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Investigation of Date Seed Ash as a pH and Lost Circulation Material (LCM) Bio-enhancer in Water-Based Drilling Fluids

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Abstract**:** *Caustic soda is widely used to keep water-based drilling mud alkaline, but it is hazardous to handle and harmful to the environment when discarded. This study investigated date seed ash (DSA), an agricultural waste, as a bio-enhancer for pH control and fluid loss reduction in water-based mud. Date seeds were dried, calcined at 700 °C, sieved to below 75 µm and extracted in water to give an alkaline solution. X-ray fluorescence showed that the ash was dominated by potassium (45.29 wt%). Four 350 mL muds were prepared following API RP 13B-1: a control containing 0.5 g NaOH, and three DSA muds containing 350, 300 and 250 mL of extract (DSA-1 to DSA-3), the last with 0.5 g NaOH added. pH, 30-minute static filtrate volume and mud cake thickness were measured. DSA-1 and DSA-2, which contained no NaOH, reached pH 10.68 and 10.08, compared with 9.00 for the control. DSA-1 reduced filtrate volume by 21.7% (9.0 against 11.5 mL), whereas DSA-2 increased it to 13.0 mL. All DSA muds formed thinner cakes (1.59 to 1.98 mm) than the control (2.38 mm). Date seed ash can replace caustic soda for pH control and can improve filtration, provided the extract proportion is optimised.*

Keywords: date seed ash; pH enhancer; fluid loss control; water-based drilling mud; bio-enhancer

1. Introduction

Drilling operations represent a critical, capital-intensive phase in upstream petroleum development, the operational success of which depends heavily on drilling fluid performance [1]. In petroleum engineering practice, drilling fluid denotes the circulating media (liquids, gases, or multi-phase systems) formulated to clean the hole, cool the bit, transport cuttings, and sustain hydrostatic wellbore integrity [1], [2]. The physiochemical and transport properties of drilling muds are inherently dynamic: they fluctuate continuously as functions of downhole pressure, circulating temperature, drilling rate of penetration (ROP), formation lithology, and reactive contaminant ingress [1]. Continuous interactions among mud constituents yield variable changes in rheological parameters, solid fraction dynamics, wall-building properties, and hydrogen ion concentration (pH) [1], [2]. Precise control of these interrelated properties is mandatory to avert catastrophic drilling anomalies, including drill string sticking, wellbore collapse, formation damage, and blowout hazards [2], [3].

Among baseline drilling mud properties, pH is an essential thermodynamic control parameter [2]. An alkaline environment between pH 8.5 and 10.5 promotes full electrokinetic hydration and dispersion of sodium bentonite platelets, neutralizing edge-to-face attractive forces to establish optimum thixotropy, yield point, and fluid loss control [4]-[6]. In contrast, exposure to acidic gases (CO_2, H_2S) or formation brine influx sharply drives mud pH below neutrality, causing severe equipment corrosion, polymer degradation, and rapid clay flocculation [7], [8]. Operating within the alkaline bracket provides four core field advantages:

1. Mitigates electrochemical corrosion fatigue across the drill string, casing, and surface manifolds [7], [8].
2. Sustains montmorillonite delamination, guaranteeing rheological stability and low filter-cake permeability [4], [5].
3. Neutralizes corrosive acid-gas contaminants while accelerating the hydration efficiency of functional polymers such as carboxymethyl cellulose (CMC) and partially hydrolyzed polyacrylamide (PHPA) [2], [6].
4. Minimizes wellbore instability across micro-fractured, water-sensitive reactive shales [9].

Conventionally, target alkalinity is maintained using industrial inorganic chemicals, including sodium hydroxide (NaOH), potassium hydroxide (KOH), soda ash (Na_2CO_3), and hydrated lime ($Ca(OH)_2$) [6], [8]. Despite their field performance, these chemical alkalizers present severe occupational and environmental hazards. Caustic soda and caustic potash are classified as Category 1B skin corrosives, posing severe chemical burn risks to rig personnel during manual hopper dosing [9]. Furthermore, offshore discharge or terrestrial spillage of caustic-laden muds and cuttings induces sharp, localized pH spikes that severely degrade benthic ecosystems and agricultural soil microbiota [9], [10].

Beyond alkalinity maintenance, drilling efficiency requires rigorous mitigation of downhole fluid escape. Lost circulation—the uncontrolled migration of drilling fluid into fractured, vugular, or high-permeability thief formations—remains a major cause of non-

productive rig time (NPT) [11], [12]. Managing these losses requires bridging and sealing agents known as lost circulation materials (LCMs) [11]. In standard permeable intervals, fine particulates also function as fluid-loss control additives, reinforcing the bentonite filter cake [12], [13]. Growing economic and environmental restrictions have prompted active research into non-toxic, biodegradable, agricultural-waste-derived LCM and filtration additives. Waste residues such as orange peels, sunflower husks, plantain peels, and palm kernel shells have proven capable of bridging pore throats, optimizing cake packing, and curtailing filtration volumes [11], [14], [15]. Date palm residues (*Phoenix dactylifera*), in particular, provide a renewable, regionally abundant source of cellulosic and mineral biomass capable of serving as functional drilling mud additives [10], [13].

Global date production exceeds 9.8 million metric tons annually, with date seeds comprising 10–15% of the total fruit weight [16], [17]. In major producing hubs across the Middle East and semi-arid agro-ecological belts in Northern Nigeria, millions of metric tons of seed waste are routinely discarded or openly incinerated [16]–[18]. Rather than remaining an environmental liability, date seeds represent an underutilized bioresource rich in carbonaceous matrices and inorganic metallic precursors [19]. Pulverized date seeds have been investigated as fluid-loss control agents in clay-free fluids [13], [20] and as composite weighting/rheology modifiers when blended with palm kernel shells [15]. Concurrently, calcining date seed biomass produces Date Seed Ash (DSA), an inorganic residue with notable alkaline properties [9].

Thermal calcination of date seeds volatilizes the organic lignocellulosic framework, converting endogenous alkali and alkaline-earth carboxylates into basic inorganic oxides (K_2O, CaO, MgO, Na_2O) [9]. When introduced into an aqueous mud continuous phase, these basic oxides rapidly hydrolyze, releasing free hydroxyl ions (OH^-) that elevate fluid pH [9]:

$$K_2O_{(s)} + H_2O_{(l)} \rightleftharpoons 2K^+_{(aq)} + 2OH^-_{(aq)}$$

$$CaO_{(s)} + H_2O_{(l)} \rightleftharpoons Ca^{2+}_{(aq)} + 2OH^-_{(aq)}$$

The elemental composition of the date seed ash employed in this study, determined via X-ray fluorescence (XRF) spectrometry, is summarized in Table 1 [9]. Potassium constitutes the dominant metallic element (45.29 wt%), followed by functional fractions of oxygen (11.07 wt%), magnesium (3.80 wt%), and calcium (3.28 wt%) [9]. This profile provides a strong stoichiometric basis for alkalinity generation, mirroring the role of commercial potassium and sodium carbonate salts [9]. Simultaneously, the potassium ions (K^+) liberated in solution offer an intrinsic clay-stabilization benefit, intercalating within reactive montmorillonite layers to restrict hydration swelling [9]. In parallel, the insoluble micro-particulate mineral fraction of DSA ($< 75\ \mu$m) functions as a micro-LCM and bridging agent, packing interstitial voids between bentonite platelets to reduce mud cake permeability [9], [12].

Table 1. Elemental composition of date seed ash determined by XRF analysis (Gbenga et al., 2026).

Element number	Element name	Weight conc. (wt%)
19	Potassium	45.29
8	Oxygen	11.07
15	Phosphorus	7.92
40	Zirconium	7.64
7	Nitrogen	6.96
50	Tin	5.86
12	Magnesium	3.80
6	Carbon	3.55
16	Sulfur	3.39
20	Calcium	3.28
14	Silicon	0.61
13	Aluminium	0.33
17	Chlorine	0.31
Other	I, Zn, Ag, Cu, Fe, Na, Ba and Mn	0.00

While date seed powder has been studied independently as a fluid loss additive and date seed ash as a basic pH modifier, few empirical studies have systematically evaluated date seed ash as a dual-function additive acting simultaneously as a bio-alkalizer and a micro-bridging/loss prevention agent in water-based drilling muds. This investigation evaluates date seed ash as a sustainable, low-cost bio-enhancer for simultaneous pH elevation and fluid loss control in water-based drilling fluids, establishing an eco-friendly pathway to eliminate caustic chemicals while driving agricultural circular-economy practices

2. Materials and Methods

2.1 Materials

The experimental water-based drilling mud (WBM) systems were formulated using commercial-grade bentonite (sodium montmorillonite) complying with API Specification 13A standards as the primary viscosifier. Drilling-grade barite ($BaSO_4$) was utilized as a weighting agent to adjust fluid density. Low-viscosity technical-grade Sodium Carboxymethyl Cellulose (CMC-LV) was used as the filtration control agent. Analytical-grade sodium hydroxide (NaOH, 98% purity, pellets) served as the benchmark chemical alkalizer.

Waste date palm seeds (*Phoenix dactylifera*) were collected from agricultural fruit distribution markets in Owerri, Imo State, Nigeria. Deionized and laboratory-treated freshwater free of polyvalent mineral contaminants was employed as the base liquid continuous phase across all formulations.

2.2 Methods

Preparation and Alkaline Extraction of Date Seed Ash (DSA)

The collected date seeds were manually separated from residual pulp, washed thoroughly with deionized water to eliminate surface impurities and residual fermentable sugars, and oven-dried at 105°C for 24 hours to constant mass. The dried seeds were transferred to a heat-resistant ceramic crucible and calcined in a high-temperature electric muffle furnace (Drawell T-Series). Thermal ramp-up was maintained under aerobic atmospheric conditions up to 700°C with a soaking duration of 3 hours to ensure complete oxidative combustion of organic fractions and conversion of inherent alkali earth minerals into basic oxides.

After furnace cooling within an airtight desiccator to ambient temperature, the calcined ash was ground using a mechanical mortar and pestle, followed by sieving through a standard 75 μm (API 200 mesh) sieve to yield fine Date Seed Ash (DSA) powder. A stock date ash solution was prepared by dispersing 500 g of sieved DSA into 1000 mL of deionized water (1: 2 w/v ratio). The mixture was homogenized on a Jenway 1100 magnetic hotplate stirrer and heated until boiling (100°C) to accelerate the dissolution kinetics of alkali metal oxides (K_2O, Na_2O, CaO) into active hydroxide complexes (K^+, Na^+, OH^-). The cooled slurry was filtered through Whatman No. 42 ashless filter paper to obtain a clarified, highly alkaline liquid extract (DSA stock solution).



Figure 1a: Dried Date Seeds



Figure 1b: Date Seed Ash (DSA) after burning



Figure 1c: Pulverized Date Seed Ash



Figure 1d: DSA solution Prepared

Formulation and Blending of Drilling Fluid Systems

Four drilling fluid formulations were prepared using a standardized laboratory Hamilton Beach multi-speed mixer in compliance with API RP 13B-1 protocols. Each system was formulated to an equivalent laboratory barrel volume of 350 mL:

1. **Control System (Conventional WBM):** 350 mL deionized water, 22.5 g bentonite, 10.0 g barite, 0.5 g CMC, and 0.5 g NaOH.
2. **DSA-2 (Full Bio-Extract):** 350 mL DSA stock solution, 22.5 g bentonite, 10.0 g barite, and 0.5 g CMC (zero synthetic NaOH).
3. **DSA-2 (Hybrid Blend 1):** 300 mL DSA stock solution, 50 mL deionized water, 22.5 g bentonite, 10.0 g barite, and 0.5 g CMC.
4. **DSA-3 (Hybrid Blend 2):** 250 mL DSA stock solution, 100 mL deionized water, 22.5 g bentonite, 10.0 g barite, 0.5 g CMC, and 0.5 g supplementary NaOH.

The order of addition followed API standards: the liquid continuous phase was placed in the stainless steel mixing cup, bentonite was added slowly over 2 minutes and sheared for 10 minutes, followed by the addition of CMC, barite, and chemical alkalizers, with final homogenization at high shear (11,500 rpm) for 20 minutes. Formulated muds were statically aged for 16 hours at ambient conditions prior to property characterization.

Table 2: Compositional Matrix of Formulated Water-Based Drilling Mud Systems (per 350 mL Lab Barrel)

Component	Control	DSA-1	DSA-2	DSA-3
Deionized Water (mL)	350	0	50	100
DSA Stock Extract (mL)	0	350	300	250
Bentonite (g)	22.5	22.5	22.5	22.5
Barite (g)	10.0	10.0	10.0	10.0
CMC (g)	0.5	0.5	0.5	0.5
NaOH (g)	0.5	0	0	0.5

pH Measurement:

The dynamic hydrogen ion activity (pH) of each formulation was quantified using a calibrated Mettler Toledo digital pH probe alongside broad-spectrum colorimetric API pH indicator strips for visual verification. Calibration was performed prior to each run using certified buffer solutions

Static Low-Pressure Low-Temperature (LPLT) Filtration:

Static filtration characteristics were evaluated using an API standard filter press (Fann Model 300 series) operating at an applied differential pressure of 100 psi (690 kPa) and at ambient temperature across Whatman No. 50 hardened filter paper with an effective filtration area of 45.8 cm^2. Filtrate discharge volumes were recorded incrementally at 5, 10, 15, 20, 25, and 30 minutes.

Filter Cake Thickness:

At the completion of the 30-minute filtration test, the filter cell was depressurized, residual mud was decanted, and the deposited mud cake was gently washed with a low-velocity stream of distilled water. Cake thickness was measured at three distinct locations using a precision digital micrometer/vernier caliper and converted to standard units (mm and fractional 1/32 inch).

3. Results and Discussions

3.1 pH Enhancement

Table 3 compares the pH of the four muds. The Control mud, treated with 0.5 g of NaOH, had a pH of 9.00. DSA-1 and DSA-2 contained no NaOH at all, yet reached pH 10.68 and 10.08, which is 1.68 and 1.08 units higher than the Control. DSA-3 contained the same 0.5 g of NaOH as the Control and reached 9.41, so the date seed ash (DSA) added a further 0.41 pH units. These results show that DSA is an effective plant-based source of alkalinity and can replace caustic soda in water-based mud.

Table 3: Comparative pH Values of Formulated Drilling Fluids

Mud Formulation	NaOH Added (g)	pH Value
Control	0.5	9.00
DSA-1	0.0	10.68
DSA-2	0.0	10.08
DSA-3	0.5	9.41

The reason is found in the XRF results (Table 1). Potassium is the main element in the ash (45.29 wt%), followed by smaller amounts of magnesium and calcium. When date seeds are burned (calcined), these elements are largely left as alkaline compounds, which dissolve in water and release hydroxide ions (OH^-) into the mud. XRF reports elements rather than compounds, so the exact alkaline compounds in the ash were not identified in this study.

The pH rose as more DSA extract was used in the mud. Recommended pH for water-based mud is about 8.5 to 10.5. DSA-2 (10.08) and DSA-3 (9.41) fall inside this range, as does the Control (9.00), while DSA-1 (10.68) is slightly above the upper limit. Staying in this range matters because it reduces corrosion of drill pipe and casing, limits the effect of acid gases such as H_2S and CO_2, and keeps bentonite well dispersed [6], [4], [7]. If a pH below 10.5 is needed, a slightly lower ash content could be used. Other plant-based additives, such as burnt palm head ash and plantain peel powder, are also known to raise mud pH, but by less than NaOH [5]. In this study, DSA raised the pH higher than the NaOH control.

3.2 Static Filtration Loss

Table 4 shows the cumulative volume of filtrate collected over the standard 30-minute test, and Figure 2 plots the same data against the square root of time. A lower filtrate volume means better fluid loss control.

Table 4: Cumulative Static LPLT Filtrate Volume (mL) vs. Elapsed Time (Minutes)

Time	Control	DSA-1	DSA-2	DSA-3
5 min	5.5	4.0	6.0	4.5
10 min	6.5	5.0	8.5	6.5
15 min	8.5	5.5	10.0	8.0
20 min	9.5	7.5	10.5	9.0
25 min	10.5	8.5	12.0	10.0
30 min	11.5	9.0	13.0	11.0

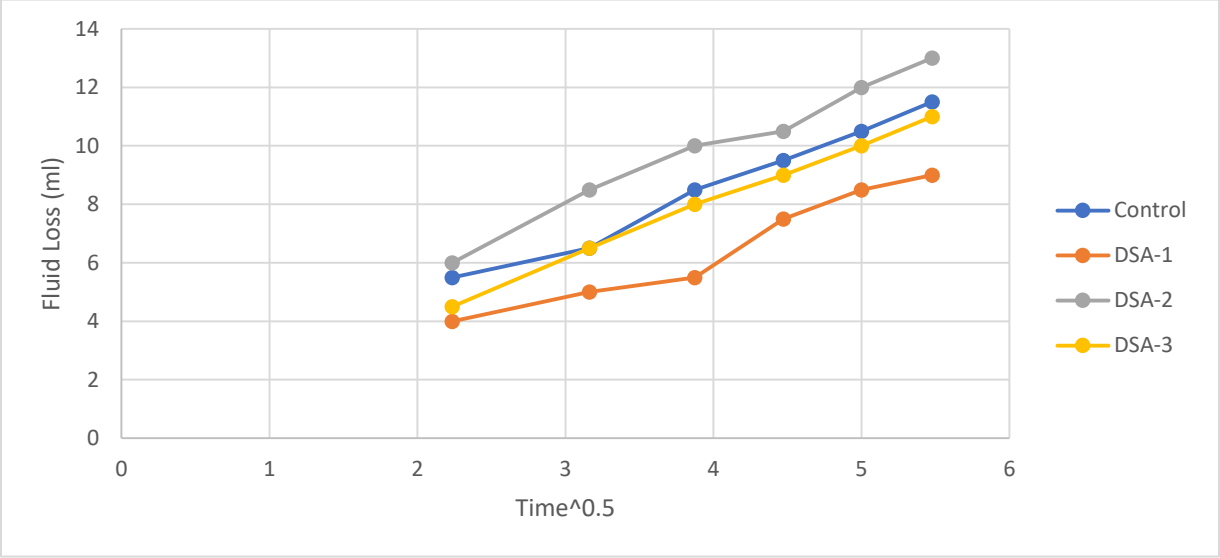


Figure 2: Graph of Cumulative Filtrate Volume vs. Square Root of Time (t^0.5)

After 30 minutes, the filtrate volumes were 9.0 mL for DSA-1, 11.0 mL for DSA-3, 11.5 mL for the Control and 13.0 mL for DSA-2. DSA-1 therefore gave the best fluid loss control, with a 21.7% reduction compared with the Control, and it had the lowest filtrate volume at every time interval. DSA-3 was slightly better than the Control (a 4.3% reduction).

Two factors probably explain the good result of DSA-1. First, its higher pH (10.68) helps the bentonite platelets hydrate and spread out evenly, so they pack into a thin, tight filter cake [4]. Second, at higher pH the CMC polymer in the mud uncoils more fully because its negatively charged carboxylate groups ($-COO^-$) repel each other, which improves its ability to block pores.

Most of the difference between the muds was created early. In the first 5 minutes the Control lost 5.5 mL, compared with 4.0 mL for DSA-1 and 4.5 mL for DSA-3. After 10 minutes, all four muds lost filtrate at a similar rate (4.0 to 5.0 mL between 10 and 30 minutes). This suggests that the muds containing ash sealed the formation face faster.

DSA-2 gave the highest fluid loss (13.0 mL, which is 13.0% above the Control), even though its pH (10.08) was higher than that of the Control. This shows that pH alone does not control filtration. Because DSA-1 and DSA-2 both had no NaOH, the difference is more likely linked to their composition, such as the amount of ash extract and the dissolved salts it carries. The ash is rich in potassium (Table 1), and a higher concentration of K^+ ions can compress the electrical layer around the clay particles. This may cause the particles to clump together and form a more permeable cake. This explanation is a suggestion and should be confirmed with the exact mud compositions and an ionic analysis.

3.3 Mud Cake Thickness and Quality

A good drilling fluid forms a thin, smooth and low-permeability filter cake on permeable rock. This helps to prevent differential pipe sticking and limits damage to the formation. Table 5 compares the cakes formed by the four muds

Table 5: Comparative Mud Cake Thickness Across Formulations

Formulation	Thickness (in)	Thickness (mm)	Integrity
Control	3.0/32 inch	2.38	Thick, fibrous
DSA-1	2.5/32 inch	1.98	Smooth, slick, low permeability
DSA-2	2.5/32 inch	1.98	Smooth, slick, low permeability
DSA-3	2.0/32 inch	1.59	Thin, compact

All DSA muds formed thinner cakes (1.59 to 1.98 mm) than the Control (2.38 mm). This is about 17% thinner for DSA-1 and DSA-2 and 33% thinner for DSA-3. The Control cake was thick and fibrous, DSA-1 and DSA-2 cakes were smooth and slick, and the DSA-3 cake was thin and compact. The fine ash particles most likely fill the tiny gaps between overlapping bentonite platelets, giving a tightly packed cake that lowers the risk of stuck pipe. Filling and sealing pores in this way is also how lost circulation materials work [11].

Cake thickness and filtrate volume did not always agree. DSA-3 had the thinnest cake but only a small reduction in filtrate, and DSA-2 had the same cake thickness as DSA-1 but lost more filtrate. Cake thickness should therefore be read together with filtrate volume and cake quality, not on its own.

4. Conclusions and Recommendations

4.1 Conclusions

This study investigated the viability of date seed ash (DSA) extract as a sustainable, bio-based alternative to conventional caustic soda (NaOH) in water-based drilling muds. The experimental outcomes demonstrate that DSA functions as a potent alkaline donor, raising the mud pH to 10.68 in DSA-1 and 10.08 in DSA-2 without the addition of synthetic NaOH, in contrast to the baseline reading of 9.00 recorded for the commercial NaOH control. This alkalinity elevation correlates directly with the volumetric concentration of DSA extract in the formulation and aligns with the potassium-rich elemental composition (45.29 wt% K) identified via XRF analysis. The hybrid formulation, DSA-3 (250 mL extract supplemented with 0.5 g NaOH), yielded an intermediate pH of 9.41, falling well within the standard operational window of 8.5 to 10.5, whereas DSA-1 slightly exceeded this boundary.

Beyond alkalinity modification, the incorporation of DSA exerted a measurable influence on fluid loss control and wall-building properties. The filtration response varied across formulations: DSA-1 exhibited the highest filtration resistance, delivering a cumulative filtrate volume of 9.0 mL representing a 21.7% reduction relative to the control system (11.5 mL)—while DSA-3 achieved a modest 4.3% reduction, and DSA-2 exhibited an elevated filtrate volume of 13.0 mL (13.0% above the control). This non-linear behavior confirms that fluid loss performance is governed by complex electrokinetic and ionic interactions within the bentonite-polymer matrix rather than pH alone. Furthermore, all DSA-treated formulations generated thinner filter cakes (1.59 to 1.98 mm) than the conventional control (2.38 mm), producing smooth, slick, and compact cake textures that lower the risk of differential pipe sticking. Overall, formulation DSA-1 showed the most promising performance by maximizing pH elevation and filtration control without caustic soda additions, validating date seed ash as an effective, low-cost bio-enhancer that valorizes agro-waste. However, these baseline conclusions remain constrained to ambient-temperature evaluations of pH, static API filtration, and cake morphology, excluding full rheological profiling, elevated thermal regimes, and direct lost circulation bridging assessments.

4.2 Recommendations

To build upon these baseline findings, subsequent investigations should focus on several critical technical pathways. First, volumetric extract concentrations should be optimized within the range of 300 to 350 mL per lab barrel to establish an equilibrium where mud pH is strictly buffered at or below 10.5 while preserving minimal fluid loss; rigorous statistical replicates should also be performed to evaluate the variance and underlying cause of the elevated filtrate volume observed in DSA-2. Second, comprehensive rheological evaluations must be conducted using rotational viscometry to quantify plastic viscosity, yield point, and gel strengths, as a viable pH enhancer must not adversely alter non-Newtonian flow behavior or cuttings transport efficiency. Third, formulations should be subjected to dynamic high-pressure high-temperature (HPHT) filtration and static thermal rolling to verify whether the alkalinity buffering capacity and bentonite-CMC stability persist under downhole geothermal conditions.

Additionally, because the aqueous extraction method used in this study filtered out solid matter, the bridging potential of raw, insoluble date seed ash should be evaluated independently. Direct particulate bridging assessments using a Particle Plugging Apparatus (PPA) or slotted-disc permeability cells across graded sieve fractions are recommended to rigorously validate date seed ash as a dual-action lost circulation material (LCM). Detailed mineralogical and ionic characterization via X-ray diffraction (XRD), scanning electron microscopy (SEM), and ion chromatography is also necessary to map the crystalline phases, particle morphology, and specific concentrations of free K^+, Ca^{2+}, and carbonate ions, which will clarify clay swelling inhibition and ionic flocculation mechanisms. Furthermore, the elevated concentrations of zirconium (7.64 wt%) and tin (5.86 wt%) reported in the preliminary XRF analysis require re-examination via duplicate analytical runs to rule out mechanical milling or crucible contamination and to ensure complete regulatory compliance regarding environmental toxicity. Finally, technical-economic analyses and benchmark comparisons against other industry alkalizers, such as soda ash, KOH, and slaked lime, should be conducted alongside ecotoxicological screenings of spent mud and cuttings to confirm field viability and commercial scalability.

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