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Preparation And Sintering Calcium Aluminate Nanopowder By Using Sol Gel Method

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ABSTRACT

The optimal synthesis temperatures and methods of using the alumina-containing waste in the process of obtaining calcium aluminate are determined. It has been established that the optimum synthesis temperature is 1100°C and corresponds to the maximum full formation of calcium aluminate with the smallest dispersion of particles, the size of 100-700 nm.

KEYWORDS

Calcium aluminate, sol-gel method, alumina containing waste, phase transformations, X-ray analysis, crystal structure, γ-alumina, refractory materials, cement, nanopowder.

INTRODUCTION

Calcium aluminates are among the most widely studied refractory compounds that are part of a number of technical products, such as alumina cement, portland cement, some special cements, abrasive materials, phosphors, etc. [1-3]. Also, they are widely used in ceramics, binders in refractory castings for the steel industry, detectors, biomaterials and optical devices. 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. They have not been found in the environment of natural materials; however, their formation as intermediate compounds is possible during the formation of igneous rocks. [4-5].

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Over the past several decades, a variety of methods have been used to synthesize calcium aluminates, including hydrothermal, combustion, Pecini, precipitation, and sol-gel. [6-10].

In this work, $CaAl_2O_4$ was synthesized by the sol-gel method. The sol-gel method allows the formation of the required 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 calcium aluminate by the sol-gel method was investigated.

MATERIAL AND METHODS

For the synthesis of calcium aluminate by the sol gel method, finely dispersed gamma-alumina was used as the main component, which is a spent catalyst of the Shurtan gas chemical complex (SGCC) in which the content of aluminum oxide is in the range of 94-96 wt. % (table 1), reagent grade calcium nitrate ($Ca(NO_3)\ 4H_2O$), and HNO_3, as well as nitric acid and citric acid was also used.

Thermal decomposition behavior was evaluated by thermo-gravimetric analysis (STA 449 F3, Netzsch, Germany). To identify the phase composition of the component used and the samples obtained, X-ray phase analysis was used on a LABX XRD-6100 SHIMADZU diffractometer using CuKα radiation (β-filter-Ni, wavelength 1.5418 Å, current mode and tube voltage 30 mA, 30 kW). Constant rotation speed of the detector 4° / min with a step of 0.02° (ω / 2θ - adhesion), the scanning angle varied from 4 to 80°. All samples were surveyed under constant conditions. International reference books of X-ray powder diffraction patterns were used in the calculations and in the identification of phases.

RESULTS AND DISCUSSION

In many gas-chemicals, when cleaning natural gas from hydrogen sulfide, the Claus method, in particular the Shurtan gas-chemical complex (SGCC), in the Republic of Uzbekistan, uses a catalytic oxidation of the latter air oxygen on the surface of a highly reduced "bauxite" catalyst with the associated receipt of "gas" sulfur.

At the same time, a susceptible synthetic granulated aluminum hydroxide is used as a catalyst, which after the expiration of the operation is translated into the dump as waste. The mass content of $A1_2O_3$ in this waste is 82-90 % [11], and after it is calcined at a temperature of 900 °C, it usually reaches values of at least 95 wt. % (table 1).

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Table 1

Chemical composition of the calcined sample of spent alumina-containing catalyst

Name of samples	Mass content of oxides, %							
	SiO_2	Al_2O_3	FeO	TiO_2	MgO	CaO	R_2O	SO_3
Calcined sample	0.54	96.02	0.05	0.11	1.34	1.44	0.50	-

The mineralogical composition of the alumina-containing spent catalyst consists of gamma forms of alumina and gibbsit (Fig. 1).

To obtain a single-phase gamma, the alumina shape was thermal processing of an aluming-containing spent catalyst at a temperature of 900 °C with a shutter speed of 2 hours.

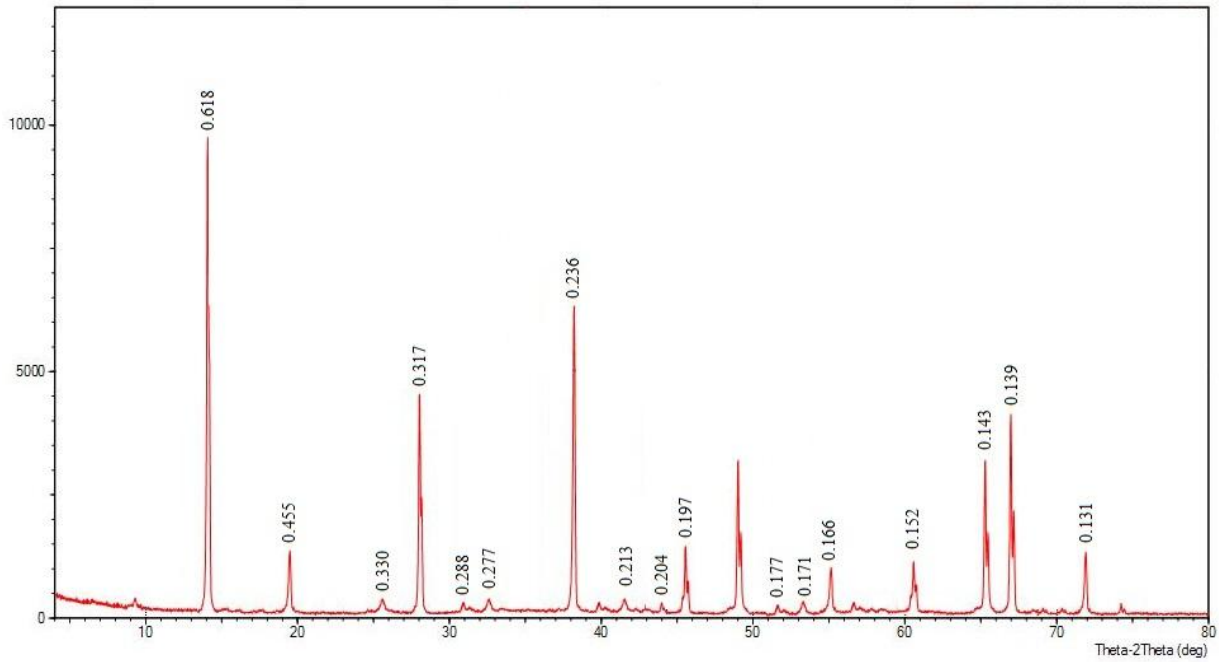

Fig. 1. X-ray of alumina-containing component

X-ray data have shown that after heat treatment of the original alumina-containing waste, there are lines of diffraction maxima with interplanar distances d = 0.455, 0.288, 0.236, 0,226, 0.197, 0.152, 0.139 nm, the gamma form of alumina γ-Al_2O_3 and d = 0.618, 0.317, 0.241, 0.185, 0.145, 0.143, 0.131 nm gibbsit mineral relatives γ-$Al(OH)_3$. At a temperature of 900, the gibbsit is completely converted to the gamma form of aluminum

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oxide, as a result of which a single-phase gamma alumina (γ-Al_2O_3) powder is obtained with an interplanar distance d = 0.455, 0.288, 0.236, 0.226, 0.197, 0.152, 0.139 nm.

The resulting γ form aluminum oxide shape was ground in agate mortar and was dissolved with an aqueous solution of HNO_3, and a 4-aqueous nitric acid calcium ($Ca(NO_3)\cdot 4H2O$) was dissolved in distilled water at room temperature. After stirring, polyvinyl alcohol (PVA) were added to the resulting solution. The precursor solution was stirred on a magnetic stirrer at 70 °C to obtain a gel-shaped mass. The resulting gelling mass was dried at a temperature of 90 °C in a drying cabinet before receiving xerogel. To determine the formation of the crystal structure of calcium aluminate and the effect of exposure time during heat treatment on the synthesis process and the complete completion of the calcium aluminate phase formation, the dried gel was burned at a temperature 1000 °C with an excerpt of 120 minutes in the SNOL 5/1300 muffle furnace.

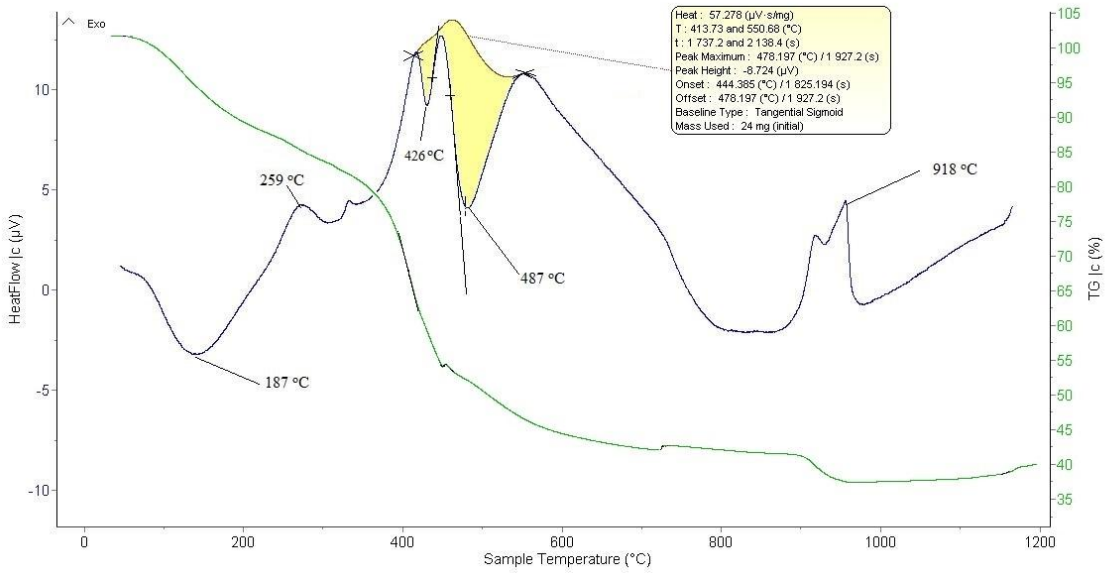

Fig. 2. The result of DSC-TG analysis of calcium aluminate precursor sol dried at 90 °C

The result of the DSC-TG pattern analysis shows that the mass loss is observed in the main one at a temperature of 110 - 978 °C. The weight loss range of the TG-curve was about 61.2%. The obtained results of differential-thermal analysis were shown (Fig. 2) that three endothermic effects were detected on the sample heating curve at temperatures 187, 426, 487, associated with the removal of H_2O molecules.

The appearance of two exothermic effects at temperatures 259, 918 °C are associated with oxidation and burning in the organic substances, as well as recrystallization of γ-Al_2O_3 and CaO on $CaAl_2O_4$. The endothermic transformation of the amorphous $Al(OH)_3$ on AlO(OH) at 187 °C may correspond to the evaporation of physically bound water. Weight loss of 426 °C to 430 °C was attributed to the dehydroxylation of mixed hydroxides

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AlO(OH) to γ-Al_2O_3 and $Ca(OH)_2$ to CaO. Weight loss occurs by the $2AlOOH \rightarrow Al_2O_3 + H_2O$ reaction by removing the H_2O molecule, which is accompanied by the appearance of thermal effects caused by a decrease in mass.

With a further increase in temperature up to 1200 °C, there is no thermal event.

The X-ray of the synthesized nanodispersed powder of calcium aluminate sol-gel method is shown in Fig. 3.

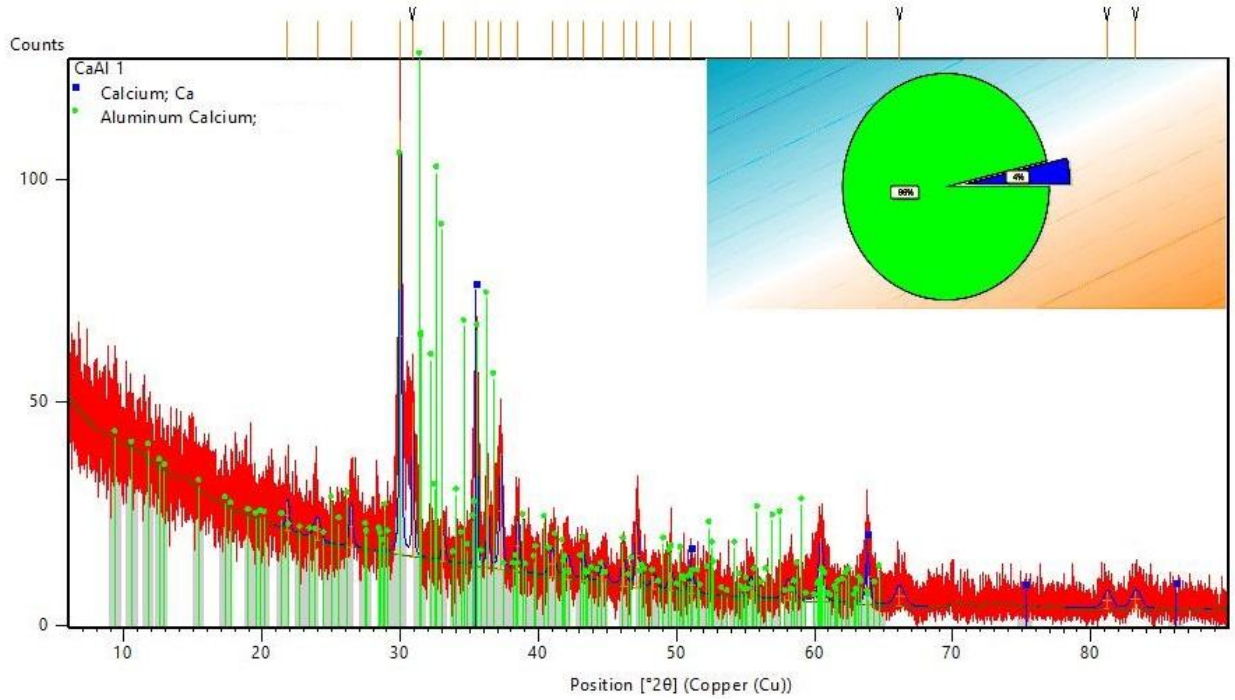

Fig. 3. X-ray of calcium aluminate synthesized sol gel method

The results of X-ray analysis have shown that the calcination process at a temperature of 1000 °C with a shutter speed of 120 minutes contributes to the maximum full of calcium aluminate. The calcination time did not show a significant effect on the calcium monoaluminate phase. The optimal calcination time for the formation of $CaAl_2O_4$ is 2 hours. Exposure for 1 hour is insufficient, and 4 hours of noticeable changes does not.

The obtained results of experimental data confirm that the process of forming the structure of crystalline phases of calcium aluminate ($CaAl_2O_4$) is fully completed.

CONCLUSION

In the course of the experimental studies of the sol-gel by the method, the optimal synthesis of calcium aluminate synthesis and the possibility of using the SGCC alumina as the initial component are determined with their synthesis. It has been established that the optimal synthesis temperature is 1000 OS, which corresponds to the maximum full formation of calcium aluminate with the smallest particle disperses to the size of 100-700 nm.

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