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Low Temperature Syntesis And Physico-Chemical Characterization Of $Mgal_2o_4$ Nanopowder

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ABSTRACT

As a result of the research carried out, conditions have been studied and created for the synthesis of spinel by using the sol-gel method. The kinetics of spinel phase formation has been studied. Received a fine ceramic powder, consisting of the main phase of spinel ($MgAl_2O_4$), which is obtained from waste and reactive magnesium oxide grade, having a particle size of 0.5 to 1 micron.

KEYWORDS

Alumina magnesium spinel, sol-gel method, kinetics, phase transformations, X-ray analysis, crystal structure, γ-alumina, refractory materials, nanopowder.

INTRODUCTION

To date, there are a number of studies on the synthesis and the study of physicochemical properties, phase transformations, isomorphic substitutions, interpretation of phase diagrams of a number of polycomponent oxide compounds of alkaline earth metals, in

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particular magnesium.

This particular place occupies a ceramic material based on pure oxides (Al_2O_3, MgO, CaO, BeO, ZrO_2 etc.) and synthesized products based on them (eg spinel) having a melting point above 2000-2500 °C, as well as high physical and technical properties such as high chemical purity [1-4], density (3.57-3.72 g / cm^3), gas-resistant [5], heat resistance, chemical resistance, mechanical hardness (7.5-8 on the Moos scale) [6,7] at high temperatures (mechanically and optically stable up to a temperature of 1250 °C and above) and a number of other properties. The magnesium aluminate spinel has a wide field of use due to its high chemical inertness and thermomechanical properties [8-10].

Therefore, they find widespread use in armored window systems, high-energy laser windows, rocket dome, electronic humidity sensors, refractories, catalytic carriers, etc. Magnesium aluminate spinel is also well known for its optical properties that make it necessary material in production transparent ceramics for use in visible [11,12]. Spectrum, near infrared and microwave frequency ranges.

Optimization of the methods of the synthesis of spinel and the study of the kinetics occurring at the same time, chemical reactions seems to be an urgent task. In this paper, an attempt was made to determine and compare the interaction of various precursors, leading to the formation of magnesium aluminate spinel.

MATERIAL AND METHODS

For the spinel synthesis, the coil gel was used by the technique of fine gamma alumine as the main component, which is a spent catalyst of the Mubarek gas processing plant (MGPP). Also used magnesium oxide obtained by roasting magnesium carbonate, HNO_3, glucose ($C_6H_{12}O_6$) anhydrous and 5% solution polyacrylamide, partially hydrolyzed using NaOH, the results of chemical analysis spent catalyst of the Mubarek GPP, was shown that the content after the injection at a temperature of 900 °C γ-Al_2O_3 is approximately 96 wt.% (table 1).

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 [13,14]. International reference books of X-ray powder diffraction patterns were used in the calculations and in the identification of phases.

RESULTS AND DISCUSSION

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-$Al(OH)_3$.

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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	MgO	CaO	R_2O
Spent catalyst MGPP	1,5	96	0,6	1,0	0,25	0,65

At a temperature of 900 °C, the gibbsit is completely converted to the gamma form of aluminum 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. X-rays analysis (Fig. 1) shows at a temperature of 500 °C almost all diffraction lines corresponds to the mineral γ-Al_2O_3. However, with an increase in temperature to 900 °C intensity, the corresponding diffraction lines increases and traces that belong to gibbsit completely disappear.

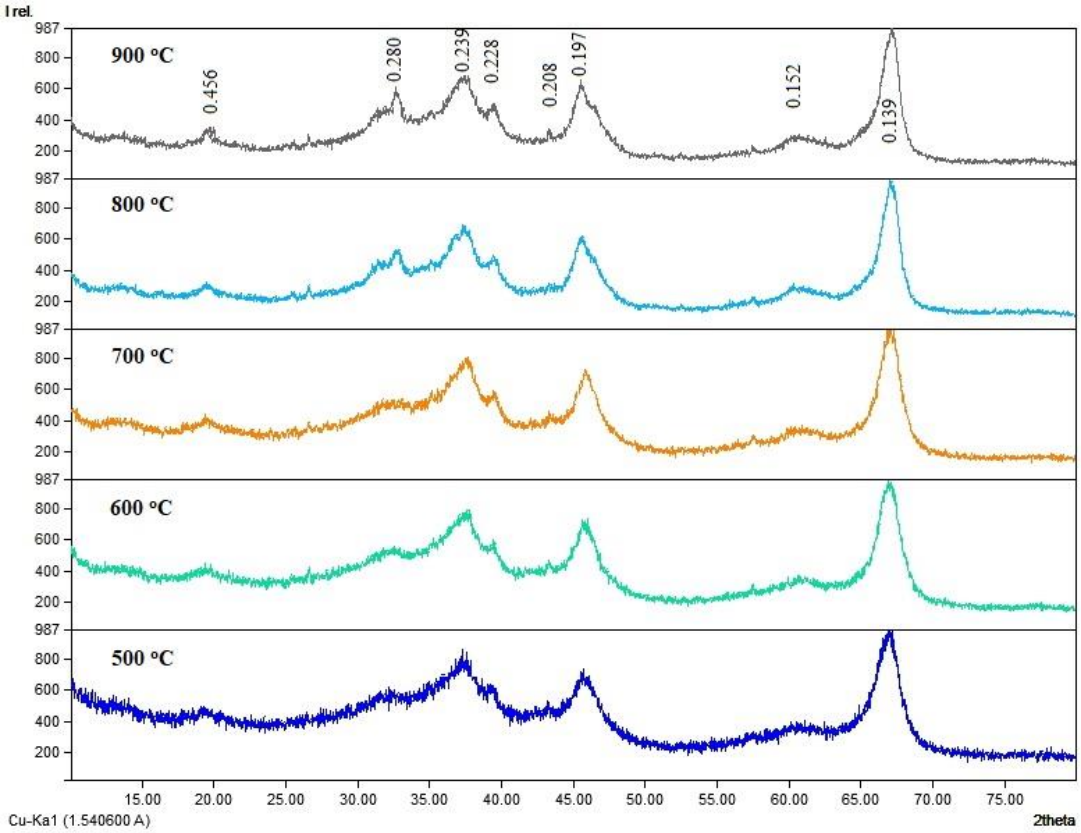

Fig. 1. X-ray of alumina-containing waste heat treated at a temperature of 500-900 °C

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The results of thermogravimetric and differential analysis shows that the mass loss is observed in the main at a temperature of 150 - 615 °C. The obtained results of differential-thermal analysis of the alumine-containing waste samples were shown (Fig. 2) that two endothermic effects were detected on the sample heating curve at temperatures 169, 578 °C associated with the removal of H_2O molecules. The appearance of two exothermic effects at temperatures of 315, 489 is associated with the oxidation and burnout of organic substances, as well as the recrystallization of gibbsite to boehmite γ-AlO(OH). The second weight loss associated with the endothermic peak at 578 °C is explained by the crystallization of boehmite in γ-Al_2O_3 (as observed in the X-ray diffraction pattern in Fig. 1). Weight loss occurs by the reaction $2AlOOH \rightarrow Al_2O_3 + H_2O$ by removing the H_2O molecule, which is accompanied by the appearance of thermal effects due to a decrease in weight. The total weight loss in the temperature range 150 - 615 °C according to the thermogravimetry curve is 14.2%. With a further increase in temperature to 1100 °C, no thermal event is observed.

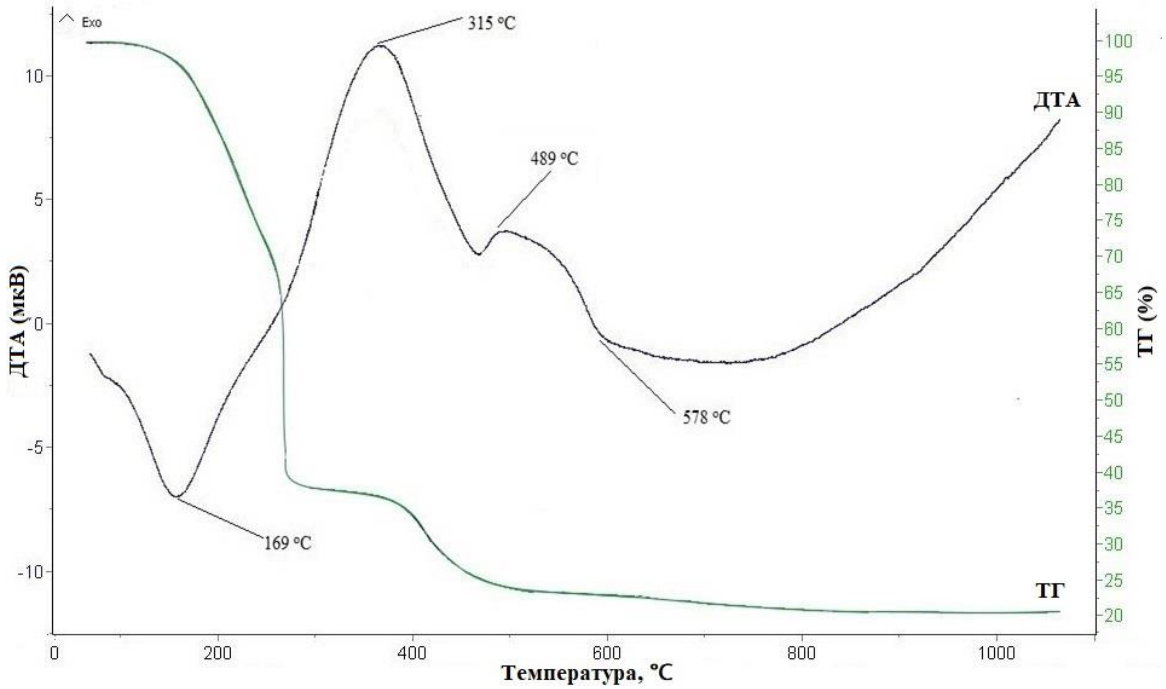

Fig. 2. The results of thermogravimetric and differential-thermal analysis of aluminum-containing waste

In the process of obtaining alumopesal spinels, the sol-gel by the method of the stoichiometric amount of the spent catalyst γ-Al_2O_3, MgO and glucose was stirred in dry form and crushed to the particle size of less than 0.1 μm. The resulting mixture was dissolved in the H_2O of partially dilute nitric acid. The precursor solution was then kept on a magnetic stirrer for 60 minutes to obtain a homogeneous mixture, and the pH was maintained to 10 by adding NaOH drops. After that, the solution was continuously stirred into the magnetic stirrer for 5 hours, maintaining its temperature in the range of 50-60 °C. After that, a 5% solution of

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polyacrylamide was added obtained by a homogeneous liquid and the gel precipitate was obtained. The finished product was filtered and dried at room temperature. The resulting samples were burned in the SNOL 5/1300 muffle furnace in the temperature range of 500-1000 °C (Fig.3), increasing the temperature for each individual sample per 100 °C with an exposure of 2 hours.

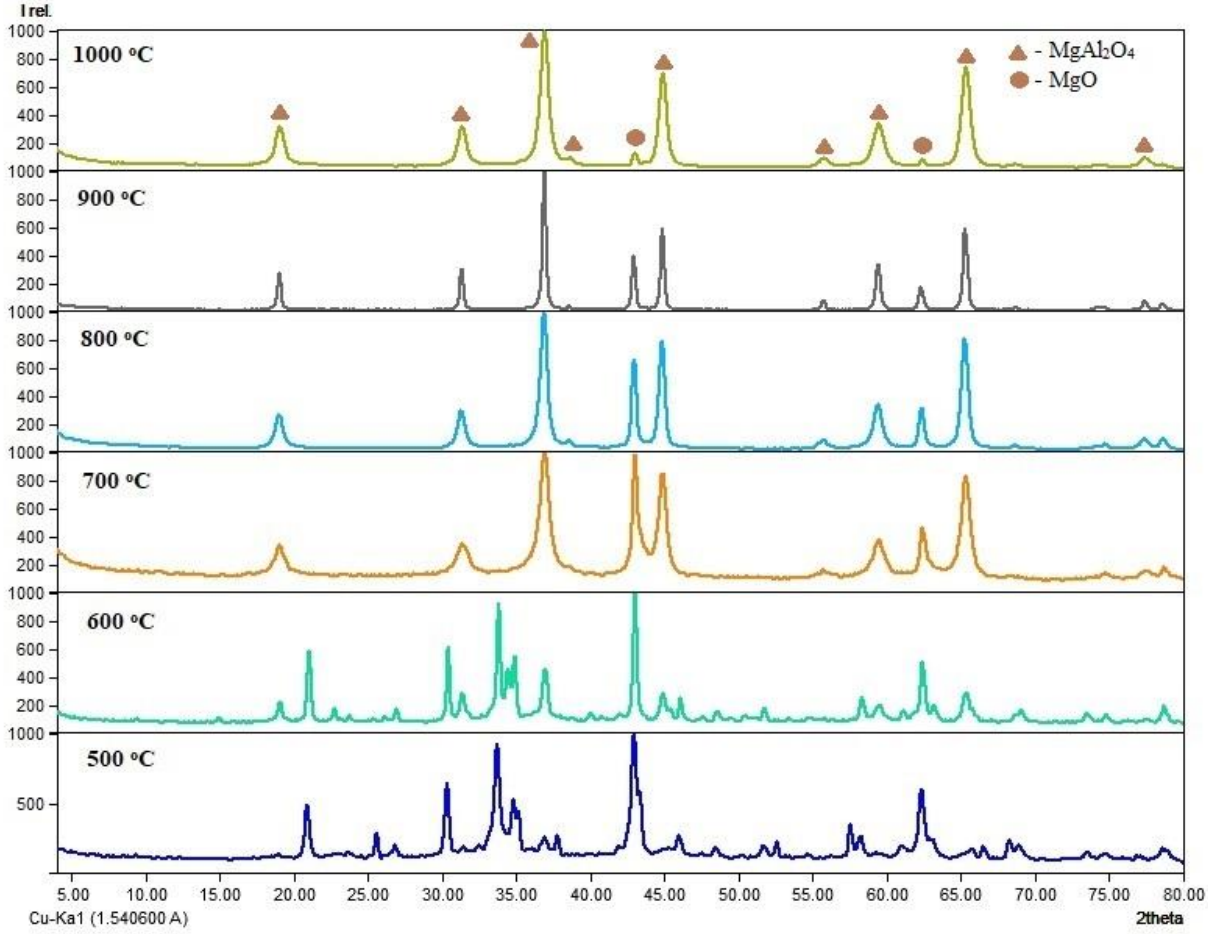

Fig. 2. X-ray patterns treated with isothermally at temperatures 500°C, 600°C, 700°C, 800°C, 900°C and 1000°C for 120 minutes.

X-ray analysis shows that the most intensive effects of diffraction lines appear in the area of the slide angle 2θ (36.9; 44.5 and 65.3 °) in all samples at three excerpt intervals (Fig. 3). These diffraction lines with HKL (311) interference indices (400) and (404) correspond to the magnesium aluminate phase. However, with an increase in the firing point to 1000 °C, the intensity and diffraction lines magnesium aluminate spinel increases, in proportion to the intensity value of magnesium oxide lines decreases. This confirms that the process of forming the crystal structure of the spinel is fully completed. It should be noted that $C_6H_{12}O_6$ was added for the purpose of mixing and homogenizing the masses. X-ray data showed that $C_6H_{12}O_6$ decomposes and does not participate in the process of mineral formation. Combustion $C_6H_{12}O_6$ at temperatures above 300 °C was accompanied by intense boiling of its crystals, which decompose and volatilize.

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CONCLUSION

Thus, as a result of the studies carried out, the conditions for the synthesis of spinel by using the sol-gel method were studied and created. Received a fine ceramic powder, consisting of the main phase of spinel ($MgAl_2O_4$), which is obtained from waste and reactive magnesium oxide grade, having a particle size of 0.5 to 1 micron.

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