Synthesis of Lightweight Aluminosilicate Proppants from Low-Grade Kaolinitic Clay And Industrial Waste

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Abstract

This study presents the synthesis of lightweight aluminosilicate ceramic proppants using Angren kaolinitic clay and ash–slag industrial waste as raw materials. The effects of composition and sintering temperature (1200–1250 °C) on phase composition and physicomechanical properties were investigated. The results show that increasing the sintering temperature enhances densification, promotes mullite formation, and improves mechanical strength and chemical stability. The optimal composition (CA-3) exhibited low bulk density (1.43 g/cm³), low crushing ratio (6.58%), and high acid resistance (5.56%). XRD analysis confirmed increased crystallinity and mullite development at higher temperatures. The developed proppants demonstrate competitive properties and offer a cost-effective and environmentally sustainable alternative to conventional materials.

Keywords: Ceramic proppants, kaolinite clay, ash–slag waste, hydraulic fracturing, mullite, industrial waste utilization.

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1. Introduction

Al-Farobi, At present, the global demand for petroleum resources continues to increase steadily. Despite the widespread implementation of advanced enhanced oil recovery (EOR) techniques, the efficiency of oil extraction from productive reservoirs remains insufficient. Under current field development conditions, a persistent decline in well productivity is observed, primarily due to reservoir depletion, deterioration of reservoir permeability and porosity characteristics, and the increasing proportion of hard-to-recover reserves. As a result, a significant fraction of hydrocarbons remains unrecovered, necessitating the development and implementation of more effective recovery methods. In this context, improving hydrocarbon recovery efficiency has become one of the key challenges facing the oil and gas industry at the present stage of its development. It is estimated that only 25–45% of global oil reserves have been recovered, while 55–75% remain trapped in reservoirs as residual oil. Therefore, there is a growing interest in the development of innovative EOR technologies aimed at reducing operational costs, increasing production rates, and enhancing the overall economic efficiency of reservoir exploitation [1 – 3].

One of the most effective methods for increasing well productivity is hydraulic fracturing (HF). The application of HF significantly enhances the permeability of the near-wellbore zone, creates additional flow channels, and intensifies the influx of reservoir fluids into the wellbore. This technique plays a particularly important role in the development of low-permeability and hard-to-recover reserves, where conventional stimulation methods are often insufficiently effective [4].

The effectiveness of hydraulic fracturing (HF) is largely governed by the properties and behavior of the proppant under reservoir conditions. Although fracture initiation and propagation are achieved through the pressure of the injected fluid, it is the proppant that ensures the long-term effectiveness of the treatment. In the absence of proppant, the induced fractures tend to close rapidly under in-situ stresses, thereby significantly diminishing the overall impact of hydraulic fracturing [5, 6].

The proppant acts as a mechanical framework that maintains fractures in an open state after the cessation of fluid injection. This results in the formation of a stable network of highly permeable flow channels that facilitate the transport of reservoir fluids toward the wellbore. Thus, while the fracturing fluid is responsible for creating the fracture, it is the proppant that determines its conductivity and, consequently, the ultimate productivity of the well.

One of the key factors determining the effectiveness of hydraulic fracturing is the proppant, with its strength, shape, and particle size playing a critical role. In addition, the particle size distribution and uniformity are of great importance: an optimal particle size ensures high filtration efficiency, whereas an excessive proportion of fine particles can lead to the blockage of flow channels. Furthermore, the proppant must exhibit high chemical and thermal stability under conditions of elevated temperatures and aggressive environments. Therefore, it represents a crucial component of hydraulic fracturing technology, and its proper selection and application directly influence well productivity, enhanced oil recovery, and the overall efficiency of reservoir development [7, 8].

Proppants are characterized by a wide range of chemical compositions, including high-alumina, aluminosilicate, magnesium silicate, and other types. Depending on their density, they are typically classified as heavy, intermediate, and lightweight. It should be noted that both natural and industrial waste materials can be utilized as raw feedstocks for their production. Under current conditions, lightweight proppants with high strength and corrosion resistance are of particular interest. Their application allows for a reduction in the required viscosity of the fracturing fluid, thereby lowering the overall operational costs of hydraulic fracturing [10, 11].

The aim of this study is to develop ceramic proppants based on natural and secondary aluminosilicate raw materials available in Uzbekistan.

Conventional ceramic proppants produced from high-alumina raw materials such as bauxite and kaolin are associated with high production costs due to energy-intensive processing and reliance on high-quality feedstocks. Therefore, the utilization of industrial solid waste has emerged as a promising and economically viable alternative. The use of secondary raw materials not only reduces production costs but also enables the development of materials with competitive properties. Angren clay and ash–slag waste, which are rich in Al_2O_3 and SiO_2, represent suitable precursors for the production of such proppants; in this study, they were used as a complete replacement for conventional raw materials. In addition, the influence of sintering temperature on phase composition and microstructure was systematically investigated, along with the evaluation of bulk density, apparent density, and compressive strength.

Ceramic proppants are typically manufactured from bauxite and enriched kaolin. The primary objective of this study is to develop a formulation for ceramic proppants based on secondary Angren kaolin and industrial waste, capable of replacing imported products while meeting relevant standard requirements. This approach offers significant advantages, including the conservation of foreign currency resources, enhancement of domestic production capacity, expansion of the raw material base, and improvement of environmental sustainability through effective waste utilization.

2. The Experimental Part

To develop proppant formulations and investigate the effect of sintering temperature on their physicomechanical properties, Angren kaolinitic clay and ash–slag waste generated during the electricity production process at the Angren thermal power plant were selected as raw materials.

The chemical and mineralogical compositions of the initial components were determined using chemical and X-ray diffraction (XRD) analyses. The chemical analysis of the samples was carried out in accordance with GOST 2642.0–2014.

The phase composition of the raw materials and synthesized samples was determined by X-ray diffraction (XRD) using a Rigaku MiniFlex diffractometer (Japan) with CuKα radiation (λ = 0.154 nm). The measurements were carried out at a scanning rate of 5°/min over a 2θ range of 10–70°. The diffraction patterns were analyzed and the crystalline phases were identified using reference data and the International Centre for Diffraction Data (ICDD) PDF-2 database.

The flexural strength of the sintered samples and the crushing resistance of the ceramic proppants at a pressure of 35 MPa were determined using an automatic strength testing machine (CFT-P300, Hangzhou Civil Instrument Equipment Co., Ltd., China) in accordance with GOST R 51761–2013 standard. The apparent density and acid solubility were also measured in accordance with the aforementioned standards.

The chemical composition of the Angren clay and ash–slag waste used for the production of aluminosilicate proppants is presented in Table 1.

Table 1. Chemical compositions of the raw materials

Raw material	Oxide content (wt.%)								LOI, mass, %
	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	
Angren kaolinitic clay	51,98	25,81	0,42	5,76	1,18	1,11	1,01	0,87	11,86
Ash–slag waste	63,56	21,02	-	3,62	6,25	0,78	2,88	1,24	0,65

The results of the chemical composition analysis presented in Table 1 indicate that the Al_2O_3 content in both the clay and ash–slag waste exceeds 20%. It should be noted that the contents of alkali oxides and calcium oxide in these materials are significantly higher compared to those in kaolinitic clay. Based on these findings, batch compositions were designed for the production of ceramic proppants, and their corresponding chemical compositions were calculated (Table 2).

Table 2. Compositions of proppants and content of oxides

Sample designation	Content of components, %		Oxide content (wt.%)					
	Angren kaolinitic clay	Ash–slag waste	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O+K_2O
CA-1	60	40	56,61	23,89	4,90	3,21	0,98	2,78
CA-2	50	50	57,77	23,42	4,69	3,72	0,95	3,01
CA-3	40	60	58,93	22,94	4,48	4,22	0,91	3,22

With increasing ash–slag waste content in the batch composition, a slight decrease in the contents of aluminum and iron oxides is observed, whereas the concentrations of the remaining oxides increase (Table 2). The elevated content of alkali oxides in the batch promotes the formation of a liquid phase and has a beneficial effect on the development of the mullite phase.

The prepared proppant compositions were sintered at temperatures of 1200 and 1250 °C with a holding time of 60 min. After sintering, the samples were evaluated for their physicomechanical properties, and their phase compositions were determined. The physicomechanical properties of the sintered samples are presented in Table 3.

Table 3. Physicomechanical properties of ceramic proppant samples

Sample designation	Bulk density (g/cm³)		Crushing ratio at 35 MPa (%)		Acid solubility (%)	
	1200°C	1250°C	1200°C	1250°C	1200°C	1250°C
CA-1	1,46	1,49	8,85	7,50	7,51	6,74
CA-2	1,44	1,46	8,50	7,11	7,08	6,23
CA-3	1,41	1,43	8,11	6,58	6,74	5,56

The analysis of the physicomechanical properties of the ceramic proppants reveals a clear dependence on sintering temperature and batch composition (Table 3). With an increase in temperature from 1200 to 1250 °C, a slight increase in bulk density is observed for all samples (CA-1: 1.46→1.49 g/cm³; CA-2: 1.44→1.46 g/cm³; CA-3: 1.41→1.43 g/cm³), which can be attributed to enhanced sintering, reduced porosity, and structural densification due to liquid phase formation.

At the same time, a significant reduction in the crushing ratio at a load of 35 MPa is observed with increasing sintering temperature. Specifically, for sample CA-1, the value decreases from 8.85 to 7.50%, for CA-2 from 8.50 to 7.11%, and for CA-3 from 8.11 to 6.58%. This indicates an enhancement in granule strength, which can be attributed to the formation of a denser and more homogeneous microstructure, as well as the development of the mullite phase, playing a key role in strengthening aluminosilicate ceramics.

A similar trend is observed for acid resistance: with increasing sintering temperature, the values improve (reflected by a decrease in acid solubility). For example, in sample CA-3, the value decreases from 6.74 to 5.56%, indicating enhanced chemical stability. This behavior can be explained by a reduction in the amorphous glassy phase and an increase in the degree of crystallinity of the material.

A comparative analysis of the samples shows that composition CA-3 exhibits the most favorable properties, characterized by the lowest bulk density combined with the lowest crushing ratio and high acid resistance. This suggests a more optimal phase composition and microstructure, providing a desirable combination of low density and high strength.

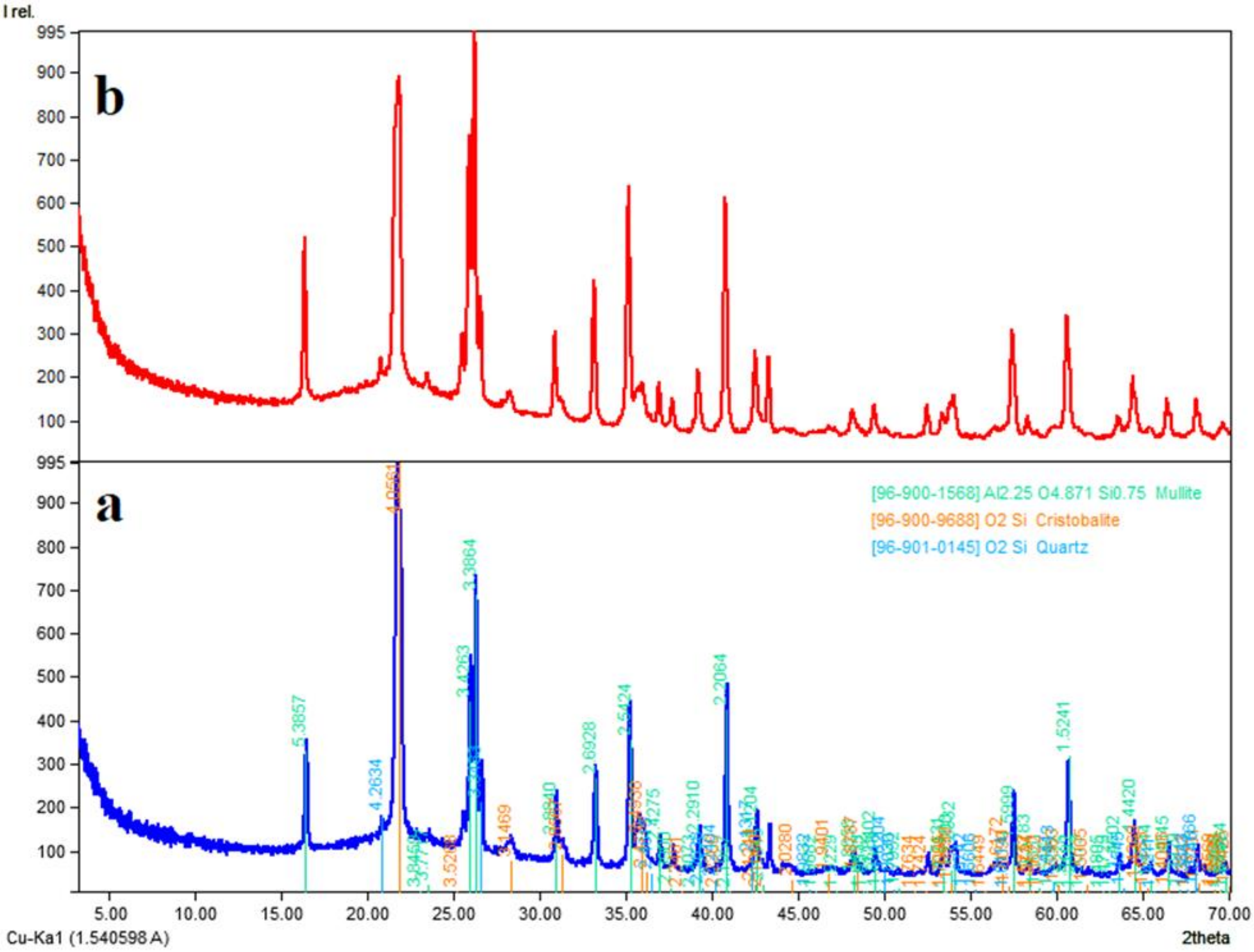

Fig. 1. XRD patterns of sample CA-3 sintered at 1200 °C (a) and 1250 °C (b) with a holding time of 60 min

X-ray diffraction analysis of sample CA-3 sintered at 1200 and 1250 °C with a holding time of 60 min revealed that, at 1200 °C, the material consists of quartz, cristobalite, and mullite phases. The relatively high intensity of quartz peaks indicates incomplete phase transformation and the initial stage of mullite formation. With an increase in temperature to 1250 °C, a noticeable increase in the intensity and sharpness of the diffraction peaks is observed, reflecting an enhanced degree of crystallinity and more pronounced formation of mullite as the dominant crystalline phase, while the content of residual quartz significantly decreases. These results suggest that increasing the sintering temperature to 1250 °C promotes more complete reactions between the initial components, leading to the formation of a more stable mullite-based structure and, consequently, improved performance characteristics of the material.

3. Conclusion

Lightweight aluminosilicate ceramic proppants were successfully synthesized using Angren kaolinitic clay and ash–slag waste as raw materials. The results demonstrate that increasing the sintering temperature from 1200 to 1250 °C significantly improves the physicomechanical properties of the proppants due to enhanced sintering, reduced porosity, and intensified mullite formation. A decrease in crushing ratio and acid solubility indicates improved mechanical strength and chemical stability of the materials. Among the investigated compositions, CA-3 exhibited the most optimal combination of properties, including low density and high strength, which makes it a promising candidate for practical applications in hydraulic fracturing. The use of industrial waste as a raw material not only reduces production costs but also contributes to environmental sustainability and resource efficiency. Thus, the developed proppants can potentially replace imported products while meeting industrial requirements.

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