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ILMIY AXBOROTNOMA

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SYNTHESIS AND PHYSICOCHEMICAL ANALYSIS OF ZEOLITE FROM LOCAL KAOLIN

Sh.Magdiev, F.Majidova, A.Nasimov, Kh.Tashpulatov, D.Toshpulatov, Sh.Mirzaev, K.Norboev, O.Normuradov
Samarkand State University named after Sharof Rashidov, Samarkand, Uzbekistan.

Sh.Magdiev Samarkand State University named after Sharof Rashidov, Samarkand, Uzbekistan.

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Abstract. In this research work, the mass (%) reduction of Fe_2O_3 in kaolin samples was achieved using various methods including mechanical, thermal, and chemical methods. Iron compounds were reduced from the sample by reacting with hydrochloric acid at certain temperatures. The samples were weighed and analyzed after each process using elemental analysis (EDX). In this process, maintaining the ratio of SiO_2 and Al_2O_3 was focused. After reducing the mass (%) of iron compounds content from the sample, the sample was processed in the hydrothermal reactor with $Ca(OH)_2$ and left in a drying cabinet for different periods of time from 72 to 96 hours. After the reaction time, it was cooled to room temperature at ± 5 °C every 1 hour. Powder X-ray diffraction (XRD) was taken and analyzed to determine the phases and crystallinity.

Keywords. EDX, hydrothermal method, Kaolin, XRD, zeolite.

Mahalliy kaolindan olingan seolitning sintez va fizik-kimyoviy tahlili

Annotatsiya. Ushbu tadqiqot ishida kaolin namunalarida Fe_2O_3 ning massa kamayishi (%) mexanik, termik va kimyoviy usullar yordamida erishildi. Temir birikmalari ma’lum haroratlarda xlorid kislotasi bilan reaksiyaga kirishib, namunadan qaytarildi. Har bir jarayondan so‘ng elementar tahlil (EDX) yordamida namunalar tortildi va tahlil qilindi. Bu jarayon SiO_2 va Al_2O_3 nisbatlarini saqlashga qaratilgan. Namunadagi temir birikmalarining massasi (%) kamaytirilgandan so‘ng, namuna gidrotermik reaktorda $Ca(OH)_2$ bilan ishlov berildi va quritish pechida turli vaqtlarda 72 dan 96 soatgacha qoldiriladi. Reaksiya vaqtidan keyin har 1 soatda xona haroratiga ±5 °C ga sovutildi. Fazalar va kristallikni aniqlash uchun kukun rentgen nurlari difraksiyasi yordamida tahlil qilindi.

Kalit so'zlar: ED RFS, gidrotermik usul, kaolin, retngen difraksiyasi, seolit.

Синтез и физико-химический анализ цеолита из местного каолина

Аннотация. В этом исследовании массовое содержание (%) Fe_2O_3 в образцах каолина было уменьшено с помощью механических, термических и химических методов. Соединения железа были удалены из образца путем реакции с соляной кислотой при определенных температурах. После каждого процесса образцы были взвешены и проанализированы с помощью элементного анализа (EDX).

Этот процесс был направлен на поддержание пропорций SiO_2 и Al_2O_3. После снижения массы (%) соединений железа в образце, он был обработан $Ca(OH)_2$ в гидротермальном реакторе и оставлен в сушильной печи на разное время от 72 до 96 часов. После времени реакции он был охлажден до комнатной температуры ±5 °C каждый час. Порошковая рентгеновская дифракция (XRD) была использована для определения фаз и кристалличности.

Ключевые слова. ЭД РФС, гидротермальный метод, каолин, рентгеновский дифракция, цеолит.

Introduction. Kaolin is mainly kaolinite from a mineral consisting of natural mud or soil It is often white-colored, red-colored soft, and plastic material [1]. The main chemical composition of kaolin consists of aluminum oxide (Al_2O_3) (around 39%), silicon dioxide (SiO_2) (around 46%), and water (H_2O) (around 14%). Kaolin is originally white in color possessing kaolin brightness [2].

Most research has been focused on using in the industry as an important mineral and physicochemical and mineralogical studies [3] Natural kaolin also contains iron(III) oxide (Fe_2O_3), which is responsible for its intensity yellowish color. The existence of such mixtures causes the quality of kaolin and expense to fall so for. This iron(III) oxide should be eliminated and several works dedicated to this issue especially focusing on chemical treatment [4]. Kaolin is also subjected to mechanical enrichment, calcination, and chemical processes to obtain zeolites [5].

Zeolites are either natural or synthetic aluminosilicates (alumosilicates). Zeolites have become very important industrial materials because of their unique properties as pore size tunability and ion-exchange ability and such unique features caused them to find their place in various fields [6-7]. In agriculture, zeolites are used to improve soil quality, because of controllable and slow moisture evaporation and they allow plants to absorb major nutrient substances [8]. Zeolites are also excellent catalysts in petrochemical and chemical processes [9]. Thus zeolites are used to purify hydrogen, methane, and carbon dioxide because they effectively adsorb gases. Moreover, they also used gas storage and separation processes which make them crucial materials [3,10]. Because of their high surface area, gases and liquids adsorb on the zeolite material. Ionic particles can be effectively segregated in zeolites from solutions, particularly ions of heavy metals and radionuclides, and these made possible application in water sewage treatment.

High-pressure gas density increases because of the adsorption coefficient increase possible [11]. Zeolites dimensions and charge allow for selective adsorption of known ions or molecules, making them useful in catalytic processes and separation mixtures [12]. Thermal stability is high at stable temperatures, which provides opportunities for their thermal impact in processes [13]. In cracking, isomerization, and alkylation processes, hydrated zeolites apply their own cations to replace melted substances, making them effective for water and soil cleaning. Alkaline and alkaline-earth cations of elements (sodium, potassium, calcium) are used in the form of dry powdery reagents or prepared solutions, with clay (kaolin) being attached [14-15].

Zeolites features, their synthesis methods, and possible applications were studied. Zeolites are examined for synthesis through various methods, each corresponding to different zeolite structures that facilitate adaptation. Hydrothermal Synthesis: This method involves high pressure and temperature, mixing silicates and aluminosilicates in a liquid solution [16]. This method is recognized for its widespread use in zeolite synthesis. Solution synthesis involves controlling reagent concentrations in experimental solutions. Reaction methods can be accelerated through microwave synthesis and enhanced efficiency via ultrasonic synthesis, among other experimental approaches [5]. Each of these methods has unique characteristics and enables the optimization of various types of zeolites [17].

Research method. After taking local white kaolin soil, grinding it into a powder in a mortar, and determining its composition using a Rigaku EDX elemental analyzer to begin impurity content (such as iron(III) oxide significant amount of Al_2O_3. Initially., 20 g of kaolin on an analytical balance. The weighed kaolin was then placed in a heat-resistant conical flask, to which 35 ml of concentrated HCl and 35 ml of distilled water (H_2O) were added.

After stirring on a magnetic stirrer at 80 °C and 100 rpm in a reflux condenser for 4-5 hours, the resulting sample was filtered through ordinary filter paper in hot water and subsequently dried in a heating oven at 145 °C for 4-5 hours. The dried product was analyzed for its composition using an elemental analyzer. Subsequently, aliquots were obtained from the precursor and reaction products in each step to attain initial results. For this Fourier resonance infrared spectrophotometer (FTIR) IRAffinity 1S (Shimadzu, Japan) and powder X-ray diffractometer (XRD) Maxima X7000 (Shimadzu, Japan) were used. In the next steps, 1.5 g of kaolin was weighed and added to 0.5 g of $Ca(OH)_2$ and mixed in 1.5 ml of H_2O. The mixture was placed in a reactor and heated to 130 °C for 72 hours in the oven (Fig). After cooling, the calcined kaolin underwent elemental analysis, FTIR, and XRD analysis, and we compared the initial analysis results.

Research results.

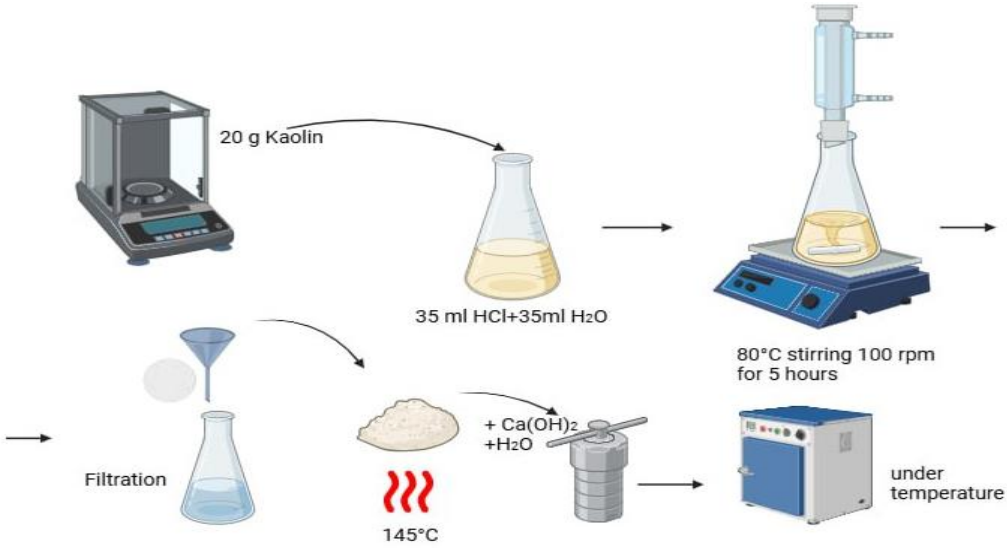

FIGURE 1. Zeolite praparation process from local kaolin

Initially chemical content, e.g. precursor composition is given in Table 1. EDX analysis results of the kaolin containing characteristic elemental peaks can be seen below (Fig. 2). Elemental analysis shows that the sample contains Fe_2O_3 (5.29 %), SiO_2 (60.5%), and Al_2O_3 (21.7%). The abundance of silicon dioxide and aluminum oxide approximately 3:1 ratio should be maintained throughout thermal, mechanical, and chemical work treatments.

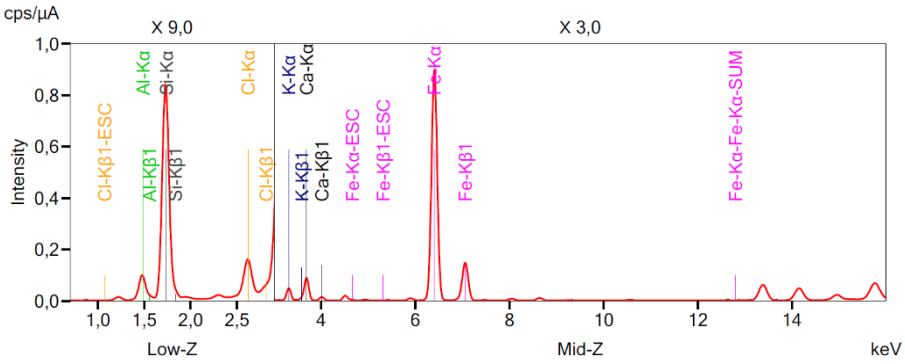

FIGURE 2. XRF spectrum of kaolin sample

After initial chemical treatment with concentrated hydrochloric acid, the content of Fe_2O_3 reduced from 5.29% to 4.978% Fe_2O_3, and the final sample contains 0.312 %. Meanwhile SiO_2 content increased from 60.50 % to 88.90 % and aluminum oxide content decreased 7.58% (Fig. 3).

TABLE 1

No.	Component	Mass %	Element line	Intensity (cps / µA)
1	Fe_2O_3	5.29	M:Fe -Kα	27.437
2	Al_2O_3	21.70	L:Al -Kα	5.389
3	Cl	0.46	L:Cl -Kα	9.313
4	K_2O	4.97	M:K -Kα	1.141
5	SiO_2	60.50	L:Si -Kα	46.280
6	CaO	7.04	M:Ca -Kα	2.198

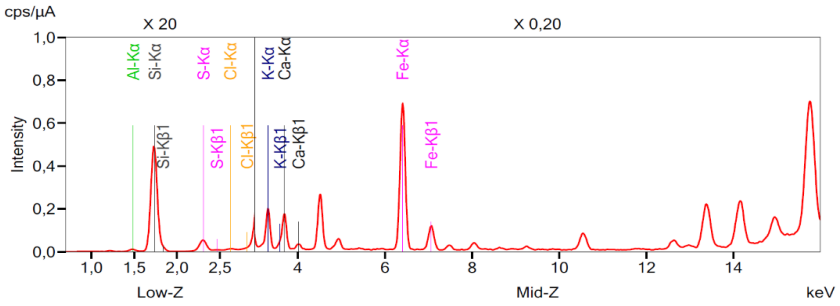

FIGURE 3. XRF spectrum of hydrochloric acid treated kaolin sample

TABLE 2

No.	Component	Mass %	Element line	Intensity (cps / µA)
1	Fe_2O_3	0.312	M:Fe -Kα	1.379
2	Al_2O_3	7.58	L:Al -Kα	1.235
3	Cl	0.04	L:Cl -Kα	0.566
4	K_2O	1.98	M:K -Kα	0.285
5	SiO_2	88.90	L:Si -Kα	60.299
6	CaO	1.18	M:Ca -Kα	0.258

Synthesis of zeolite was performed with calcium oxide, which required a relatively high cleaning process. This was due to the reaction involving calcium hydroxide in the initial sample, containing substances Al_2O_3 and SiO_2, which reacted to form a typical zeolite structure, indicating that the process was successful.

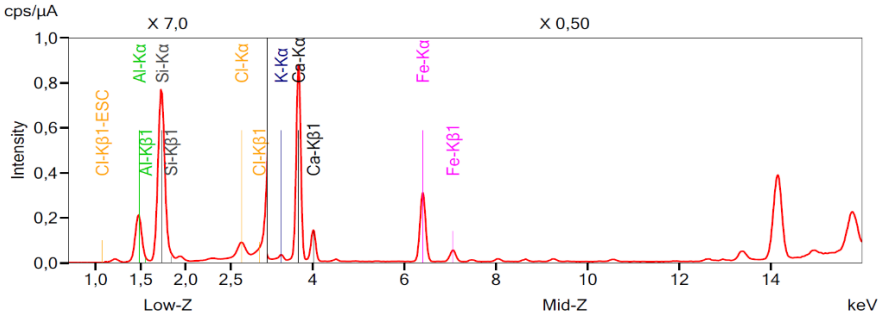

FIGURE 4. XRF spectrum of slaked lime treated kaolin sample

TABLE 3

No.	Component	Mass %	Element line	Intensity (cps / µA)
1	Cl	0.150	L:Cl – Kα	3.294
2	CaO	11.850	M:Ca – Kα	3.717
3	K_2O	0.405	M:K – Kα	0.085
4	Fe_2O_3	0.325	M:Fe – Kα	1.558
5	Al_2O_3	36.500	L:Al – Kα	8.438
6	SiO_2	50.770	L:Si – Kα	32.858

To evaluate the crystallinity of the final product, powder XRD analysis was performed. The analysis results is given in Fig. 5. From the results following conclusions made:

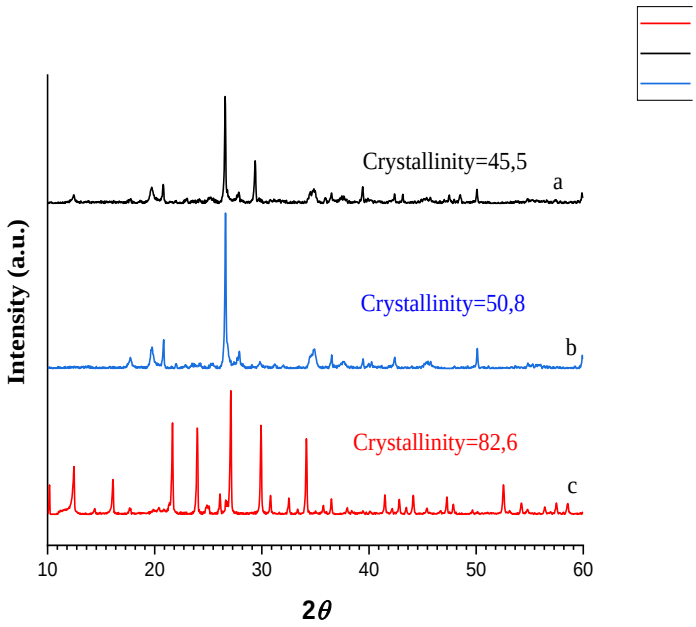

Figure 5. Powder XRD pattern of obtained zeolite sample

a) – in the XRD pattern reflection due to (001), (020), (110), (002), (111) planes are clearly resolved. This sample crystallinity is found to be 45.5% and 54.5% amorphous aggregation remaining.
b) – in hydrochloric acid treated sample, there are not many reflection changed. But crystallinity increased to 5.3%.
c) – in calcium hydroxide (slaked lime) treated sample, the diffraction pattern greatly changed. Thus the following diffraction angles show well -resolved reflection peaks: 12.4; 15.98; 21.62; 24.03; 26.07; 27.13; 29.86; 30.83; 32.47; 34.22; 36.45; 41.50; 42.56; 44.11; 47.22; 47.89; 52.56; 54.20; 56.63; 57.49; 58.47. Moreover, the crystallinity also significantly improved and reached 82.6 % of and remaining 17.4% amorphous.

Conclusions. The local kaolin contained SiO_2 (60.5%) and Al_2O_3 (21.7%) found to be suitable raw materials because of, an approximate 3:1 ratio. Local kaolin was treated mechanically, thermally, and chemically to improve its quality. Particularly, Fe_2O_3 content reduction was maintained as a major issue. To eliminate iron content, samples treated with hydrochloric acid and reduced zeolite samples were achieved. Moreover, slaked lime treatment leads to a more organized zeolite structure. The obtained zeolite synthesis route is confirmed to be the plausible way with future perspectives. Zeolites prepared using this method can be used in various applications including gas and petroleum refinery, and sewage water treatment.

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