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Chemical and mineralogical composition of karnab kaolin raw material and its leaching methods

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Abstract: This article provides an in-depth study of the chemical and mineralogical composition of kaolin raw material from the Karnab deposit, its industrial applications, and beneficiation methods. The article describes effective techniques for removing harmful impurities present in Karnab kaolin, such as iron and titanium oxides, as well as alkali and alkaline earth metal oxides. The kaolin is bleached and its physical and optical properties are improved through the use of various chemical reagents (e.g., sulfuric, hydrochloric, and organic acids), flotation, hydrothermal, and autoclave processing technologies.

Keywords: Sanitary ceramics, kaolin, element, chemical, mineralogical, composition, beneficiation methods, oxalic acid.

Introduction: The issue of kaolin raw material quality remains a pressing problem, primarily due to the depletion or complete exhaustion of high-quality kaolin deposits. This situation directly affects the quality of the products manufactured from it. In recent years, a noticeable disparity has emerged between the increasing quality requirements for ceramic products—particularly sanitary ceramics—and the declining quality of available kaolin.

The term "kaolin" originates from the name of the Kao-Lin mountain in China and refers to a product of long-term weathering of rocks. Its chemical formula is $Al_2O_3{\cdot}2SiO_2{\cdot}2H_2O$, and in its natural form, it occurs as a mineral raw material containing various impurities such as feldspars, mica, iron and titanium oxides, and other minerals. In the composition of kaolin raw material, the kaolinite mineral dominates significantly in the mixture with quartz. It also contains remnants of unweathered primary rocks, alkali metal minerals, and iron oxide impurities [1].

The development of compositions for refractory materials requires the use of both primary and secondary kaolin. Kaolin serves as a key raw material in the production of fine ceramics, including porcelain and faience, as well as in the manufacture of white cement. In the chemical industry, kaolin is used for producing aluminum sulfate, aluminum oxide, and ultramarine pigment. It also functions as a carrier and filler for pesticides and fertilizers, and as a core material in catalysts for chemical reactions. Globally, a significant portion of kaolin is consumed in the paper industry, where it is used as a filler and brightening agent in paper production. Additionally, kaolin is widely applied as a filler in the manufacture of paints and coatings, rubber, plastics, adhesives, and perfumery products [2].

The Karnab kaolin deposit, located in the Samarkand region of the Pakhtachi district, 30 km southwest of the Ziyovuddin railway station and northeast of the Karnab village, is considered a promising kaolin source [3]. However, despite the large kaolin reserves, it is not widely utilized. The reason for this is that the kaolin raw materials are not sufficiently pure, leading several plants that use kaolin to rely on importing high-quality kaolin. As a result, the price of the final products significantly increases, and they struggle to find a place in the global market. For this reason, the study of kaolin beneficiation has always been a relevant area of research.

The low content of coloring oxides (Fe_2O_3, TiO_2), alkali oxides (K_2O, Na_2O), and alkaline earth metal oxides (CaO, MgO) in kaolin, along with the effective methods for removing them during the beneficiation process, indicates the potential for improving the quality of kaolin raw material. The crystal structure of kaolin consists of infinite layers based on SiO_4^{4-} tetrahedra. The kaolin crystal structure is composed of two layers: one is a silicon-oxygen tetrahedral layer, and the other is an alumino-oxygen hydroxyl octahedral layer. Kaolin has a hardness of 2-3 on the Mohs scale, a density of 2.58-2.63 g/cm^3, and when touched, it forms a greasy, clay-like mass. Under electron microscopy, fine hexagonal crystals are observed when highly magnified [5-6].

Chemical composition of the initial kaolin raw material from the Karnab deposit (mass. %)

Table-1

Samples	Oxide content (mass. %)										LOI, mass.%
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	SO_3	ZrO_2	FeO	K_2O	
KK-1	48,6	25,2	2,45	0,86	0,67	0,54	0,18	1,52	1,48	1,94	9,02
KK-2	51,3	27,6	1,95	0,78	0,36	0,75	0,36	1,16	1,89	1,75	8,73
KK-3	50,2	18,8	1,82	1,08	0,45	0,46	0,48	1,98	1,93	1,83	9,76
KK-4	49,7	28,3	2,16	1,12	0,78	0,89	0,56	2,08	1,65	1,98	8,44
KK$_{midl}$	49.9	24,9	2,09	0,96	0,56	0,66	0,39	1,68	1,73	1,87	8.98

Mineralogical composition of the initial kaolin raw material from the Karnab deposit: Kaolinite ($Al_2O_3{\cdot}2SiO_2{\cdot}H_2O$) d=0,566; 0,394; 0,255; 0,252; 0,237; 0,233; 0,198; 0,166; 0,148 nm; Pyrophyllite ($Al(OH)Si_2O_5$) d=0,102; 0,443; 0,333; 0,200; 0,185; 0,165 nm; Quartz (SiO_2) d=0,310; 0,283; 0,249; 0,245; 0,223 nm; Illite K< $(Al,Fe)_2[OH]_2(AlSi_3O_{10})nH_2O$ d=0,370; 0,343; 0,320 nm; Muscovite ($KAl_2(OH)_2Si_3AlO_{10}$) d=0,198; 0,181; nm; Biotite $K(Fe,Mg)_3[OH]_2(Al,Fe)Si_3O_{10}$) d=0,658; 0,265; 0,551 nm; Bementite ($Al_2O_3\cdot H_2O$) d=0,619; 0,233; 0,205; 0,166; 0,161; 0,153; 0,145 nm; Leucite ($K_2O\cdot Al_2O_4\cdot 4SiO_2$) d=0,534; 0,431; 0,413; nm; Orthoclase ($K_2O\cdot Al_2O_3\cdot 6SiO_2$) d=0,380; 0,185; 0,153; nm; Anorthite ($CaO\cdot Al_2O_3\cdot 2SiO_2$) d=0,934; 0,443; 0,320; 0,315; 0,283; 0,255; nm; Gallozaite ($Al_2O_3\cdot 2SiO_2\cdot 4\ H_2O$) d=0,980; 0,708; 0,601; 0,310 nm [4].

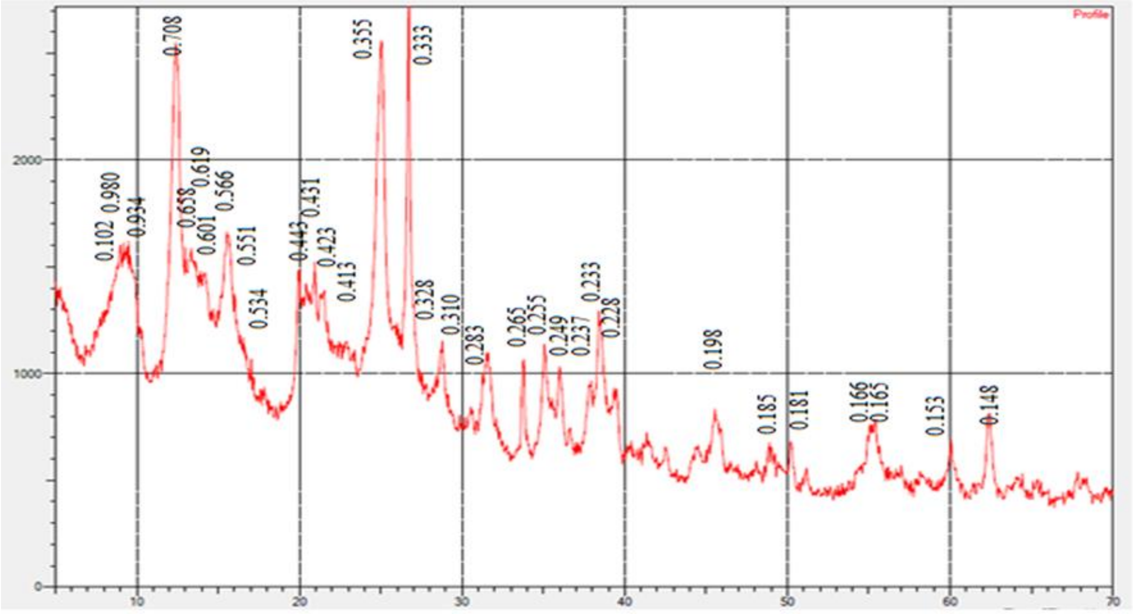

Figure 1. Mineralogical composition of the initial kaolin raw material from the Karnab deposit

Methods of purifying kaolin from harmful impurities

1. When thiomelamine is applied in a sulfuric acid environment ($KHSO_4$, H_2SO_4), kaolin products with a whiteness level of 90-94.2% are obtained.

2. In various types of foamy flotation, $CaCO_3$ (US patent No. 2990958) is used as a carrier for the particles.

3. In the formation of flocculation, half-valent cationic compounds are activated by processing in NH_4^+ salts (US patents No. 3371988; 3701417; 3837482; and 3862027).Dispersants such as Na_2SiO_3, PAN, and sodium hexametaphosphate are used. As a dispersing agent, Na_2SiO_3 should be applied at 1.0 kg/t and 0.25 kg/t NaOH with a pH range of 8.5-9.5. The clay is mixed with water for 6 hours. The disadvantage of this method is the low separation efficiency and yield.

4. In addition to $Ca(OH)_2$, 10% CH_3COOH acid, 0.05% polyacrylamide solution, potassium-aluminum bitterstones, and HCl-chloride acid mixtures are considered somewhat better as coagulants because the storage of lime slurry in warehouses for 1-2 months to fully convert to $CaCO_3$ is considered a weakness.

5. In the method of bleaching kaolin for use in the paper industry in Germany, a two-valent iron phosphate compound is formed in kaolin slurry, and the process is based on the effect of $Na_2S_2O_4$ solution in an acidic environment. The main disadvantages of chemical treatment are the complexity of the technological scheme and the reduction in kaolin quality at high temperatures.

6. In the reduction of Fe^{3+} to Fe^{2+} to remove iron oxides, a kaolin suspension is first prepared in an HCl-chloride acid environment. It is then vigorously mixed with periodically added HCl-chloride acid, followed by the addition of sodium hydrosulfite ($Na_2S_2O_4\cdot 2H_2O$) to the suspension.

7. In the reduction of Fe^{3+} to Fe^{2+}, a suspension of cationic compounds and sodium hydrosulfite is obtained in a sulfuric acid environment, and they make up 15% of the total solution. The disadvantage of this method is that the iron transitions into an isomorphic state and becomes incorporated into the clay lattice or the composition of silicate minerals.

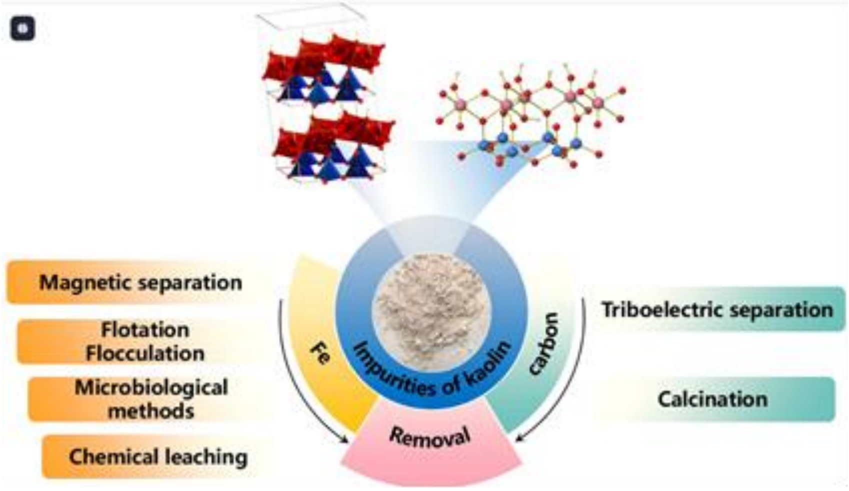

Figure 2. Methods of Kaolin Purification

8. The kaolin flotation chamber has a volume of 1 liter and operates in a mechanical machine with an impeller frequency of 2000 gr. The optimal flotation conditions are as follows: h:l = 1:4, sulfuric acid consumption is 0.8 kg/t, and collector ANP consumption is 0.9 kg/t. The concentrate yield is 63.2%. The concentrate composition contains high-quality Al_2O_3 at 36.12%. The clay product is dewatered in an autoclave at 2 atm pressure for 2 hours. Autoclave treatment reduces the iron content to 0.57%. The brightness level increases to 68-80%. The sulfuric acid consumption is 50 kg/t [7].

An easy and effective method of kaolin enrichment is the wet method, which involves the removal of harmful oxides in a hydrochloric acid medium. Two approaches using hydrochloric acid were studied: thermal boiling and autoclave hydrothermal methods.

THE EXPERIMENTAL PART

This article presents a method for removing iron from kaolin clays using oxalic acid, and the results obtained indicate the potential for its diverse industrial applications.

The raw kaolin (average particle size of 35 microns) from the Karnab deposit contained 2.09% iron oxide and had a whiteness level of 65.7%. The experiments were carried out in a 500 ml round-bottom flask equipped with a central heating and stirring system. For each experiment, 400 ml of oxalic acid solution ($C_2H_2O_4$, reagent grade) at various concentrations (0.01, 0.10, and 0.50 M) was added to the flask, and the desired temperature was set. Then, 40.0 g of clay was added under low-speed magnetic stirring (up to 600 rpm). At specific time intervals (5, 15, 30, 60, 90, and 120 minutes), 10 ml of solution was sampled and immediately centrifuged at 300 g for 15 minutes. A 5 ml aliquot of the centrifuged solution was taken to determine the total iron content. All washing tests were performed under atmospheric pressure. In general, a solid-to-liquid ratio of 10 g of kaolin to 100 ml of washing solution was used. Each experiment was conducted twice. The studied variables included oxalic acid concentrations (0.01, 0.10, and 0.50 M) and temperatures ranging from 25°C to 100°C.

In the solution, oxalic acid ($H_2C_2O_4$) dissociates to release a hydrogen ion and a hydrogen oxalate ion ($HC_2O_4^-$):

$$H_2C_2O_4 \rightarrow H^+ + HC_2O_4^-$$

Subsequently, the formed hydrogen oxalate ion further dissociates to release an oxalate ion ($C_2O_4^{2-}$):

$$HC_2O_4^- \rightarrow H^+ + C_2O_4^{2-}$$

Among these species, hydrogen oxalate (bi-oxalate) is the main compound responsible for the dissolution of iron:

$$Fe_2O_3 + H^+ + 5HC_2O_4^- \rightarrow 2Fe(C_2O_4)_2^{2-} + 3H_2O + 2CO_2$$

Therefore, the conditions of the medium must support the generation of $HC_2O_4^-$ ions to serve as an effective leaching agent. For this reason, evaluating the temperature and concentration of oxalic acid is essential.

The dissolution of iron (%) over time at 100°C was observed using different concentrations of oxalic acid (0.01, 0.10, and 0.50 M). An increase in iron dissolution was noted with increasing oxalic acid concentration. Within a period of 0 to 120 minutes (2 hours), a maximum dissolution rate of 98% was achieved. This behavior is attributed to the increased concentrations of oxalate and hydrogen ions associated with higher acid concentrations, which in turn promote the formation of hydrogen oxalate ions ($HC_2O_4^-$)

The increase in hydrogen ions, which directly affects iron dissolution and pH, is consistent with the findings reported by Ambikadevi and Lalithambika in 2000. They identified oxalic acid as the most effective acid for dissolving iron (in the form of goethite and hematite, unlike in this study) from kaolin minerals, and concluded that increasing the acid concentration (from 0.05 to 0.15 M) significantly improves the efficiency of iron dissolution.

Kaolin concentrates obtained using these methods meet the requirements of the second-grade GOST 21286–82 standard, making them suitable for porcelain materials and sanitary-ceramic products. To obtain first-grade kaolins, 300 ml of water is added to 100 grams of raw kaolin. Then, 4 grams of various acids, such as tartaric, citric, oxalic, and ascorbic acids, are added depending on the process, forming different complex compounds, and sulfuric acid (H_2SO_4) is added until the pH reaches 2.5. After calcination, the whiteness of the ceramic body is 89-92%. The sulfuric acid treatment follows the reactions below [8].

$2FeS_2+14H_2SO_{4(k)}\rightarrow Fe_2(SO_4)_3+15SO_2\uparrow+14H_2O$

$Mn_2O_3+6H_2SO_{4(50\%\text{-li sov.})}\rightarrow 2Mn_2(SO_4)_3+3H_2O$

$MnO_2+6H_2SO_{4(k)}\rightarrow 2Mn_2(SO_4)_3+O_2\uparrow+6H_2O\ (t\leq 110^{\circ}C)$

$2NaHSO_3+2Fe_2O_3+3H_2SO_4\rightarrow Na_2SO_4+2FeSO_4+4H_2O$

$Na_2S_2O_4+3Fe_2O_3+5H_2SO_4\rightarrow Na_2SO_4+6FeSO_4+5H_2O$

$TiO_2+2H_2SO_4\rightarrow Ti(SO_4)_2+2H_2O$

Chemical composition (mass %) of the kaolin from the Karnab deposit after leaching

Table-2

Samples	Oxide content (mass. %)										LOI, %
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	SO_3	ZrO_2	FeO	K_2O	
KK-1	54,2	35,4	0,36	0,32	0,37	0,15	0,09	0,68	0,76	0,65	7,13
KK-2	52,3	37,2	0,72	0,28	0,34	0,18	0,12	0,52	0,54	0,72	7,68
KK-3	55,5	36,3	0,63	0,36	0,29	0,25	0,11	0,80	0,68	0,82	8,14
KK-4	53,7	35,8	0,56	0,41	0,27	0,19	0,07	0,47	0,59	0,59	7,83
KK_{midl}	53,9	36,1	0,56	0,34	0,31	0,19	0,09	0,61	0,64	0,69	7,69

CONCLUSİON

In conclusion, a deep analysis of the composition of kaolin ore from the Karnab deposit and its enrichment reveals the potential for producing high-quality ceramic products. The research shows that the raw kaolin contains iron, titanium, potassium, magnesium, and other impurities, which limit its industrial use. Therefore, the article extensively analyzes various effective cleaning and enrichment methods, including chemical treatment in sulfate and chloride acid media, flotation, and autoclave hydrothermal processes.

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