it usually reaches values of at least 95 mass % (Table 1) [11].

The mineralogical composition of the alumina-containing spent catalyst consists of gamma forms of alumina and gibbsite (Figure 1a).

Table 1

Chemical composition of the original and calcined waste sample alumina-containing catalyst ShGKhK

Name of samples	Mass content of oxides, %								LOI wt.%
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MgO	CaO	R_2O	SO_3	
Original	0.46	82.20	0.04	0.09	1.15	1.23	0.43	0.1	14.15
Heated	0.54	96.02	0.05	0.11	1.34	1.44	0.50	-	-

To obtain a single-phase gamma form of alumina (Figure 1b), the alumina-containing spent catalyst was heat treated at a temperature of 900 °C for 2 hours. X-ray data showed that after heat treatment of the original alumina-containing waste, lines of diffraction maxima with interplanar distances d = 0.455, 0.288, 0.236, 0.226, 0.197, 0.152, 0.139 nm are observed , related to the gamma form of alumina γ-Al2O3 and d = 0, 618, 0.317, 0.241, 0.185, 0.145, 0.143, 0.131 nm related to the gibbsite mineral γ Al(OH)3. At a temperature of 900 oC gibbsite is completely transformed into gamma form of aluminum oxide, resulting in a single-phase gamma alumina powder γ-Al2O3 with an interplanar distance d = 0.456, 0.280, 0.238, 0.227, 0.197, 0.151, 0.139 nm.

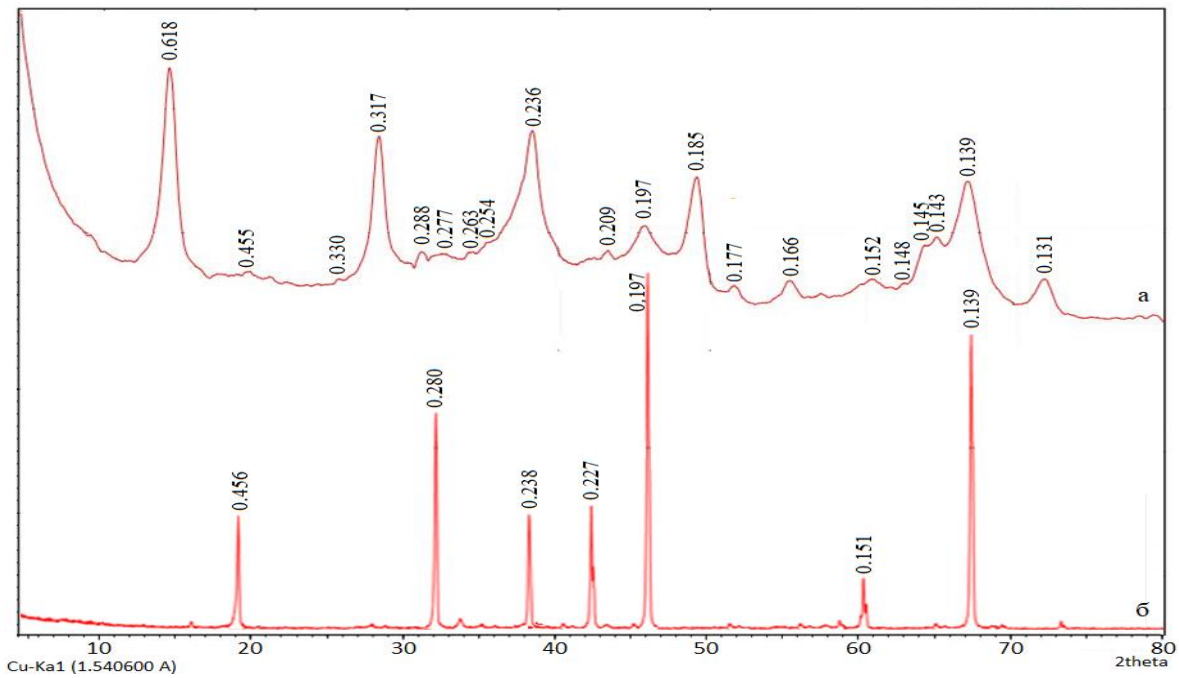

Figure 1. X-ray diffraction patterns of the alumina-containing component

a) original and b) thermally processed at a temperature of 900 ℃

The resulting γ - form of aluminum oxide was crushed in an agate mortar and dissolved in an aqueous solution of HNO 3 , and 4-water calcium nitrate (Ca(NO3) 4H2O) was dissolved in distilled water at room temperature. After stirring, polyvinyl alcohol (PVA) was added to the resulting solution. The precursor solution was stirred on a magnetic stirrer at a temperature of 70 °C until a gel-like mass was obtained. The resulting gel-like mass was dried at a temperature of 130 °C in an oven to obtain xerogels. To determine the formation of the crystal structure of tricalcium aluminate and the effect of exposure time during heat treatment on the synthesis process and the

complete completion of phase formation tricalcium aluminate, the dried gel was fired at a temperature range from 900 to 1100 °C 120 minutes in a muffle furnace SNOL 5/1300.

The X-ray diffraction pattern of the synthesized nanodispersed tricalcium aluminate powder by the sol-gel method is shown in Figure 2.

The results of X-ray analysis showed that the calcination process at temperature 900°C revealed diffraction lines with interference indices - hkl (231), (422), (342), (440), (462), (800), (844), (5102), and (1204) corresponding to the tricalcium phase aluminate and the intermediate mineral maenite Ca12Al14O33 (Figure 2c). As the firing temperature increases, 1000 °C the intensity and diffraction lines of tricalcium aluminate increases, and in proportion to the intensity of the maenite lines it decreases (Figure 2b).

However, when the temperature increases to 1100 °C, the presence of a line of diffraction maxima is observed, corresponding to the tricalcium mineral phase aluminate In this case, residual diffraction lines of the mineral maenite are also observed , with an insignificant amount (Figure 2a).

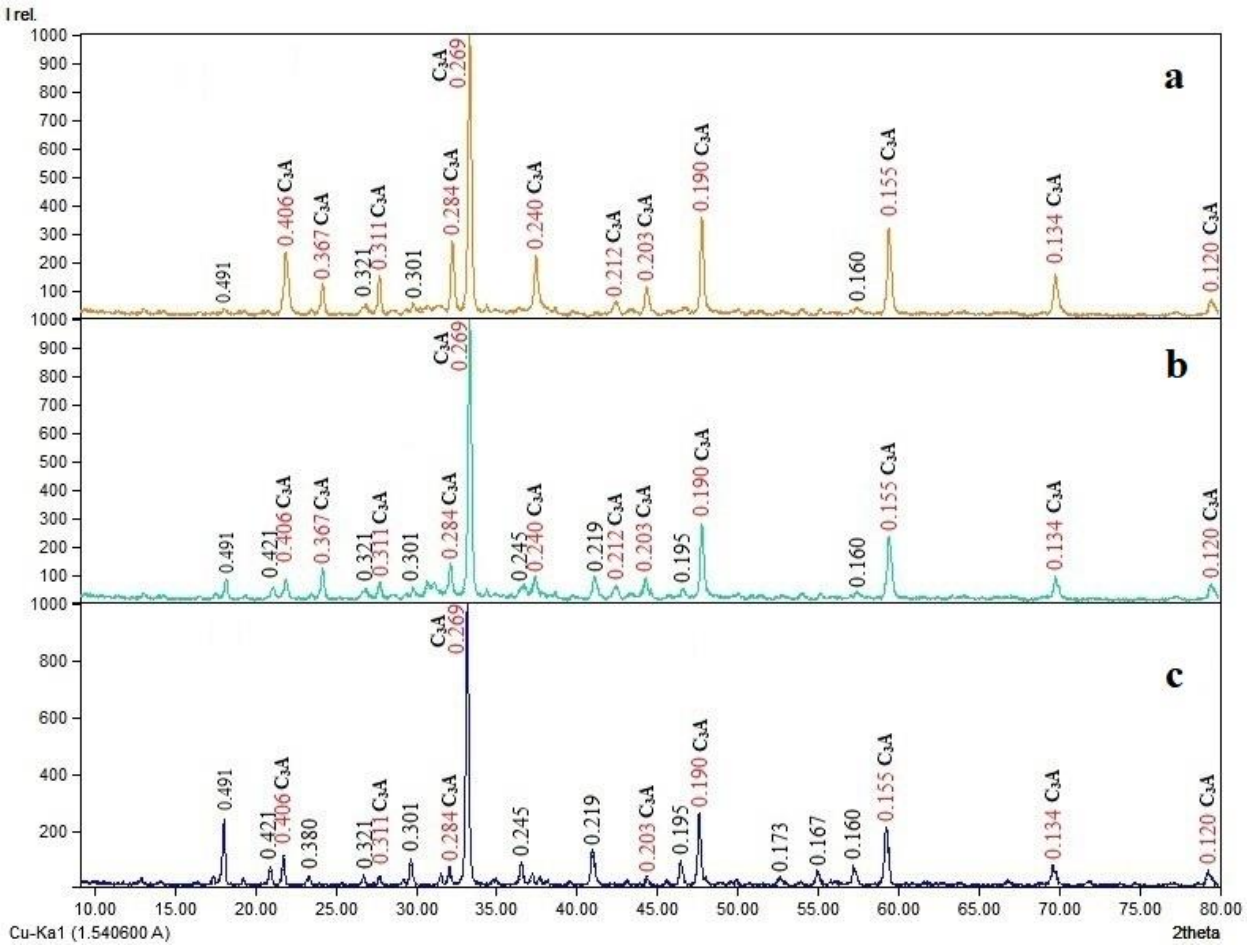

Figure 2. X-ray diffraction patterns of tricalcium aluminate synthesized by the sol-gel method. a) t - 900 °C b) t - 1000 °C c) t - 1100 °C

The experimental results obtained confirm that the process of formation of the structure of the crystalline phases of tricalcium aluminate (Ca3Al2O4) is completely completed.

CONCLUSION

In the course of experimental studies by the sol-gel method, the optimal temperatures for the synthesis of tricalcium aluminate and the possibility of using alumina- containing SHGCC waste as an initial component in their synthesis were determined . It has been established that the optimal synthesis temperature is 1100 oC , which corresponds to the most complete formation of tricalcium aluminate with the smallest particle dispersion up to a size of 100-700 nm .

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