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INTERPHASE PARTITIONING OF IRON AND ALUMINUM DURING ALKALINE PROCESSING OF ARAL SEA SAPROPEL

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Abstract

Aims: To determine how potassium hydroxide concentration, temperature, and liquid-to-solid ratio affect pH and the transfer of aluminum and iron from sapropel into the liquid phase, and to quantify retention of both elements in the residual solid under resource-efficient conditions.

Study Design: Factorial laboratory screening of three potassium hydroxide concentrations (1, 2, and 3%). – P. six temperatures (40–90 °C). – P. and five liquid-to-solid ratios (1:1–3:1).

Place and Duration of Study: Sapropel was collected from the dried bed of the Aral Sea, Uzbekistan, and processed at the Department of General Sciences, Asia International University, Bukhara, Uzbekistan. The experimental dates were not documented in the source record.

Methodology: Air-dried, ground sapropel was treated with aqueous potassium hydroxide under continuous mixing. The separated liquid phase was analyzed potentiometrically for pH and instrumentally for aluminum and iron. Liquid-phase transfer was calculated from the element mass in solution relative to its initial mass in the sapropel.

Results: Across all tested conditions, pH ranged from 6.53 to 10.92, aluminum transfer from 0.72 to 14.20%, and iron transfer from 2.5 to 9.7%. Aluminum mobilization increased sharply at 3% potassium hydroxide and pH above 9.3. Under the selected conditions of 2% potassium hydroxide, a liquid-to-solid ratio of 2.5:1, and 80 °C (pH 8.66). – P. 3.19% of aluminum and 5.0% of iron entered the liquid phase; 96.81% and 95.0%, respectively, remained in the solid.

Conclusion: Moderate alkalinity preserved most aluminum- and iron-bearing mineral components while supporting humic-substance processing. The residue is therefore a plausible precursor for sorbent development, although adsorption capacity, kinetics, selectivity, and regeneration must be established directly before application.

Keywords: sapropel; potassium hydroxide; aluminum; iron; phase partitioning; solid residue; sorbent precursor; Aral Sea

1. Introduction

Alkaline extraction of humic and fulvic substances from sapropel collected on the dried bed of the Aral Sea produces two material streams: a liquid phase enriched in soluble humic fractions and a mineral-rich solid residue. Using both streams would support a low-waste valorization pathway. Potassium hydroxide is particularly relevant when the liquid product is intended as a fertilizer precursor, while the residual solid may provide a locally available adsorbent feedstock (Cheng et al., 2019; Platonova & Adeeva, 2018).

Toxic elements such as lead, cadmium, copper, zinc, and arsenic remain important contaminants of industrial and agricultural waters. Adsorption is widely investigated for their removal because it can be operationally simple and compatible with low-cost mineral materials. Iron and aluminum (hydr)oxides are especially relevant: surface hydroxyl groups can bind cations and oxyanions through inner-sphere complexation, ion exchange, and coprecipitation (Hira et al., 2023; Hua et al., 2012; Mashale et al., 2025). Magnetite, goethite, hematite, and ferrihydrite have accordingly been studied as active phases or components of sorbent systems (Boruah et al., 2023; Das et al., 2011).

Retention of iron and aluminum during alkaline treatment is therefore important to the potential value of the sapropel residue. Aluminum hydroxide is amphoteric: sparingly soluble $\text{Al}(\text{OH})_3$ can form soluble aluminate species, principally $[\text{Al}(\text{OH})_4]^-$, as alkalinity increases. The solubility of aluminum hydroxide rises substantially in strongly basic media (Gayer et al., 1958). Iron(III)-bearing phases are comparatively persistent over the moderately alkaline range studied, although their mineralogical form and transformation behavior depend on pH, temperature, and reaction time (Das et al., 2011). A process optimized only for liquid-phase humic recovery could consequently overlook loss of mineral adsorption sites from the solid product.

This study evaluated the effects of potassium hydroxide concentration, temperature, and L/S ratio on pH and the interphase partitioning of aluminum and iron during sapropel processing. The specific objectives were to quantify the fractions entering the liquid phase, identify conditions that preserve both

elements in the solid, and define the scientific basis for subsequent sorbent-development studies. No direct adsorption test was performed; therefore, the residue is evaluated only as a potential sorbent precursor.

2. Methodology

2.1 Study Material and Sample Preparation

The study material was sapropel sample S-0 collected from the dried bed of the Aral Sea. The sample was air-dried, ground, and homogenized to obtain a uniform material before treatment.

2.2 Alkaline Processing

Sapropel was treated with aqueous KOH solutions at concentrations of 1, 2, or 3%. The investigated L/S ratios were 1:1, 1.5:1, 2:1, 2.5:1, and 3:1, and the treatment temperatures were 40, 50, 60, 70, 80, and 90 °C. Continuous mixing was maintained during treatment. The suspension was then separated into liquid and solid phases by filtration or centrifugation. Treatment duration was held constant across runs, but its numerical value was not available in the source record.

2.3 Analytical Measurements and Mass Balance

The pH of the separated liquid phase was measured potentiometrically. Aluminum and iron concentrations in solution were determined using the laboratory's instrumental analytical workflow based on atomic absorption spectrometry and spectrophotometry. The source record did not preserve instrument models, calibration ranges, detection limits, or the allocation of individual samples between the two analytical techniques; these details should be supplied from the laboratory log before peer-review submission.

The fraction of each element transferred to the liquid phase was calculated as:

$$E = (msol / m0) \times 100\%, \quad (1)$$

where E is the fraction transferred to the liquid phase (%). – P . $msol$ is the mass of the element in solution, and $m0$ is its initial total mass in the sapropel. Retention in the solid phase was calculated as $100 - E$ (%). The mass-balance approach follows established quantitative characterization principles for humic and mineral materials (Swift, 1996).

2.4 Data Presentation

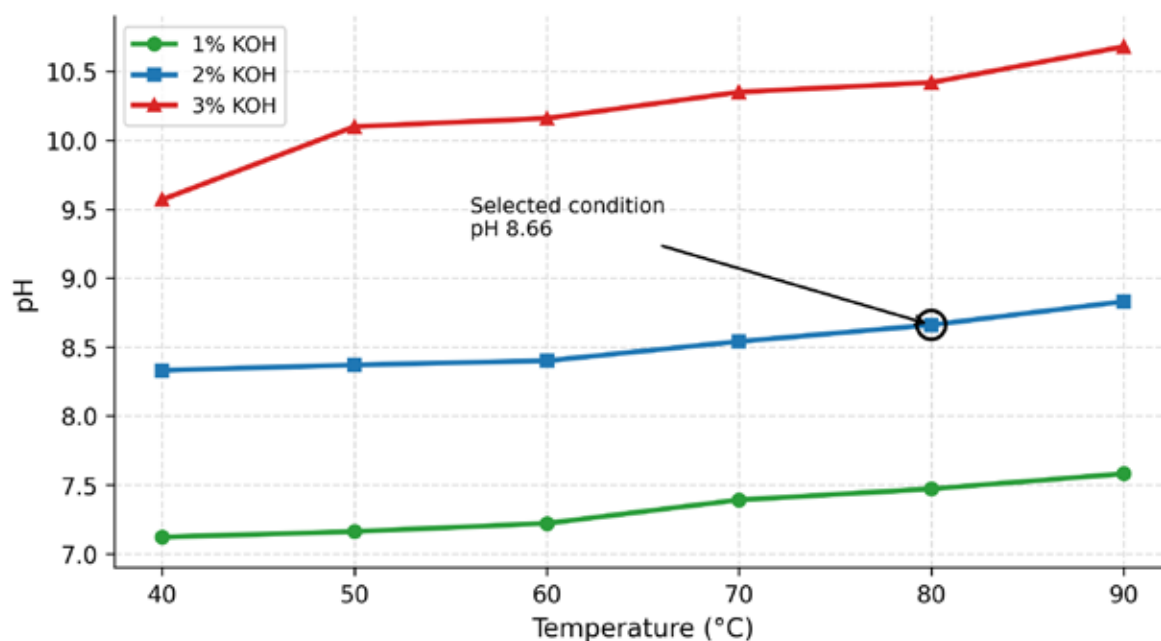
The source dataset reported measured values for each tested condition but did not include replicate counts, uncertainty estimates, or inferential statistics. Results are therefore presented descriptively as percentages, ranges, and absolute percentage-point differences. The English figures reproduce the numerical series shown in the source manuscript; no interpolation was used to generate new experimental observations.

3. Results And Discussion

3.1 Effect of Process Variables on pH

KOH concentration was the principal determinant of pH. Across the complete experimental matrix, pH ranged from 6.53 to 7.78 at 1% KOH, from 7.92 to 8.91 at 2% KOH, and from 9.35 to 10.92 at 3% KOH. Temperature and L/S ratio had smaller effects, generally increasing pH by approximately 0.1–0.3 units at successive levels. At L/S = 2.5:1, the temperature profiles remained clearly separated by KOH concentration (Fig. 1). The selected condition of 2% KOH and 80 °C gave pH 8.66, corresponding to moderate alkalinity rather than the strongly alkaline region reached at 3% KOH.

Figure 1. Effect of temperature and KOH concentration on the pH of the liquid phase at L/S = 2.5:1



3.2 Aluminum Mobilization

Aluminum transfer was highly sensitive to alkalinity. Across all conditions, liquid-phase transfer was 0.72–1.69% at 1% KOH and 1.37–3.72% at 2% KOH. At 3% KOH and pH 9.35–10.92, it increased to 4.94–14.20%. At fixed L/S = 2.5:1, aluminum transfer increased with temperature at every KOH concentration, but the separation between the 2 and 3% series was much larger than the temperature effect within either series (Fig. 2). This pattern is consistent with the amphoteric dissolution of aluminum hydroxide and the formation of soluble aluminate species in basic media (Gayer et al., 1958).

3.3 Iron Mobilization

Iron transfer remained comparatively low over the investigated matrix: 2.5–3.7% at 1% KOH, 3.9–5.5% at 2% KOH, and 6.4–9.7% at 3% KOH. At L/S = 2.5:1, increasing temperature from 40 to 90 °C at 2% KOH raised the liquid-phase fraction from 4.5 to 5.3% (Fig. 3). Iron was therefore retained predominantly in the solid under all tested conditions. The response is compatible with persistence of iron(III) (hydr)oxide phases under moderate alkalinity, although mineralogical analysis would be required to identify the phases actually present (Das et al., 2011).

Figure 2. *Effect of temperature and KOH concentration on aluminum transfer to the liquid phase at $L/S = 2.5:1$*

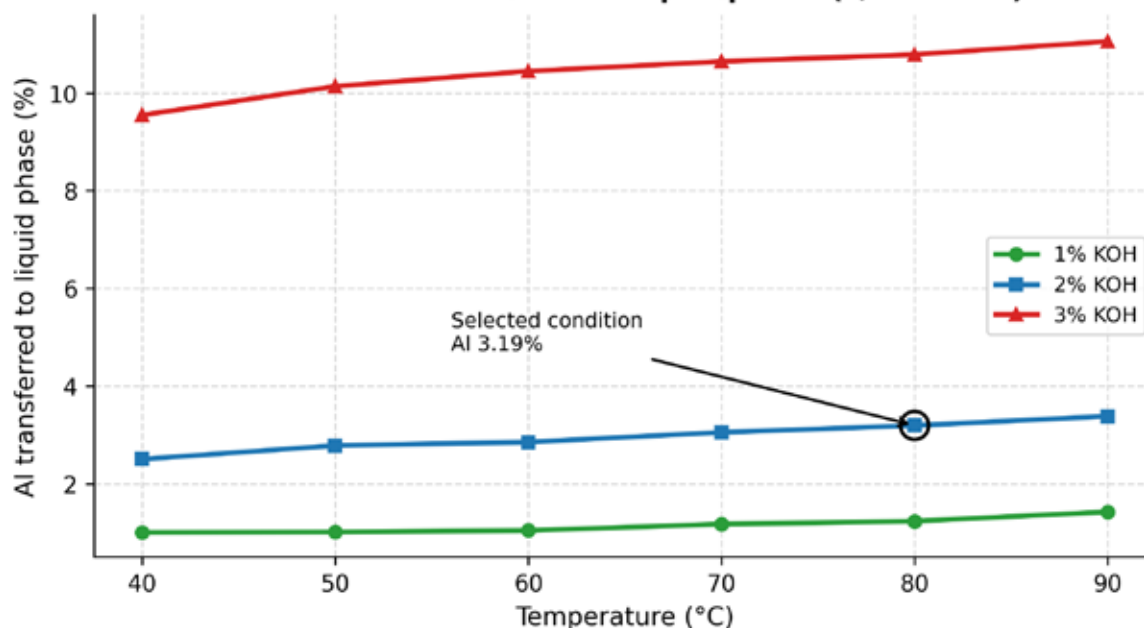
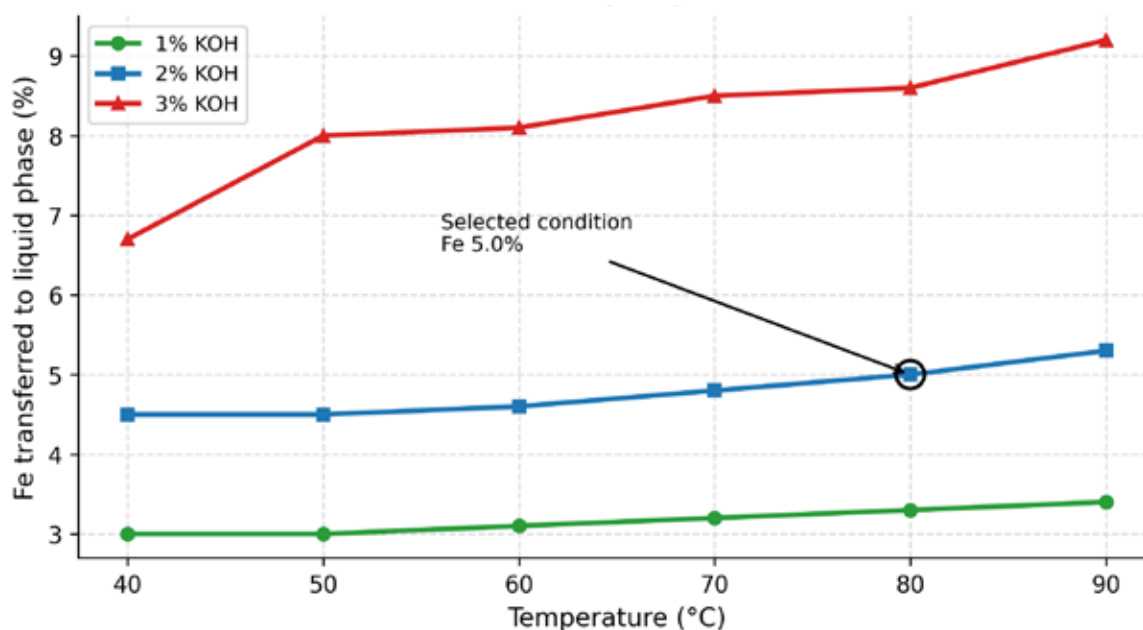


Figure 3. *Effect of temperature and KOH concentration on iron transfer to the liquid phase at $L/S = 2.5:1$*

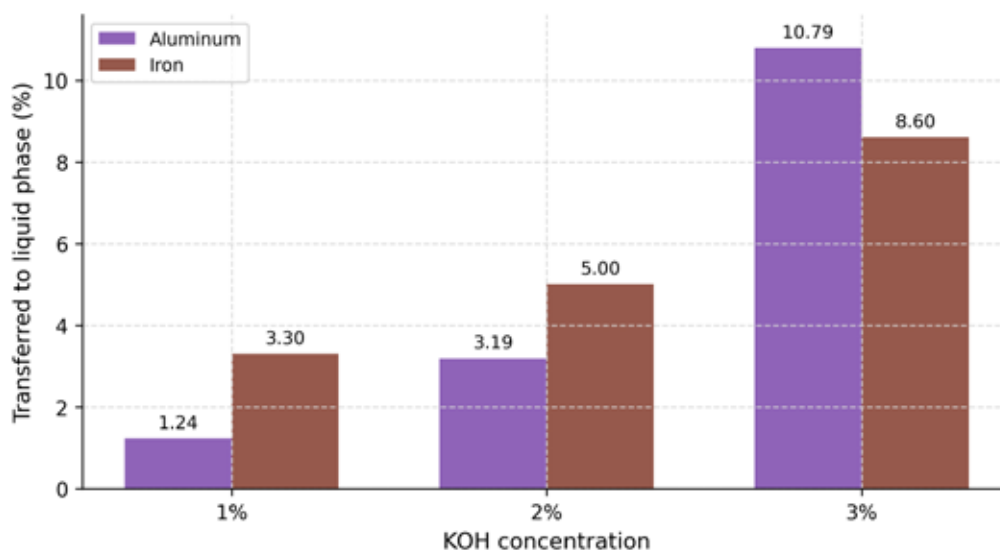


3.4 Relative Effect of KOH Concentration

At 80 °C and $L/S = 2.5:1$, increasing KOH from 1 to 2% raised aluminum transfer from 1.24 to 3.19% and iron transfer from 3.30 to 5.00%. A further increase to 3% KOH raised aluminum transfer to 10.79% and iron transfer to 8.60% (Fig. 4). Thus, the step from 2 to 3% KOH increased

aluminum mobilization by 7.60 percentage points and more than tripled its liquid-phase fraction. The corresponding change for iron was 3.60 percentage points. KOH concentration, acting through pH, was consequently the dominant control on phase partitioning; temperature had a secondary effect, and L/S ratio had the smallest effect within the ranges studied.

Figure 4. Effect of KOH concentration on aluminum and iron transfer at 80 °C and L/S = 2.5:1



3.5 Interphase Partitioning under Selected Conditions

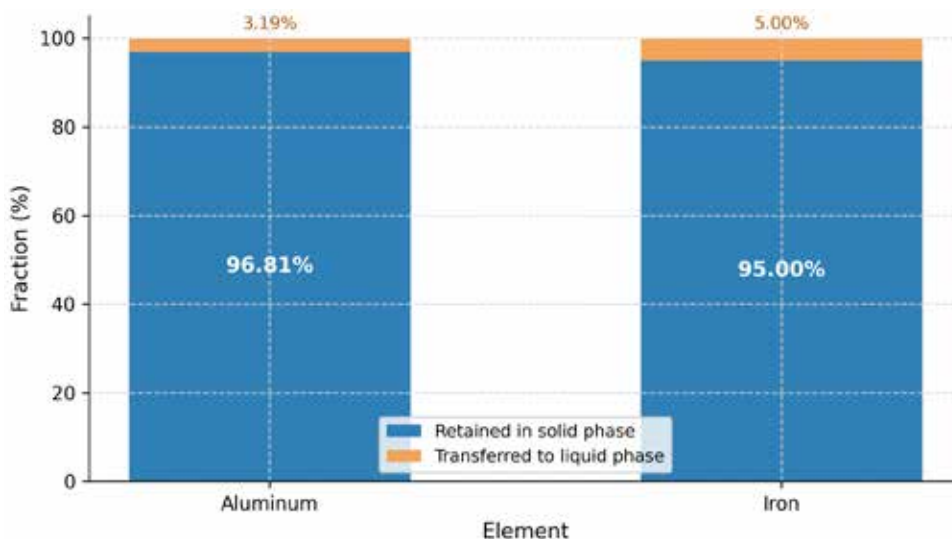
The resource-efficient condition selected for integrated sapropel processing was 2% KOH, L/S = 2.5:1, and 80 °C. At pH 8.66, only 3.19% of aluminum and 5.00% of iron

entered the liquid phase; correspondingly, 96.81% and 95.00% were retained in the solid (Table 1; Fig. 5). These values indicate preservation of the metal-bearing mineral fraction while avoiding the stronger aluminum dissolution observed at 3% KOH.

Table 1. Interphase partitioning of aluminum and iron at 2% KOH, L/S = 2.5:1, and 80 °C

Parameter	Aluminum (Al)	Iron (Fe)	Interpretation
Transferred to liquid phase (%)	3.19	5.00	Entered the fertilizer stream
Retained in solid phase (%)	96.81	95.00	Preserved in the residue
Liquid-phase pH	8.66	8.66	Moderately alkaline medium

Figure 5. Partitioning of aluminum and iron between solid and liquid phases under the selected conditions (2% KOH, L/S = 2.5:1, and 80 °C)



The retention of approximately 95–97% of both elements is favorable for further material development. Iron- and aluminum-bearing (hydr)oxides can provide hydroxylated surfaces for cation and oxyanion binding through surface complexation and ligand exchange (Hira et al., 2023; Li et al., 2016; Mashale et al., 2025). Residual humic fragments may contribute additional ion-exchange sites. However, elemental retention alone does not establish adsorption performance. Surface area, pore structure, mineralogy, point of zero charge, leaching stability, adsorption isotherms, kinetics, selectivity in multicomponent solutions, and regeneration must be measured before the material can be described as an effective sorbent.

The selected 2% KOH condition also limits aluminum entry into the liquid fertilizer precursor. This may be desirable because excessive soluble aluminum can be phytotoxic under some soil conditions. Compared with 2% KOH, 3% KOH provides only a small additional benefit for humic-substance recovery in the associated process dataset, whereas it increases aluminum loss more than threefold. The 2% condition therefore offers a defensible compromise among liquid-product recovery, retention of mineral surface functionality, and reagent demand.

3.6 Study Limitations

This screening study is limited by incomplete analytical metadata in the source record, absence of replicate statistics and error estimates, and lack of direct characterization of the residual solid. The reported partitioning values should therefore be interpreted as descriptive process-screening results. Independent replication with documented instruments, calibration, quality-control samples, and full mass-balance closure is required. Direct adsorption tests are also necessary to verify the proposed downstream use.

4. Conclusion

KOH concentration was the dominant control on pH and on the partitioning of aluminum and iron during alkaline processing of Aral Sea sapropel. Aluminum transfer rose sharply above pH 9 at 3% KOH, reaching 14.20% across the full experimental matrix, whereas it remained at or below 3.72% at 1–2% KOH. Iron was retained more strongly,

with 2.5–9.7% transferred to the liquid phase across all conditions.

At 2% KOH, L/S = 2.5:1, and 80 °C, the liquid phase had pH 8.66 and contained 3.19% of the initial aluminum and 5.00% of the initial iron. Thus, 96.81% of aluminum and 95.00% of iron remained in the solid residue. These conditions preserve most metal-bearing mineral components while avoiding the substantial aluminum mobilization caused by 3% KOH. The residue is a promising sorbent precursor, but its adsorption capacity and operational performance require direct experimental confirmation.

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Competing Interests

The author has declared that no competing interests exist.

Authors' Contributions

Shakhnoza Shukhrat qizi Rashitova conceived the study, conducted the experimental work, analyzed and interpreted the data, prepared the original manuscript and figures, and read and approved the final manuscript.

Consent

Not applicable. This study did not involve human participants or identifiable personal information.

Ethical Approval

Not applicable. The study involved laboratory processing of a natural mineral-organic material and did not involve humans or animals.

Data Availability

All data evaluated in this study are presented in the article. The author should retain the original laboratory records for editorial or peer-review verification.

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