Effect of Filler Particle Size on the Physical and Mechanical Properties of Ceramic Materials

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Abstract: The effect of a high-alumina filler on the physicomechanical properties of ceramic materials was investigated. It is shown that, by controlling the specific surface area of chamotte and the filler content, high-alumina ceramics can be obtained with sufficient mechanical strength and an optimized pore structure. Under these conditions, the fired ceramic or refractory material exhibits maximum mechanical strength and the highest resistance to failure under thermal loading.

Keywords: High-alumina filler, Chamotte, Kaolin, Alumina-containing waste, Pore structure, Physicomechanical properties, Specific surface area, Average pore diameter.

INTRODUCTION

It is known from classical silicate material technology [1-3] that high-alumina ceramic and refractory materials are characterized by a high aluminum-to-silicon oxide ratio and are widely used in high-temperature heating units and furnaces with periodic heating and cooling, as well as in various enterprises whose technological processes involve high temperatures. It should be noted that these high-alumina materials generally have a granular structure and are more heat-resistant than sintered ceramic and refractory products of similar composition [4-7].

Recently, the Republic of Uzbekistan has also devoted considerable attention to the development of high-temperature material compositions based on domestic raw materials, in particular high-alumina ceramics and refractories of corundum-mullite mineralogical composition for the production of various ceramic and refractory products for technical, construction, and household purposes [8-10].

However, in known studies [11, 12], the relationship between the porometric structure of the filler and the physicomechanical properties of high-alumina materials is considered primarily at a qualitative level, without a systematic quantitative comparison of the specific surface area of the filler, the average pore diameter, and the strength characteristics of the fired material. However, the functional dependence of the physicomechanical properties of fired (ceramic and refractory) materials on the pore diameter of the filler has not been sufficiently studied. Furthermore, the issue of using alumina-containing catalyst waste from the Shurtan Gas Chemical Complex as raw material for high-alumina filler has not been previously studied. This is the scientific novelty and difference of this work from previous studies: for the first time, a quantitative relationship has been established between the specific surface area of the fireclay filler obtained from man-made alumina-containing raw materials, the structure of the pore space and the mechanical strength of high-alumina ceramics, which made it possible to determine the optimal composition and simultaneously propose a practical solution for waste disposal, reducing the need for expensive imported technical alumina.

The aim of this work is to develop compositions and study the physical and mechanical characteristics of the porometric series of high-alumina ceramic materials depending on the granulometric composition (from the average pore diameter size) of the filler, in particular chamotte.

MATERIALS AND METHODS

A high-alumina filler obtained from a mixture of alumina and chamotte (alumina + kaolin) was used as the filler for the experimental mixtures. The chamotte was obtained by firing a mixture of kaolin and alumina-containing catalyst waste from the Shurtan Gas Chemical Complex.

To develop the filler charge composition, enriched AKF-78 kaolin from the Angren deposit was used, and spent alumina-containing catalyst from the Shurtan Gas Chemical Complex (SGCC) was used as the alumina.

The chamotte used was obtained from a raw mix of enriched AKF-78 kaolin and alumina-containing catalyst waste in an equal amount of components, *i.e.*, at a 50:50 ratio, then firing the mixture at 1100°C with a 0.5 hour hold at the final temperature.

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To prepare the ceramic The plastic mass was produced using variegated clays and secondary kaolins from the Angren deposit, as well as over 100% liquid glass by weight. It should be noted that the samples used from promising raw material deposits in Uzbekistan are suitable for developing the composition of high-alumina ceramic and refractory materials. Furthermore, it can be emphasized that the availability of a raw material base [13-15], in the form of natural clay raw materials or high-quality technical alumina, is a prerequisite for the serial production of in-demand high-alumina materials.

Given the fact that the high-alumina mineral raw materials available in the Republic cannot be used directly for the production of high-alumina ceramic and refractory materials, as they lack a stable chemical and mineralogical composition and contain numerous impurities, they require beneficiation [16, 17].

Furthermore, technologies based on the use of technical alumina are primarily used to develop the composition of high-alumina products for critical applications. However, the use of imported technical alumina is very expensive due to its high cost. Furthermore, domestic production of alumina from mineral raw materials is lacking. Therefore, the inclusion of alumina-containing catalyst waste from the Shurtan Gas Chemical Complex in the production of high-alumina ceramic and refractory materials is of practical importance. Its Al_2O_3 content (more than 85 wt.%, and after calcination, over 96 wt.%) is comparable to technical alumina. Since this secondary raw material is a cheaper man-made component for ceramic bodies, its use simultaneously addresses the issue of waste disposal from gas chemical production.

The porometric series of experimental high-alumina bodies was compiled based on the quantitative increase in chamotte content. The specific surface area of chamotte varied within the range of 0.44-2.88 m^2/kg. The specific surface area of chamotte was varied with a virtually constant ratio of SiO_2 and $SiO_2/(SiO_2+Al_2O_3)$ (0.2-0.3) in all bodies to ensure a uniform phase composition.

The concentration (porometric) series of materials were composed in such a way that the plastic part of the charge mass of samples of ceramic materials remained constant when the specific surface area of fireclay and the amount of the aluminous component changed, *i.e*. amount of filler.

Laboratory samples were molded plastically at a moisture content of the ceramic mass of 20-22%, then dried and fired in a silit furnace at a temperature of 1400°C with exposure for 2 hours at the final temperature in an oxidizing environment.

The specific surface area and average pore diameter of the filler were determined using a Quantachrome Nova 1000e static adsorption unit. Before measurements, the samples were pre-treated in vacuum at 100 °C for 12 hours. The nitrogen partial pressure values varied from 0.005 to 0.995 P/P0 for the nitrogen adsorption and desorption curves. The average pore diameter was calculated using the BET formula: Dav=4V/S, where V is the volume of adsorbed nitrogen. The specific surface area of the fireclay filler measured in this way is functionally related to the granulometric (fractional) composition of the fireclay used in the formation of the porosimetric range of ceramic and refractory materials. Since, as the content of the finely dispersed chamotte fraction in the filler composition increases, the specific surface area of o9the filler increases, which formed the basis for constructing the porometric series given below.

RESULTS AND DISCUSSION

The results of the analysis of the chemical composition of the raw materials used and the charge composition developed on their basis are presented in Table **1** and Table **2**, respectively.

It should be noted that the alumina (Al_2O_3) content in the spent catalyst waste from the Shurtan Gas

Table 1: Chemical Composition of the Raw Materials Used

Raw Material	Oxide Content, wt.%									*LOI,* wt.%
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MgO	CaO	Na_2O	K_2O	SO_3	
Enriched AKF-78	50,34	32,75	0,57	0,52	0,30	1,05	0,06	0,31	0,32	12,53
Angren secondary kaolin	67,40	18,28	3,14	0,58	0,34	0,55	0,35	0,25	1,18	9,35
Angren clay	52,10	25,67	5,77	0,38	1,19	1,10	0,95	0,91	1,71	10,16
Waste catalyst (SGCC)	6,03	85,25	1,68	0,90	1,27	1,25	0,53	0,15	<0,10	1,55
Burnt waste catalyst (SGCC)	0,18	98,77	0,24	0,31	0,29	0,09	0,05	0,04	weak	0,02

Note: loss on ignition (LOI): hygroscopic, constitutional, and crystallization water, organic and volatile substances, and carbon (IV) oxide.

Table 2: Developed Ceramic Mass Batch Composition

Raw materials, wt.%			
Mixtures of plastic ceramic mass			High-alumina filler
Enriched AKF-78 kaolin	Angren secondary kaolin	Angren clay	(alumina/chamotte)
40	10	5	45

Chemical Complex (SGCC) is above 85 wt.% and after calcination at 1300°C it increases to over 96 wt.%. The phase composition of the original waste consists of γ-alumina and gibbsite minerals, while the resulting calcination product mainly consists of high-temperature forms of aluminum oxide, *i.e.*, α-corundum (Al_2O_3).

Table **3** shows the results of the study of the physical and mechanical characteristics of the porometric series of filler using only a large fraction of chamotte, 1.0-2.0 mm in size.

It should be noted that when changing the content of the alumina component in the batch within the range of 5-30 wt.% and the specific surface area of the chamotte in the range of 0.44-2.88 m^2/kg, the maximum strength value of the M2 sample can be achieved with a ratio of alumina and chamotte of 25:10 with a chamotte fraction of about 2.5 mm.

Analysis of the data in Table **3** shows that with a consistent increase in the specific surface area of the chamotte filler from 0.44 to 2.88 m^2/kg (compositions M1-M7), the porosity of the samples as a whole decreases from 38.0 to 33.2%, while the apparent density remains virtually unchanged (2.01-2.04 g/cm^3), *i.e*. the change in properties is determined not so much by the total volume of pores as by their size and distribution. The average pore diameter changes non-monotonically: the minimum value (3.68 μm) was recorded for sample M2 (alumina:chamotte ratio = 25:10, specific surface area 0.83 m^2/kg), whereas with a further increase in the specific surface area of the filler (compositions M3-M7) the pore diameter increases to 10.42 μm. This explains why sample M2 has the highest compressive strength (55.1 MPa) and flexural strength (14.5 MPa) compared to other compositions: the combination of a moderate specific surface area of the filler and the smallest average pore diameter forms a more uniform and fine-pored structure of the shard, reducing the concentration of stress under mechanical loading. At alumina-to-chamotte ratios below 25:10 (compositions M3-M7), increasing the proportion of finely dispersed chamotte and the specific surface area of the filler leads to coarser pores and, consequently, to a decrease in compressive strength to 18.0-24.2 MPa and flexural strength to 7.8-10.8 MPa, despite similar values of total porosity and density. Thus, the alumina-to-chamotte ratio in the filler has a more pronounced effect on the physical and mechanical properties, depending on the pore structure, compared to the bulk porosity.

The Figure **1** shows the curve plotting the mechanical flexural strength, porosity, and average pore diameter of ceramic samples from the experimental mixes of the porometric series as a function of the alumina-to-chamotte ratio.

Table **3** and the Figure **1** show that the best mechanical strength corresponds to ceramic samples with average pore sizes up to 4 μm.

Thus, the analysis of the obtained results shows that the porosity values for the studied porosimetric series are quite high, *i.e*., in the range of 33.2-38.3%, and the pore sizes vary within the range of 3.68-10.4

Table 3: Physicomechanical Properties of Ceramic Mass Samples using Coarse-Fraction Chamotte

Mass index	Alumina/Chamotte ratio value	Specific surface area, m²/kg	Porosity, P, %	Density, ρ, g/cm³	Average pore diameter, Dav, μm	Flexural strength, σflex, MPa	Compressive strength, Rcomp, MPa
M1	30:5=6.0	0.44	38.0	2.01	4.41	13.4	27.2
M2	25:10=2.5	0.83	38.3	2.02	3.68	14.5	55.1
M3	20:15=1.33	1.20	35.7	2.03	4.40	10.2	18.2
M4	15:20=0.75	1.65	34.9	2.03	5.11	10.8	18.0
M5	10:25=0.4	2.04	33.2	2.04	6.50	9.7	23.8
M6	5:30=0.17	2.43	33.8	2.03	6.71	9.9	24.2
M7	0:35=0	2.88	33.4	2.02	10.42	7.8	18.5

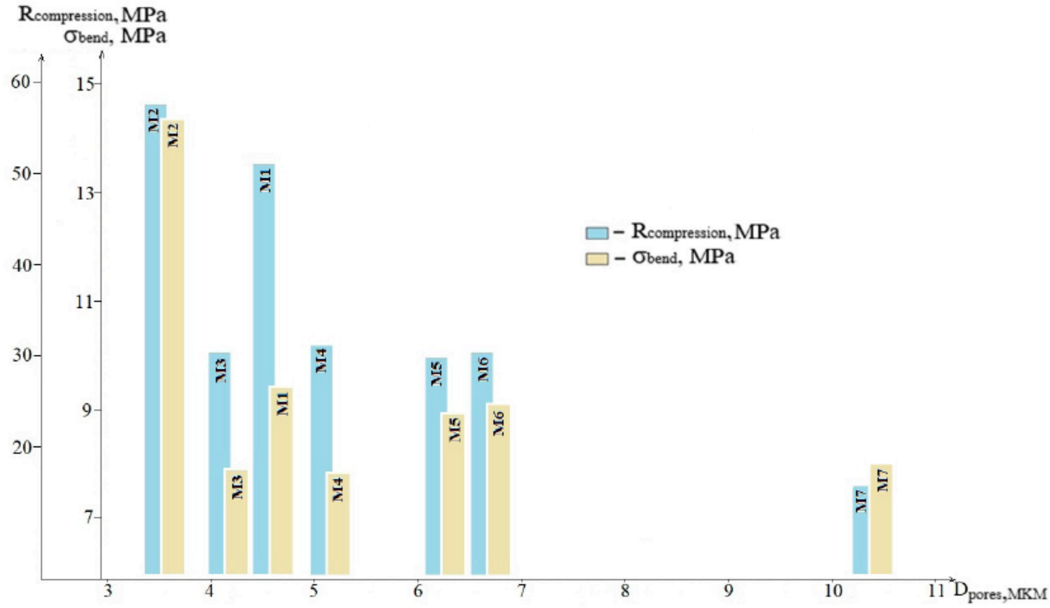

Figure 1: Dependences of mechanical compressive and flexural strength of a porometric series of samples on the average pore diameter of the filler.

µm. It is known [18-20] that the most favorable pore sizes in refractory materials are 2-4 µm, *i.e*., this structure of the pore space of the material is least susceptible to destruction under the action of thermal loads during the operation of refractories. For this reason, the sample from mass M2 was of interest in the development of the composition of general-purpose refractory materials.

CONCLUSION

The conducted studies showed that the optimal composition of the porometric series is sample M2, in which the alumina to chamotte ratio is 25:10 (specific filler surface area of 0.83 m^2/kg, average pore diameter of 3.68 µm), providing maximum compressive (55.1 MPa) and flexural (14.5 MPa) strength with a porosity of 38.3% and a density of 2.02 g/cm^3.

Overall, the obtained research results allow for the targeted selection of the parameters of ceramic or refractory materials for mechanical compressive and flexural strength in accordance with specified technical requirements by regulating the specific filler surface area and the fractional composition of the chamotte.

It should be noted that the results of thermal stability tests on the developed samples, particularly sample M2, showed an 8.0% strength loss after 5 thermal cycles, depending on their mechanical strength. These data also confirm the potential for using the formulation and particle size distribution of this mass in the production of refractory and ceramic materials, particularly for producing fireclay.

Thus, based on the experimental studies, it has been established that by adjusting the specific surface area of the filler, in this case, fireclay, it is possible to produce high-alumina ceramics with sufficient mechanical strength and an optimal pore structure, making this material the least susceptible to failure under thermal loads.

AUTHOR'S CONTRIBUTION STATEMENT

Eminov A.A. Conceptualization, Writing draft preparation, Investigation, Formal analysis, Visualization. **Eminov Al.A.** Data curation, Investigation. **Kadyrova Z.R.** Supervision, Validation, Reviewing and Editing. **Tairov S.S.** Visualization, Methodology, Software.

CONFLICTS OF INTEREST STATEMENT

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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