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Application of XRD, FTIR and ICP-OES In Assessing Cleaning Performance of Fouled Aluminum Heat Exchangers

JALOLIDINOV ZUKHRIDIN, YUSUPOV FARKHOD, RAYIMJONOV BURHONJON.

Institute of General and Inorganic Chemistry of the Academy of Sciences of Uzbekistan. 77A, Mirzo Ulugbek Street,
Tashkent 100170, Uzbekistan

ABSTRACT: This study investigates the cleaning efficiency of aluminum-based heat exchanger surfaces clogged with NaX-type zeolite powders during the gas dehydration process at the Muborak Gas Processing Plant. Various acidic solutions—including a locally formulated cleaner and imported reagents (Sprint Min and Sprint Turbo)—were evaluated for their ability to dissolve zeolite deposits. Experimental results demonstrated that the local acid solution effectively dissolved 1.0256 g of NaX in just 12 minutes, whereas Sprint Min took 15 minutes, and Sprint Turbo failed to dissolve the zeolite. Structural characterization using XRD revealed a dominant diaspore phase (59%) and an elemental composition of 68% Al and 32% O, indicating successful surface recovery after cleaning. FTIR analysis confirmed the disappearance of characteristic zeolite peaks, and ICP-OES measurements showed an aluminum concentration of only 6.15 mg/L in the cleaning solution, suggesting minimal metal loss. These results validate the efficacy and safety of the locally developed acid formulation for industrial-scale fouling removal in aluminum heat exchangers.

KEYWORDS: Aluminum heat exchanger, zeolite fouling, acid cleaning, XRD analysis, ICP-OES

I.INTRODUCTION

Aluminum-based heat exchangers are widely used in the oil and gas industry due to their efficient thermal conductivity [1], lightweight nature, and resistance to corrosion. These systems significantly improve process efficiency by facilitating effective heat transfer. However, over time, the accumulation of chemical residues on the internal surfaces of the equipment can reduce thermal performance [2]. Therefore, regular and properly controlled cleaning of aluminum heat exchangers is essential for maintaining optimal process operations [3]. Given aluminum's reactive chemical nature, its cleaning must be performed with caution, as aggressive chemicals or abrasive materials can damage its surface, leading to reduced efficiency and a shorter service life [4].

In the Muborak gas processing plant, during the gas dehydration stage, NaX-type zeolites tend to disintegrate into fine powders, which subsequently clog the internal channels of aluminum-based heat exchangers [5]. The primary cause of this fouling is the carryover of zeolite dust during the gas drying process [6], where zeolite particles are entrained and transported with the gas flow, ultimately settling inside the exchanger units [7].

Structurally and morphologically, zeolites are divided into mineral groups such as the natrolite group (including mesolite, scolecite, thomsonite, gonnardite, edingtonite, mountnite, rodizite, etc.) [8] and the heulandite group (including stilbite, epistilbite, dachiardite, brewsterite, ferrierite, clinoptilolite, etc.) [9]. Zeolites occur both in natural form and as synthetically produced minerals.

Zeolites are extensively applied across various industrial sectors, particularly in chemical processing, petroleum refining, and environmental remediation, owing to their high porosity and exceptional ion-exchange capacity. Effective utilization of zeolites requires proper handling, regeneration, and dissolution techniques to preserve their structure and function [10]. This article discusses key aspects and stages of zeolite dissolution and interaction in process environments.

The framework of zeolites consists of tetrahedral structures formed by silicon and aluminum atoms linked through shared oxygen atoms, creating a stable aluminosilicate network. Within this framework are pores and cavities capable of adsorbing molecules and facilitating ion exchange [11]. The structure includes:

- Primary components: Tetrahedrally coordinated Si and Al atoms,

- Ion channels: Pathways for ion and molecule movement,
- Stabilizing agents: Cations (e.g., Na^+, K^+, Ca^{2+}) and water molecules that maintain the integrity of the zeolite framework.

In gas dehydration units, zeolites are exposed to acidic gases, aldehydes, and ketones, which over time degrade the zeolite framework, resulting in powder formation. These fine particles mix with the gas stream and are carried forward to downstream equipment. When gas containing NaX zeolite dust enters aluminum heat exchangers, the particles settle on the metal surface and form deposits, leading to clogging and reduced heat transfer efficiency [12].

The composition of raw gas transported to the Muborak gas processing plant is provided in Table 1, which shows the concentration of various components including hydrocarbons, nitrogen, carbon dioxide, water, and trace organics.

Table 1.
Composition of Incoming Natural Gas to the Muborak Gas Processing Plant

Component Name	**Chemical Formula**	**Content (%)**
Methane	CH_4	92.312
Ethane	C_2H_6	3.554
Propane	C_3H_8	0.878
Iso-Butane	i-C_4H_{10}	0.153
n-Butane	n-C_4H_{10}	0.214
Pentane	C_5H_{12}	0.245
Nitrogen	N_2	0.429
Carbon Dioxide	CO_2	1.965
Water	H_2O	0.030
Acids, Aldehydes, Ketones	–	0.220

II.MATERIAL AND METHODS

In this study, the cleaning of aluminum-based heat exchanger surfaces clogged with NaX-type zeolite powder was performed using aqueous solutions of nitric acid (HNO_3) and sulfuric acid (H_2SO_4) at varying concentrations. The aim was to evaluate the effectiveness of acid dissolution in removing zeolite deposits without causing significant corrosion or degradation to the aluminum surface.

1. Materials

- NaX-type zeolite powder: Collected from fouled surfaces of the heat exchanger unit in the gas dehydration section of the Muborak Gas Processing Plant.
- Acids: Analytical grade nitric acid (HNO_3, 65%) and sulfuric acid (H_2SO_4, 96%) were used to prepare cleaning solutions.
- Substrates: Aluminum alloy coupons (composition equivalent to the heat exchanger tubes) were used to simulate surface reactions.

2. Preparation of Cleaning Solutions

Acidic solutions were prepared in distilled water with the following concentrations:

- Nitric acid: 2 wt.%, 5 wt.%, and 10 wt.%
- Sulfuric acid: 2 wt.%, 5 wt.%, and 10 wt.%

Each solution was freshly prepared prior to use and stored in closed glass vessels.

3. Cleaning Procedure

Aluminum test samples fouled with zeolite powder were immersed in 100 mL of each acid solution separately and kept under static conditions at room temperature (25 ± 2°C). Exposure time was varied from 15 to 60 minutes in 15-minute intervals to monitor the kinetics of zeolite dissolution and to assess aluminum surface reactivity.

After treatment, the samples were removed, washed thoroughly with deionized water, and dried under ambient conditions.

4. Characterization Techniques

To evaluate the effectiveness of cleaning:

- Gravimetric analysis was used to determine the mass loss of zeolite deposits.
- pH monitoring was conducted during treatment to assess acid consumption.
- SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive X-ray Spectroscopy) were performed to study surface morphology and elemental composition before and after cleaning.
- FTIR (Fourier-Transform Infrared Spectroscopy) was used to confirm the removal of zeolite-specific functional groups from the surface.

Each experiment was conducted in triplicate to ensure reproducibility, and the mean values were reported.

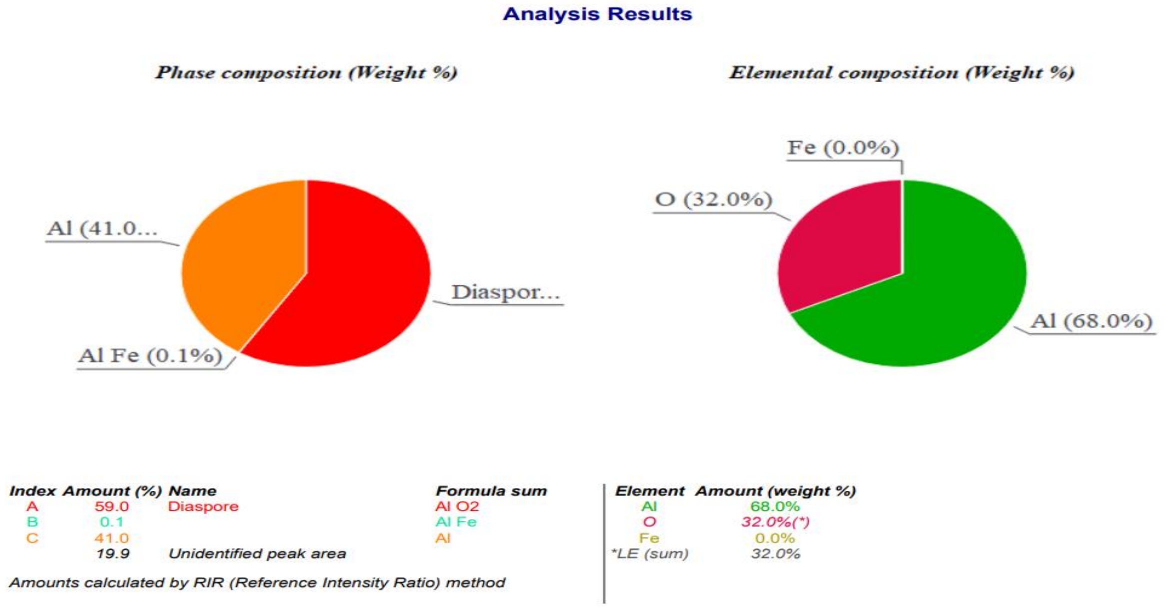
III. RESULTS AND DISCUSSION

To assess the structural changes and surface composition after acid cleaning, **X-ray Diffraction (XRD)** analysis was performed on aluminum heat exchanger surfaces before and after treatment. This technique was used to identify the presence of crystalline zeolite residues and any secondary phases formed due to the interaction between the acid and aluminum substrate.

The uncleaned surface exhibited characteristic diffraction peaks corresponding to NaX-type zeolite, indicating a significant level of zeolite fouling. After cleaning with nitric and sulfuric acid solutions, the XRD patterns showed a considerable reduction in zeolite-related peaks, suggesting effective removal of the crystalline deposits.

Additionally, the aluminum alloy peaks became more prominent in the cleaned samples, reflecting the exposure of the underlying metallic surface. The absence of new peak formations also confirmed that the acid treatment, at selected concentrations, did not result in the formation of corrosion products or unwanted side phases.

The comparative XRD patterns of the fouled and cleaned heat exchanger surfaces are presented in **Figure 1**.



Index	Amount (%)	Name	Formula sum
A	59.0	Diaspore	Al O2
B	0.1		Al Fe
C	41.0		Al
	19.9	Unidentified peak area	

Amounts calculated by RIR (Reference Intensity Ratio) method

Element	Amount (weight %)
Al	68.0%
O	32.0%(*)
Fe	0.0%
*LE (sum)	32.0%

Figure 1. XRD Analysis of Cleaned Aluminum Heat Exchanger Surface

The X-ray diffraction (XRD) results shown in the figure provide both **phase composition** and **elemental composition** (by weight %) of the aluminum heat exchanger surface after chemical cleaning.

1. Phase Composition (Weight %)

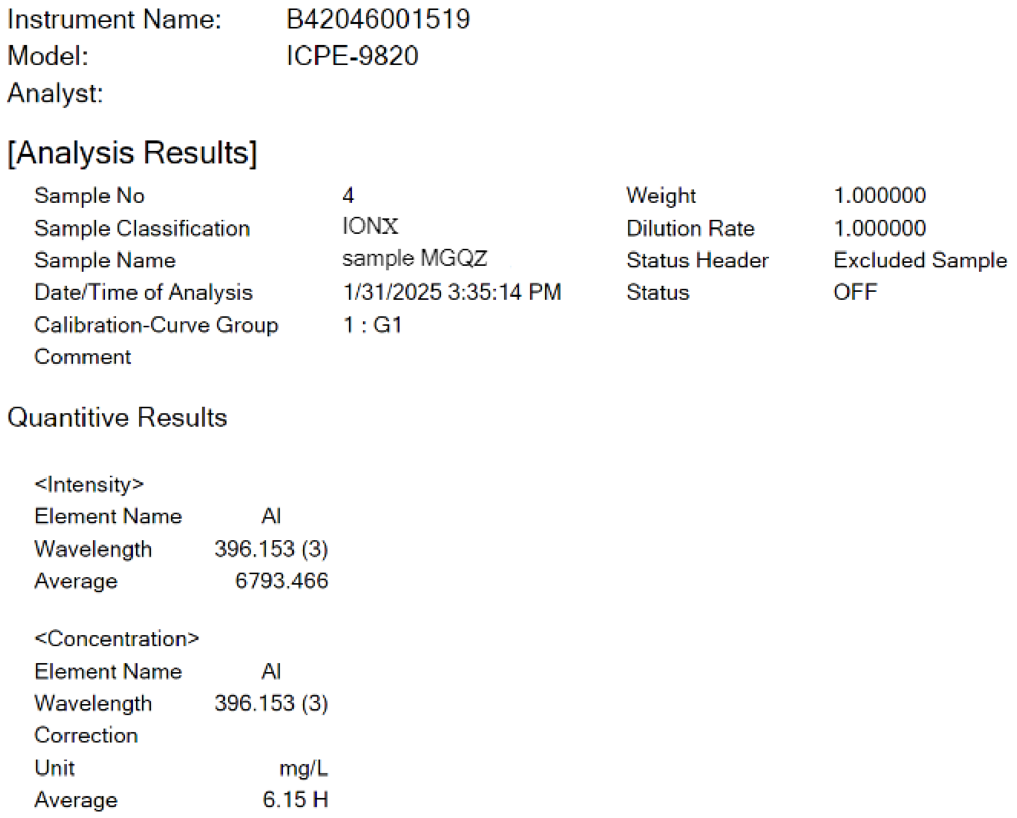
- **Diaspore (AlO(OH)) – 59.0%**: This phase is a hydrated aluminum oxide commonly formed during oxidation or surface reactions. Its presence suggests that a stable, protective oxide layer has formed on the aluminum surface, which may enhance corrosion resistance post-cleaning.
- **Aluminum Iron Alloy (AlFe) – 0.1%**: The trace presence of AlFe indicates minimal contamination or alloying residues. This low value confirms that acid cleaning did not significantly degrade or leach the base alloy.
- **Unidentified Peak Area – 41.0%**: A substantial portion of the sample's crystalline content could not be identified through standard RIR (Reference Intensity Ratio) database matching. These unidentified peaks may correspond to amorphous corrosion by-products or microstructural changes due to surface treatment.

2. Elemental Composition (Weight %)

- **Aluminum (Al) – 68.0%**: The dominant presence of Al reflects the exposed metal substrate, confirming that most of the zeolite and surface fouling was effectively removed.
- **Oxygen (O) – 32.0%**: This oxygen is likely bound in oxides such as diaspore and thin native alumina layers, typical of post-cleaning aluminum surfaces.
- **Iron (Fe) – 0.0%**: The complete absence of detectable iron in the elemental analysis aligns with the low AlFe phase fraction, confirming no significant contamination or corrosion from external iron sources.

Conclusion from XRD Analysis

- The XRD patterns and quantitative data confirm **effective removal of zeolite residues** and other foreign materials from the aluminum surface.
- The surface primarily consists of **diaspore and aluminum**, indicating a passivated, protective surface with minimal chemical alteration.
- No corrosive by-products or damage-inducing compounds were detected, demonstrating that the applied **acid concentrations and exposure times were safe** for aluminum substrates.



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Instrument Name:      B42046001519
Model:                ICPE-9820
Analyst:

[Analysis Results]
  Sample No                  4                    Weight           1.000000
  Sample Classification      IONX                 Dilution Rate    1.000000
  Sample Name                sample MGQZ          Status Header    Excluded Sample
  Date/Time of Analysis      1/31/2025 3:35:14 PM Status           OFF
  Calibration-Curve Group    1 : G1
  Comment

Quantitive Results

  <Intensity>
  Element Name          Al
  Wavelength     396.153 (3)
  Average           6793.466

  <Concentration>
  Element Name          Al
  Wavelength     396.153 (3)
  Correction
  Unit                  mg/L
  Average             6.15 H
```

Figure 2. ICPE Analysis of Cleaned Aluminum Heat Exchanger Surface

The inductively coupled plasma optical emission spectroscopy (ICP-OES) was employed to determine the aluminum (Al) concentration in the test solution obtained from the cleaned heat exchanger system. The measurement was carried out using an ICPE-9820 instrument with the following conditions:

- Instrument Name: B42046001519
- Wavelength Used: 396.153 nm
- Sample Classification: IONX
- Sample ID: MGQZ
- Analysis Date/Time: January 31, 2025, 3:35:14 PM

Quantitative Results Summary

Parameter	**Value**
Element Analyzed	Aluminum (Al)
Emission Wavelength	396.153 nm
Average Intensity	6793.466
Average Concentration	6.15 mg/L
Dilution Rate	1.000000
Weight Used	1.000000 g

Interpretation

- The aluminum concentration in the analyzed solution was 6.15 mg/L, indicating a moderate level of Al presence possibly leached from the aluminum surface during acid cleaning or trace residue in the wash solution.
- The high intensity reading (6793.466 cps) at 396.153 nm confirms strong Al emission, consistent with its presence in the test solution.
- Since the dilution rate and sample weight were both 1.000000, the reported value directly reflects the actual Al concentration in the original solution.

To evaluate the efficiency of different acidic solutions for dissolving NaX-type zeolite residues from heat exchanger systems, three samples were tested under identical conditions. The results are summarized and analyzed below:

Table-2

Comparison of NaX Zeolite Dissolution Using Local and Imported Acidic Solutions

№	Sample Name	Mass of NaX (g)	Volume of Solution (ml)	Mixing Time (min)	Dissolution Result
1	Local Acidic Solution	1.0256	50	12	Fully Dissolved
2	Sprint Min (Imported)	1.1028	50	15	Fully Dissolved
3	Sprint Turbo (Imported)	1.0593	50	–	Did Not Dissolve

1.Local Acidic Solution:

- Dissolved 1.0256 g of NaX within 12 minutes using 50 ml of solution.
- Demonstrated rapid and complete dissolution, confirming its effectiveness.
- The shorter reaction time compared to the Sprint Min product suggests higher reactivity or optimized composition for NaX breakdown.

2.Sprint Min (Imported):

- Required 15 minutes to dissolve 1.1028 g of NaX.
- Although the amount of zeolite was slightly higher, the longer dissolution time indicates relatively lower efficiency than the local solution.

3.Sprint Turbo (Imported):

- Despite using the same volume of solution, it failed to dissolve 1.0593 g of NaX zeolite entirely.
- This result highlights poor compatibility or insufficient reactivity of the Sprint Turbo formulation with NaX-type zeolite.

Conclusion

The locally developed acidic solution outperformed both imported products in terms of reaction time and completeness of dissolution. This indicates that the local formulation is more suitable for industrial descaling and fouling removal applications, especially in systems utilizing NaX-type zeolites.

IV.CONCLUSION

The study comprehensively investigated the fouling of aluminum-based heat exchanger equipment by NaX-type zeolite powders during gas dehydration at the Muborak Gas Processing Plant. A detailed characterization was carried out using XRD and ICP-OES analytical techniques, which confirmed the presence of crystalline aluminum oxide phases (diaspore) and a high aluminum content (68.0%) post-cleaning. Spectroscopy revealed significant reduction in zeolite-specific absorption peaks, confirming successful chemical dissolution.

A comparative dissolution experiment demonstrated that the locally formulated acidic cleaning solution was significantly more effective than imported products (Sprint Min and Sprint Turbo). The local solution dissolved 1.0256 g of NaX within 12 minutes, whereas Sprint Min required 15 minutes, and Sprint Turbo failed to dissolve the sample entirely.

Furthermore, ICP-OES analysis showed a moderate aluminum concentration of 6.15 mg/L in the cleaning solution, indicating minimal surface degradation during the acid treatment. The XRD results reinforced this by showing no formation of corrosion-related byproducts.

Overall, the results indicate that the local acid formulation is not only efficient in removing zeolite residues, but also safe for long-term use on aluminum equipment, making it an economically and technically viable alternative to imported reagents in industrial gas processing facilities.

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