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Industrial Wastes As Prospective Raw Materials For The Synthesis Of Ceramic Materials

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Abstract

The growing accumulation of industrial wastes and the depletion of natural mineral resources have intensified interest in the development of alternative raw materials for ceramic technologies. In the present study, spent alumina-containing catalyst waste generated during natural gas purification at the Shurtan Gas Chemical Complex was investigated as a prospective raw material for ceramic material synthesis. The chemical composition, phase transformations, and thermal behavior of the waste material before and after thermal treatment were studied by chemical analysis, X-ray diffraction (XRD), differential thermal analysis (DTA), and thermogravimetric analysis (TG). The initial waste mainly consisted of gibbsite and γ-alumina phases, while calcination at 900 °C promoted complete dehydration and the formation of single-phase $\gamma\text{-}Al_2O_3$ *with an alumina content exceeding 96 wt.%. Thermal analysis revealed that the principal weight loss occurred within the temperature range of 150–615 °C and was associated with dehydration and phase transformation processes. The obtained results demonstrate the possibility of utilizing alumina-containing industrial waste as a promising secondary raw material for the production of advanced ceramic materials.*

Keywords: Industrial waste; alumina-containing waste; $\gamma\text{-}Al_2O_3$; thermal treatment; X-ray diffraction; phase transformation; ceramic materials; waste valorization.

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1. Introduction

The rapid growth of industrial production has led to the large-scale accumulation of solid technogenic wastes, creating serious environmental and economic challenges worldwide. At the same time, increasing demand for high-performance ceramic materials requires the development of sustainable and cost-effective raw material sources. In this regard, industrial wastes rich in aluminosilicate and oxide components are considered promising alternative raw materials for ceramic technologies due to their chemical composition, availability, and low cost [1-5].

Among various industrial by-products, alumina-containing wastes generated in petrochemical and gas-processing industries attract particular attention because of their high Al_2O_3 content and potential applicability in advanced ceramic systems. The reutilization of such wastes not only reduces the environmental burden associated with their disposal but also contributes to resource conservation and the implementation of circular economy principles [6-8].

High-alumina wastes are widely used in the synthesis of refractory ceramics, cordierite-based materials, porous ceramics, catalysts, and ceramic composites. Previous studies have demonstrated that thermal treatment significantly influences the phase composition, microstructure, and physicochemical properties of alumina-containing systems. In particular, the formation of metastable γ-Al_2O_3 phases during dehydration processes plays an important role in determining the reactivity and sintering behavior of ceramic raw materials [9-11].

In the present work, spent alumina-containing catalyst waste obtained from the Shurtan Gas Chemical Complex was investigated as a prospective raw material for ceramic material synthesis. Special attention was focused on the chemical composition, phase evolution, and thermal transformations occurring during calcination in the temperature range of 500–900 °C. The obtained results provide important information regarding the possibility of utilizing this industrial waste in advanced ceramic technologies.

Materials and Methods

Under industrial conditions, during the purification of natural gas from hydrogen sulfide by the Claus process, particularly at the Shurtan Gas Chemical Complex, catalytic oxidation of hydrogen sulfide with atmospheric oxygen is carried out on the surface of a high-alumina “bauxite” catalyst to produce commercial elemental (“gas”) sulfur. The catalyst used is a synthetic granular highly porous aluminum hydroxide which, during operation at temperatures above 400 °C, undergoes partial dehydration with the formation of γ-aluminum oxide (γ-Al_2O_3).

The alumina content in the initial catalyst as well as in the thermally treated samples is presented in Table 2.2. The chemical composition was determined according to the procedure specified in ГОСТ 2642.0–86. Samples for analysis were taken from 200 g portions. In accordance with the standard procedure, the samples were preliminarily crushed to pass through a No. 05 sieve, thoroughly mixed, and reduced by quartering to a mass of 50–60 g. Subsequently, the material was additionally ground to pass through a No. 02 sieve, followed by final homogenization to obtain a representative sample weighing 10–20 g. Final grinding was carried out until complete passage through a No. 0063 sieve. The prepared samples were then subjected to chemical composition analysis in compliance with ГОСТ 2642.0–86.

Phase identification of the raw materials and synthesized samples was carried out by X-ray diffraction (XRD) analysis using Shimadzu LABX XRD-6100 (CuKα radiation, Ni filter, wavelength $\lambda = 1.5418$ Å, tube operating conditions: 30 mA and 30 kV). The detector rotation speed was maintained at 4°/min with a step size of 0.02°, while the scanning range varied from 4° to 80° (2θ). Phase identification and calculations were performed using the international powder diffraction database ICDD PDF-2 (2007 release). The quantitative phase content was determined using the “Crystall Impact Match” software (version 4) in combination with the ICDD PDF-2 database.

Phase transformations and thermal behavior of the samples were investigated by differential thermal analysis (DTA) and thermogravimetric analysis (TG) using a Setaram LABSYS evo thermal analyzer equipped with a Paulik–Paulik–Erdey system. Measurements were performed in platinum crucibles at a heating rate of 10 °C/min using a Pt–Pt/Rh (type S) thermocouple. The sample mass was approximately 2 g. In addition, thermal dilatometric measurements were carried out to evaluate changes in the linear dimensions of the samples during heating.

Results and Discussion

The mass fraction of Al_2O_3 in this material ranges from 82 to 90 wt.%, while after calcination at 900 °C it generally reaches values of not less than 96 wt.%.

Table 1

Chemical composition of the spent alumina-containing catalyst before and after thermal treatment

Samples	Oxide content (wt.%)								LOI (wt.%)
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MgO	CaO	R_2O	SO_3	
Initial ShGCC catalyst	0,46	82,20	0,04	0,09	1,15	1,23	0,43	0,10	14,15
Calcined ShGCC catalyst	0,54	96,02	0,05	0,11	1,34	1,44	0,50	–	–

The results of the X-ray diffraction analysis showed that the mineralogical composition of the initial spent catalyst includes gibbsite with interplanar spacings of d = 0.618, 0.317, 0.241, 0.185, 0.145, 0.143, and 0.131 nm, as well as γ-alumina with characteristic interplanar spacings of d = 0.455, 0.288, 0.236, 0.226, 0.197, 0.152, and 0.139 nm (Fig. 1a).

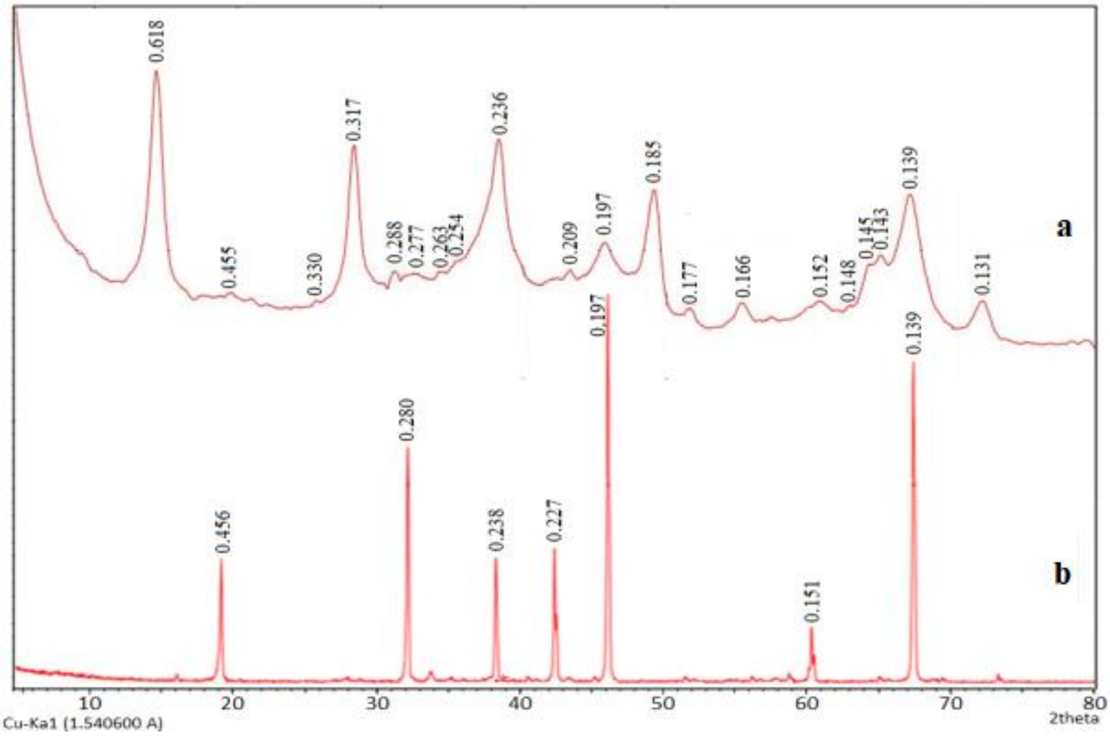

Fig. 1. XRD patterns of alumina-containing waste materials:

a) initial sample; b) sample thermally treated at 900 °C

To increase the content of single-phase Al_2O_3, the alumina-containing waste was thermally treated at 900 °C for 120 min. As a result, γ-Al_2O_3 crystallizing in a cubic structure was obtained. The XRD patterns of the heat-treated samples revealed diffraction maxima corresponding to γ-Al_2O_3 with interplanar spacings of d = 0.456, 0.280, 0.238, 0.227, 0.197, 0.151, and 0.139 nm (Fig. 1b). At the same time, the diffraction peaks associated with gibbsite disappeared due to the dehydration process.

To investigate the kinetics of phase transformations occurring during thermal treatment, the alumina-containing waste was calcined in the temperature range of 500–900 °C at intervals of 100 °C (Fig. 2).

The XRD analysis (Fig. 2) showed that at 500 °C almost all diffraction peaks correspond to the γ-Al_2O_3 phase, although a small number of weak diffraction lines attributed to gibbsite were still detected. However, with increasing temperature up to 900 °C, the intensity of the diffraction peaks corresponding to γ-Al_2O_3 increased, while the weak reflections associated with gibbsite disappeared completely.

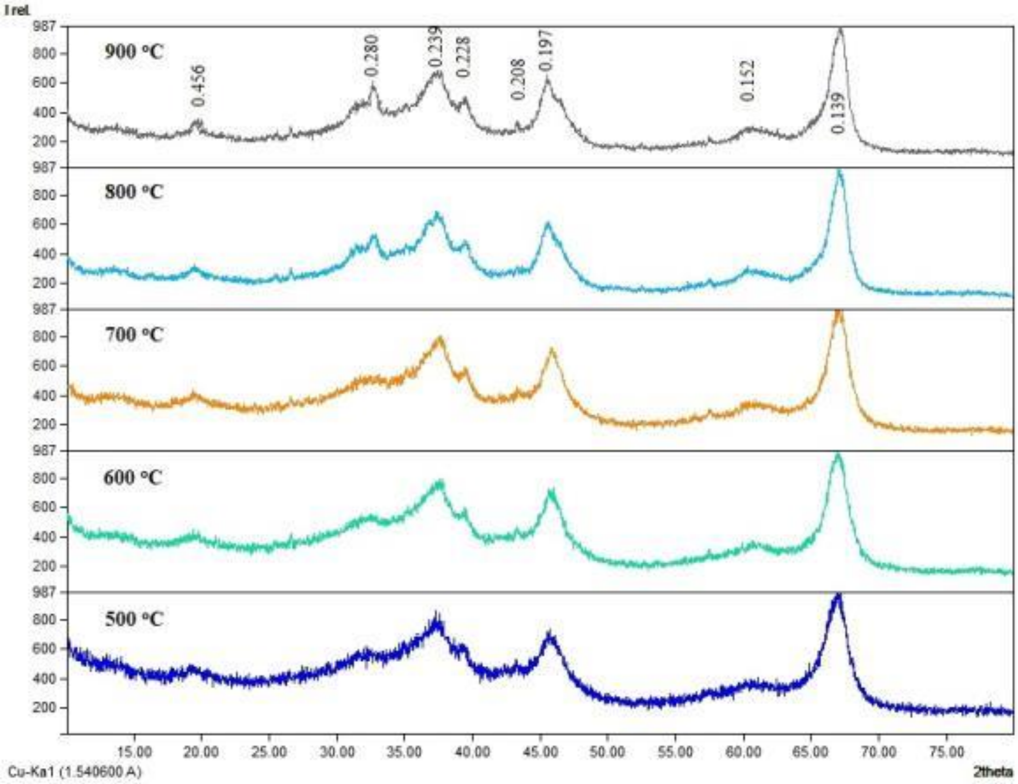

Fig. 2. XRD patterns of alumina-containing waste materials thermally treated at 500–900 °C.

The thermogravimetric (TG) and differential thermal analysis (DTA) results showed that the major weight loss occurred within the temperature range of 150–615 °C. As shown in Fig. 3, two endothermic effects were observed at 169 and 578 °C, which are associated with the removal of H_2O molecules [12]. In addition, two exothermic effects at 315 and 489 °C were attributed to the oxidation and burnout of organic matter, as well as to the recrystallization of gibbsite into boehmite γ-AlO(OH). The second weight-loss stage, accompanied by the endothermic effect at 578 °C, is related to the transformation of boehmite into γ-Al_2O_3, which is consistent with the XRD results presented in Fig. 2.

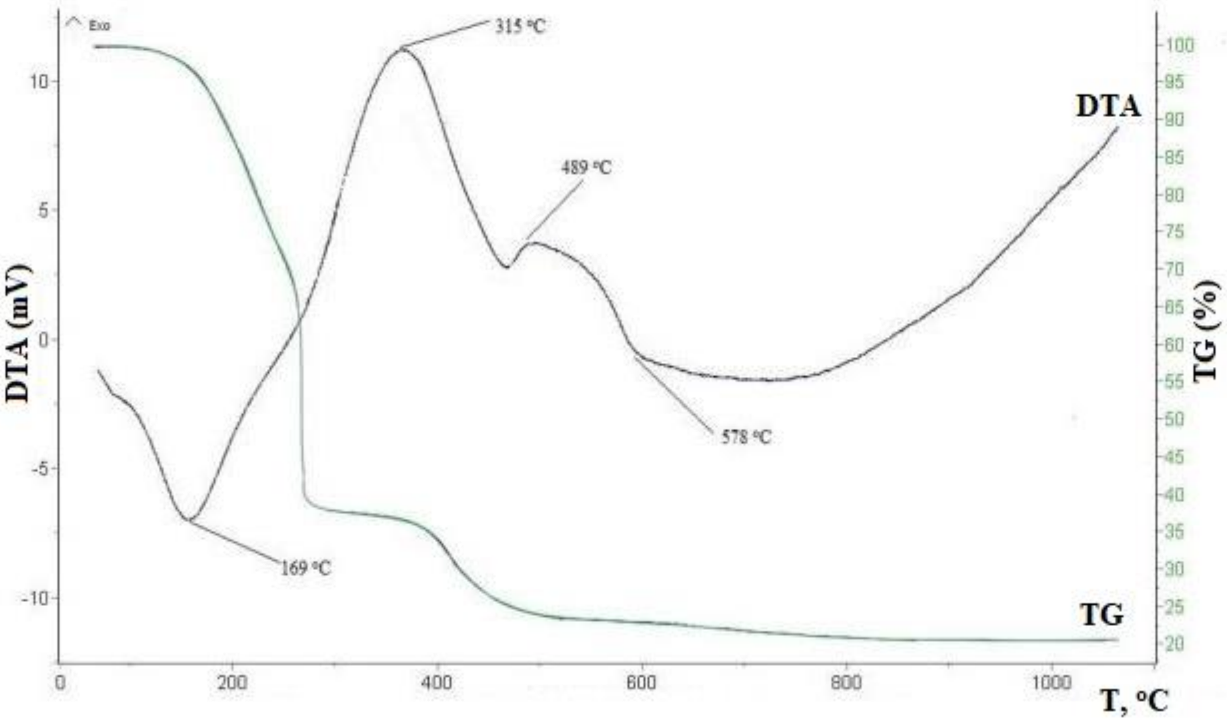

Fig. 4. DTA–TG curves of the alumina-containing waste material.

The weight loss occurs according to the reaction $2AlOOH \rightarrow Al_2O_3 + H_2O$ due to the removal of H_2O molecules, which is accompanied by thermal effects associated with mass reduction. The total weight loss in the temperature range of 150–615 °C, according to the thermogravimetric curve, was 14.2%. With further heating up to 1100 °C, no additional thermal events were observed.

Conclusions

The possibility of utilizing spent alumina-containing catalyst waste from the Shurtan Gas Chemical Complex as a prospective raw material for ceramic material synthesis was investigated. Chemical analysis showed that the Al_2O_3 content increased from 82–90 wt.% in the initial material to more than 96 wt.% after calcination at 900 °C.

XRD analysis demonstrated that the initial waste mainly consisted of gibbsite and γ-Al_2O_3 phases. Thermal treatment at 900 °C resulted in complete dehydration of gibbsite and the formation of predominantly single-phase γ-Al_2O_3 with a cubic crystal structure.

Thermal analysis revealed that the major mass loss occurred within the temperature range of 150–615 °C and was associated with dehydration, oxidation of residual organic compounds, and phase transformations involving the conversion of gibbsite to boehmite and subsequently to γ-Al_2O_3. No significant thermal effects were observed above 1100 °C, indicating the thermal stability of the obtained material.

The obtained results confirm that alumina-containing industrial waste can be considered a promising secondary raw material for the production of advanced ceramic materials and high-alumina ceramic systems.

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