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Preparation of Zeolite from Recycled Alumina and Modified Bentonite

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This study investigated the organic constituents of alumina waste generated as a byproduct of polyethylene production. Organic compounds adsorbed on spent hexane-saturated alumina were recovered using Soxhlet extraction, followed by solvent evaporation, derivatisation and IR spectroscopic analysis. The results confirmed the presence of diverse organic functional groups on the alumina surface. The principal objective was to establish a feasible route for synthesising artificial zeolite from recycled alumina for efficient natural gas purification. In this context, the spent alumina was evaluated as a secondary raw material and its suitability for zeolite preparation was scientifically validated. In addition, bentonite samples collected from four locations of the Azkamar deposit in the Navoi region were systematically modified and characterised. Their potential use as binder components during zeolite granulation was assessed, with particular emphasis on their effects on granule mechanical strength and structural stability. The chemical compositions of the raw and modified spent alumina and Azkamar bentonite samples were compared using X-ray fluorescence analysis. These findings demonstrate that the production of zeolitic materials for gas purification from recycled industrial resources offers significant scientific value and strong practical potential.

Keywords: Aluminum oxide, Polyethylene synthesis, Synthetic zeolite, Bentonite modification, H-form bentonite, Adsorbent.

INTRODUCTION

Adsorbent materials are widely used in catalysis, chemical processing, environmental remediation and gas purification [1]. Continuous advances in sorption technology have promoted the development of materials with improved composition, morphology and surface properties to enhance adsorption efficiency and operational performance [2,3]. Among these, composite-active materials have gained increasing attention because their matrix-based architecture combines mechanical strength with functional fillers, enabling the design of durable and application-oriented adsorbents [4-6]. The matrix phase plays a key role in determining important properties such as strength, ductibility and resistance to mechanical or hydraulic stress.

The growing demand for advanced adsorbents is further driven by the need for efficient purification systems and protective technologies operating under harsh conditions. High performance sorbents are expected to provide high adsorption capacity, rapid mass transfer, mechanical stability and low toxicity during prolonged use [7-10]. These requirements have encouraged the exploration of alternative raw materials for zeolite production with improved economic viability and environmental sustainability.

Synthetic zeolites are among the most important industrial adsorbents due to their well-defined microporous frameworks, high specific surface area, ion-exchange capacity and molecular sieving properties [11-15]. These characteristics make them highly effective for drying, purification and selective separation of gases and liquids. In addition, they play a vital role in petrochemical refining and catalytic conversion processes [16-18]. Their tunable pore architecture and chemical composition further extend their applications to wastewater treatment, metal recovery and heterogeneous catalysis [19-22].

Modern materials research increasingly focuses on the reducing raw material consumption, minimizing industrial

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waste and converting secondary resources into value-added products. In this context, the synthesis of zeolites from low-cost aluminosilicate precursors has received considerable interest. Hydrothermal conversion of kaolinite and related minerals has been extensively investigated because of the availability and low cost of these materials [23,24]. However, natural kaolin often contains impurities such as Fe, Ti, Mn, Ca, Mg and Ni, which may adversely influence crystallinity, hardness, catalytic behaviour and production efficiency of the resulting zeolite materials [25,26].

Ziegler-Natta catalyst remains among the most efficient and widely used catalytic systems for polyethylene manufacture [27-29]. During polymer processing, alumina is commonly applied as an adsorbent to remove the residual catalyst fragments, organic additives and catalyst poisons from polyethylene products [30-32]. Once saturated, the spent alumina is separated as a secondary industrial byproduct that may be regenerated or redirected into other technological applications [33-35]. Therefore, investigation of the physico-chemical properties of spent alumina and its utilisation as a precursor for new functional materials has become an important scientific and technological objective [36,37].

The utilisation of waste alumina for zeolite production offers an eco-friendly and economical route for advanced adsorbent manufacture, while reducing waste and producing efficient materials for gas treatment and related separations. This study focused on converting spent alumina into artificial zeolite as a value-added adsorbent for natural gas purification. The discarded alumina was investigated as an alternative precursor material and its applicability for zeolite synthesis was confirmed through physico-chemical evaluation. To improve shaping and performance characteristics, bentonite samples obtained from the Azkamar deposit were modified and characterised for possible use as binder materials during zeolite preparation.

EXPERIMENTAL

Spent γ-alumina (Al_2O_3) was obtained as an industrial by-product from the flue-gas treatment section of a petrochemical plant and used as the alumina precursor. Its oxide composition was first determined by X-ray fluorescence (XRF). Sodium bentonite (montmorillonite-rich clay) was collected from the Azkamar deposit (Dashkesan region), crushed to fine particles below 5 mm and sieved through a mesh sieve for use as the binder source.

All reagents were used as received. Analytical-grade *n*-hexane (≥99%, Merck, USA) was employed for Soxhlet extraction of residual hydrocarbons from spent alumina. Conc. HCl (33%, Merck, USA) was used for bentonite purification, while NaOH pellets (≥98%, Merck, USA) were used to prepare alkaline synthesis media. Commercial sodium silicate solution (~30 wt.% SiO_2, 10 wt.% Na_2O, Sigma-Aldrich, USA) served as silica source. FTIR-grade KBr was used when pellet preparation was required. Deionized water (resistivity >18 MΩ·cm) was used throughout all washing and dilution steps.

Characterization: Elemental oxide compositions were measured using an energy-dispersive XRF spectrometer. The functional groups in extracted organic residues were analysed using a Bruker FTIR spectrometer over the range 4000-400 cm^{-1} region. Mixing operations were carried out using a high-speed overhead stirrer (up to 1200 rpm). Total dissolved solids and pH were monitored using a digital TDS/pH meter. Drying was conducted in a convection oven maintained at 105 ºC. Grinding was performed using a planetary ball mill followed by sieving through a 100 μm mesh. Hydrothermal synthesis was carried out in a Teflon-lined stainless-steel autoclave.

Pretreatment of spent alumina: Raw spent alumina, containing more than 95% Al_2O_3 with minor impurities, was purified before synthesis. Approximately 50-100 g of material was loaded into a Soxhlet thimble and extracted with *n*-hexane for 6-8 h to remove residual oils and hydrocarbons. After solvent removal, the recovered solid was dried at 105 ºC and calcined at 250-300 ºC for 2-4 h. The calcined product was then milled for 1-2 h and sieved to below 100 μm. This purified powder was used as the aluminum source for zeolite preparation. The appearance of the processed samples is shown in Fig. 1, while the XRF results after extraction and calcination are summarised in Table-1 and Fig. 2.

Fig. 1. Appearance of aluminum oxide

TABLE-1
RESULTS OF CHEMICAL ELEMENT ANALYSIS OF CALCINED Al_2O_3

No.	Burnt Al_2O_3	Stat. Err
1	Al_2O_3	97.8 mass%
2	V_2O_5	0.447 mass%
3	SiO_2	0.355 mass%
4	TiO_2	0.73 mass%
5	CaO	0.114 mass%
6	SO_3	0.101 mass%
7	Fe_2O_3	0.0428 mass%
8	Ga_2O	0.0104 mass%
9	NiO	0.0104 mass%
10	Cr_2O_3	0.0101 mass%
11	ZnO	0.0073 mass%
12	CuO	0.0047 mass%
13	SnO_2	0.0042 mass%
14	TeO_2	0.0017 mass%
15	Dy_2O_3	ND mass%
16	MnO	ND mass%
17	Ag_2O	0.0006 mass%
18	ZrO_2	0.0006 mass%
19	MoO_3	0.0005 mass%
20	Ta_2O_5	ND mass%

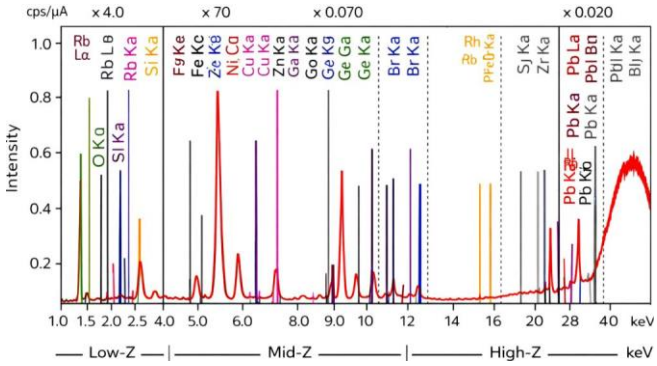

Fig. 2. X-ray fluorescence of calcined aluminum oxide

Purification of bentonite: Natural bentonite from the Azkamar deposit was selected as a low-cost binder because of its swelling behavior and plasticity. Approximately 7.1 kg of bentonite was dispersed in 70 L of water at a solid-to-liquid ratio of 1:10 and stirred for 3 h at 1200 rpm using a Stegler mixer. The initial total dissolved solids (TDS) of the suspension, measured using an HM Digital TDS-3 meter, was 1050 mg/L. Carbonate impurities were removed by gradual addition of 33% HCl until gas evolution ceased. The TDS increased to 6000 mg/L, indicating conversion of poorly soluble carbonate phases into soluble chloride salts.

The suspension was then washed with distilled water through repeated cycles of agitation, settling and decantation until neutral pH was reached and the TDS decreased to 0-100 mg/L. The purified slurry was filtered through filter cloth for 3 days and dried at 105 ºC for 12 h in a GX30B drying oven to obtain H-form modified bentonite. The raw and treated samples were characterized by X-ray fluorescence (XRF) using an EDAX instrument, and the results are presented in Table-2.

Post-treatment analysis showed decreases of approximately 70% in Ca and 81% in Cl contents, confirming effective removal of exchangeable ions and purification of the clay matrix.

TABLE-2
X-RAY FLUORESCENCE (XRF) OXIDE COMPOSITION OF BENTONITE BEFORE AND AFTER MODIFICATION

No.	Oxide component	Modification (mass %)	
		Before	After
1	SiO_2	64.6	68.5
2	Al_2O_3	16.4	17.6
3	Fe_2O_3	8.06	7.86
4	CaO	3.84	1.15
5	K_2O	3.03	3.04
6	TiO_2	1.10	1.09
7	SO_3	0.568	0.205
8	V_2O_5	0.106	0.107
9	Cr_2O_3	0.0476	0.0472
10	ZrO_2	0.0370	0.0312
11	MnO	0.0325	0.0288
12	NiO	0.0207	0.0281
13	SrO	0.0362	0.0193
14	Rb_2O	0.0201	0.0183
15	ZnO	0.0149	0.0147
16	BaO	0.0135	0.0121
17	SnO_2	0.0135	0.0095
18	Co_2O_3	0.0119	(0.0092)
19	CuO	(0.0082)	0.0073
20	Tm_2O_3	0.0082	Not detected

Values in parentheses indicate trace concentration close to the detection limit.

These changes improved the swelling behaviour, surface reactivity and ion-exchange capacity of bentonite, enhancing its suitability as a binder for zeolite granulation. The modified bentonite was finally milled and sieved to below 0.1 mm prior to use.

Zeolite synthesis: Zeolite was synthesized through an alkali hydrothermal route. Sodium hydroxide was dissolved in deionized water to prepare an 8-10 M solution. Pretreated alumina was added under vigorous stirring to generate sodium aluminate. Sodium silicate solution was then introduced slowly while mixing continuously. The molar composition of the gel was adjusted Na_2O:Al_2O_3:SiO_2 in a 2:1:1 ratio. The reaction gel was stirred for 1 h, transferred to a Teflon-lined autoclave, and crystallized at 90-100 ºC for 24-48 h. After cooling, the solid product was filtered, washed to neutral pH and dried at 100 ºC.

Granulation with bentonite binder: The synthesized zeolite powder was blended with purified bentonite (5-15 wt.%) and sufficient water to obtain a plastic mass. The mixture was extruded or pelletized into granules of approximately 3-5 mm diameter. Green pellets were dried at 110 ºC for 12 h and then calcined at 500-550 ºC for 4 h to improve mechanical strength. The resulting zeolite-bentonite composite pellets were stored for subsequent adsorption studies.

RESULTS AND DISCUSSION

The XRF analysis of the spent γ-alumina collected from the flue-gas treatment unit confirmed that the material was predominantly composed of Al_2O_3 (90.3 wt.%), together with minor levels of SiO_2 (1.72 wt.%), Cl (1.27 wt.%), V_2O_5 (0.657 wt.%), TiO_2 (0.165 wt.%) and trace metallic oxides.

These results demonstrate that the waste alumina remains a highly valuable aluminium-rich precursor suitable for reuse in zeolite synthesis rather than disposal.

After Soxhlet extraction with *n*-hexane followed by calcination at 250-300 ºC, the Al_2O_3 content increased markedly to 97.8 wt.%, while silica and other impurities were significantly reduced. This enrichment indicates successful removal of adsorbed hydrocarbons, volatile contaminants and surface residues. The purification step therefore converted an industrial waste stream into a chemically suitable feedstock for aluminosilicate synthesis. The increase in alumina purity is particularly important because aluminium atoms substitute for silicon in the zeolite framework during hydrothermal crystallization, generating negatively charged lattice sites responsible for cation exchange and adsorption behaviour [38]. Thus, the recovered alumina offers both environmental and economic advantages as a substitute for commercial aluminium sources.

FTIR analysis of the *n*-hexane extract revealed the absorption bands associated with long-chain hydrocarbons and functional groups corresponding to lauryl nitrile, polyethylene derivatives, 1-dodecanethiol and 1-hexanol (Fig. 3). These compounds are consistent with residues originating from polyethylene processing additives, lubricants, catalyst poisons or polymer fragments adsorbed during plant operation. Their presence confirms that the spent alumina had acted as an adsorptive scavenger during petrochemical processing [39]. However, the removal of these organics before zeolite synthesis was essential because residual carbonaceous species could inhibit crystallization, block pores or reduce final adsorption capacity.

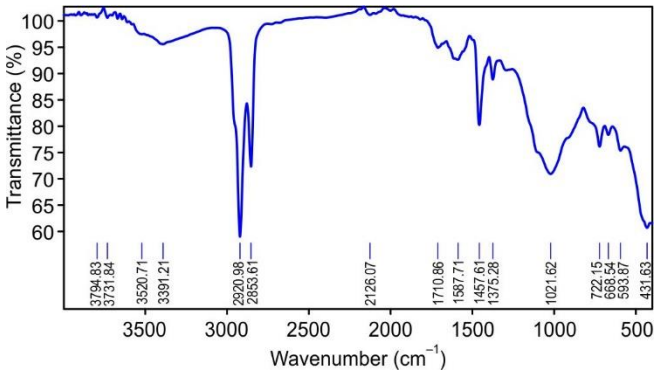

Fig. 3. Infrared spectrum of aluminum oxide extract

Accordingly, the extraction-calcination sequence is a key activation stage in valorizing the waste alumina. Furthermore, untreated bentonite commonly contains carbonate minerals and exchangeable salts that can reduce granule quality and interfere with zeolite performance. Acid treatment with 33% HCl effectively removed these impurities, as indicated by gas evolution during treatment and the rise in TDS from 1050 to 6000 mg/L due to conversion of insoluble carbonates into soluble chlorides. Subsequent washing reduced the TDS to 0-100 mg/L and the neutral pH obtained, confirming the successful elimination of dissolved salts. XRF analysis showed that CaO decreased from 3.84 to 1.15 wt.% (about 70% reduction), while sulfate-containing species also decreased. Meanwhile, SiO_2 increased from 64.6 to 68.5 wt.% and Al_2O_3 from 16.4 to 17.6 wt.%, reflecting the purification and enrichment of the bentonite phase. The decrease in calcium content is especially beneficial since replacement of exchangeable Ca^{2+} ions with H^+ expands the clay interlayers, improves swelling and enhances the surface activity. These changes increase adhesion between zeolite particles during pelletization and improve granule strength without introducing excessive inert matter.

The purified alumina and modified bentonite together create an advantageous raw material system for zeolite manufacture. The alumina provides a concentrated aluminium source, while sodium silicate supplies silica under alkaline hydrothermal conditions. Since the spent alumina already contains minor silica and transition-metal traces, it may also promote nucleation and framework development during crystallization. The chosen synthesis ratio of $Na_2O:Al_2O_3:SiO_2$ = 2:1:1 falls within the range commonly used for zeolite A-type materials, suggesting that the prepared gel composition was appropriate for forming crystalline sodium aluminosilicate phases. The use of waste-derived alumina therefore demonstrates a technically feasible route for partial replacement of conventional high-purity reagents.

For industrial fixed-bed applications, modified zeolite as adsorbent was obtained in granular form with sufficient mechanical resistance and low fines generation. Furthermore, modified bentonite used in this study acted as an efficient inorganic binder, enhancing pellet integrity through improved swelling behaviour and stronger particle-to-particle adhesion during extrusion and calcination. Removal of carbonate matter and coarse crystalline impurities during acid activation minimized weak points within the pellet matrix and reduced the probability of pore blockage. This enabled the production of compact and structurally uniform granules. Therefore, the resulting zeolite-bentonite composite is expected to exhibit improved compressive strength, reduced abrasion tendency and prolonged service life under cyclic adsorption conditions.

Conclusion

This study demonstrated an effective route for converting spent γ-alumina generated from petrochemical flue-gas treatment into a valuable precursor for synthetic zeolite production. XRF analysis confirmed that the waste material was rich in Al_2O_3, and after solvent extraction and calcination, the alumina content increased to 97.8 wt.%, indicating successful removal of organic and inorganic contaminants. The treated alumina therefore represents a suitable and economical alternative to conventional commercial aluminium sources for zeolite synthesis. FTIR investigation of the extracted organic fraction revealed the presence of hydrocarbon-based compounds associated with polymer-processing residues, confirming the adsorptive role of alumina during industrial process. Their removal prior to hydrothermal synthesis was essential to improve precursor purity and ensure effective zeolite crystallisation. Natural bentonite obtained from the Azkamar deposit was successfully modified through acid treatment and washing, resulting in significant reductions in Ca and Cl contents. These changes enhanced the swelling behaviour, surface activity, and ion-exchange characteristics of the clay. As a consequence, the modified bentonite functioned as an efficient inorganic binder for zeolite, improving pellet cohesion and expected

mechanical durability without excessive pore blockage. The hydrothermal processing of the purified alumina with sodium silicate under alkaline conditions confirmed the technical feasibility of producing zeolitic materials from recycled industrial waste.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language. The authors reviewed and edited the content and take full responsibility for the published work.

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