CRYSTALLISATION PATHWAYS, PROCESS OPTIMISATION AND PRODUCT QUALITY OF INDIGENOUS POTASH/TRONA FROM YOBE STATE: PROSPECTS FOR OILFIELD APPLICATIONS

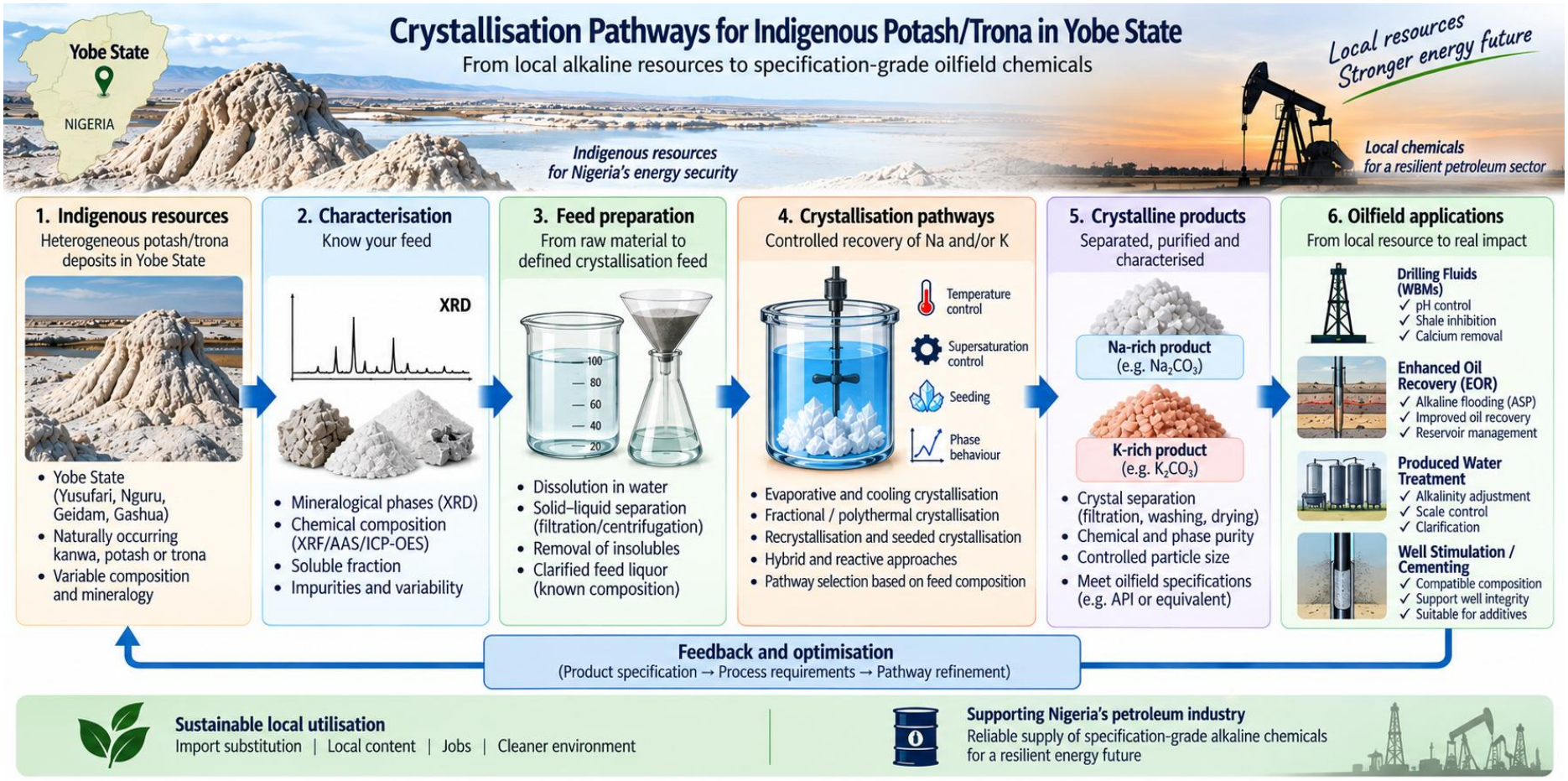
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Abstract

Yobe State in northeastern Nigeria contains indigenous potash and trona resources with potential as locally sourced alkaline mineral feedstocks for petroleum applications, including drilling fluids, enhanced oil recovery (EOR) and produced-water treatment. However, materials locally described as kanwa, potash or trona are chemically and mineralogically heterogeneous; therefore, feed-specific characterisation is essential before process design. This review examines crystallisation pathways for Yobe potash/trona using a composition-driven resource–process–product framework. It discusses the conversion of heterogeneous raw materials into defined crystallisation liquors through dissolution, clarification and targeted pretreatment, and evaluates evaporative, cooling, fractional/polythermal, recrystallisation, seeded and hybrid crystallisation routes. Particular attention is given to mixed Na–K feedstocks, for which fractional crystallisation may enable sequential recovery of Na- and K-rich products, although Yobe-specific solid–liquid equilibrium and kinetic data remain limited. Product quality is assessed in terms of composition, phase purity, physical characteristics, stability, impurity control and application-specific requirements. The review links processing intensity to product specification and oilfield suitability, emphasising the need for rigorous qualification against petroleum-industry requirements. Priority areas for future work include Yobe-specific phase-equilibrium and kinetic studies, selective Na/K recovery, product validation, pilot-scale development and techno-economic assessment.

Keywords: Yobe State; potash; trona; crystallisation pathways; fractional crystallisation; Na/K separation; product quality; oilfield applications

Graphical Abstract



Introduction

The petroleum industry relies on a continuous supply of specification-grade alkaline chemicals. Materials such as sodium carbonate (soda ash) and potassium carbonate are critical components in upstream operations, serving as essential pH buffers and calcium-precipitating agents in water-based drilling fluids, as well as primary alkaline agents in alkaline-surfactant-polymer (ASP) enhanced oil recovery (EOR)[1]. In Nigeria, dependence on imported refined alkaline chemicals creates an important supply-chain consideration and strengthens the case for developing domestic alternatives from locally available mineral resources[2].

In northern Nigeria, naturally occurring alkaline materials are commonly referred to as *kanwa*, potash and trona. However, these terms do not necessarily represent chemically or mineralogically uniform materials, as composition may vary with geological origin and locality. Their potential for value addition therefore depends on their verified mineralogical identity, chemical composition and suitability for controlled processing[3].

Yobe State, located in northeastern Nigeria within the greater Lake Chad Basin, contains indigenous potash/trona resources with reported traditional and practical uses. Studies of materials from Yusufari Local Government Area have shown considerable variation in K, Na, Ca and Mg contents between sampling locations[4]. Such variability is important for processing because materials with the same indigenous designation may exhibit different dissolution characteristics and crystallisation behaviour. In particular, elemental K or Na concentrations alone are insufficient to establish the specific mineral phases present; mineralogical and chemical characterisation should therefore precede process selection[5].

Crystallisation provides a practical route for concentrating, separating and upgrading dissolved alkaline salts through controlled changes in composition, temperature and supersaturation[6].

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[3] Garba NN, Ibrahim HJ, Onoja RA, Bello S, Nasiru R. Radiological safety assessment of mined Trona commonly consumed in Nigeria. Int J Environ Anal Chem. 2022;102(19):8434–44. doi:10.1080/03067319.2020.1849658

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[5] Hupp BN, Donovan JJ. Quantitative mineralogy for facies definition in the Marcellus Shale (Appalachian Basin, USA) using XRD-XRF integration. Sediment Geol. 2018;371:16–31. doi:10.1016/J.SEDGEO.2018.04.007

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This is especially relevant to mixed Na–K systems, where crystallisation of one component changes the composition of the remaining liquor and may influence subsequent phase formation. Studies of model carbonate systems have demonstrated composition- and temperature-dependent crystallisation behaviour, while multicomponent salt systems show that operating conditions and seeding can influence crystal composition and characteristics[7]. However, these studies provide transferable process knowledge rather than direct phase-equilibrium data for Yobe materials.

Despite evidence of indigenous alkaline resources in Yobe, limited information is available linking resource composition and mineralogy with feed-liquor chemistry, crystallisation behaviour, selective Na/K recovery and final product quality. This limits the development of reproducible value-added products. Therefore, this review focuses on a composition-driven approach in which feedstock characterisation guides crystallisation-pathway selection, process control and product quality assessment, with emphasis on producing alkaline chemicals capable of meeting relevant petroleum-sector requirements.

Indigenous Potash and Trona Resources of Yobe State

Occurrence and compositional variability

Yobe State lies within the northeastern Nigerian sector of the Lake Chad Basin, where naturally occurring evaporitic alkaline materials, including trona and potash-bearing deposits, have been reported. These resources are locally known as *kanwa*, potash or trona and have traditionally been used for domestic, livestock and other applications [11].

Available analytical studies indicate that these materials are compositionally heterogeneous. Sudi et al. (2016), for example, investigated potash deposits from Madukuri, Kotufa and Kirba in Yusufari Local Government Area and reported K as the dominant measured element, followed by Na, Ca and Mg (Table 1). Potassium concentrations varied substantially among the samples, from approximately 855 to 25,436 mg kg^{-1}, while Na concentrations ranged from about 3,535 to 5,535 mg kg^{-1}. Such variation demonstrates that materials collected from different locations cannot be assumed to have identical chemical characteristics[8].

Processing studies on Nigerian trona also indicate variability in the composition and physical form of indigenous materials[9], for example, evaluated trona samples from Nguru, Geidam and

Luo X, Li X, Wei C, Deng Z, Liu Y, Li M, et al. Recovery of NaCl and Na2SO4 from high salinity brine by purification and evaporation. Desalination. 2022;530:115631. doi:10.1016/j.desal.2022.115631

[7] Barakan S, Ayaluey MN, Shayanfar S, Aghazadeh V. Production and characterisation of sodium and potassium carbonate salts from carbonation alkaline aluminate liquor. Mineral Processing and Extractive Metallurgy. 2022;131(3):211–9. doi:10.1080/25726641.2021.1935163; Sergeev D, Yazhenskikh E, Haseli P, Liu M, Ziegner M, Bruno F, et al. Experimental study of thermodynamic properties and phase equilibria in Na_2CO_3–K_2CO_3 system. Calphad. 2020;71:101992. doi:10.1016/j.calphad.2020.101992

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Gashua in Yobe State and reported differences among cake, ash and flake forms, with corresponding differences in sodium carbonate recovery[10]. These observations reinforce the need to treat Yobe potash/trona as a heterogeneous resource rather than a single uniform feedstock.

For crystallisation-based beneficiation, this variability is particularly important because differences in soluble-ion composition can alter saturation conditions, phase formation and impurity incorporation[11]. Consequently, process conditions established for one deposit or batch should not automatically be transferred to another without feed-specific characterisation.

Indigenous nomenclature and mineralogical identity

A major difficulty in assessing Yobe potash/trona resources is the difference between indigenous nomenclature and mineralogical classification. In conventional usage, trona refers specifically to hydrated sodium sesquicarbonate, $Na_3H(CO_3)_2 \cdot 2H_2O$[12], whereas potash is a broader term for potassium-containing salts or products[13]. In northern Nigeria, however, terms such as *kanwa*, potash and trona may be applied to naturally occurring alkaline materials based on locality, appearance or traditional use rather than verified mineralogical composition[14].

This distinction is critical for process development. Elemental analysis showing high K or Na does not, by itself, establish the mineral phases responsible for those elements. For example, potassium may occur in carbonate, bicarbonate, chloride or sulfate phases, while sodium may

Grain Size Fractionation, CO2-Enhanced Aqueous Extraction and Characterization of Trona, a Non-marine Evaporite Mineral, Originating from Semi-arid Soil Sediments. Journal of the Geological Society of India. 2022;98(7):991–9. doi:10.1007/S12594-022-2105-Z; Imafidon KE, Egberanmwen ID, Omoregie IP. Toxicological and biochemical investigations in rats administered "kaun" (trona) a natural food additive used in Nigeria. J Nutr Intermed Metab. 2016;6:22–5. doi:10.1016/j.jnim.2016.05.003

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[14] Sinquin B, Loas G, Bouhier S, Frein L, Vallet M, Alouini M, et al. Laboratory Assessment of Different Kanwa-Based Mineral Lick for Ruminant Nutrition. Journal of Applied Life Sciences International. 2022;25(6):17–24. doi:10.34894/VQ1DJA

occur in carbonate, bicarbonate or hydrated mixed phases[15]. Therefore, the Yusufari data provide evidence of K-rich alkaline materials, but do not establish that K_2CO_3 is the dominant crystalline phase. Similarly, a Na-rich material should not automatically be classified as trona solely from its sodium concentration.

Accordingly, mineralogical identification should precede crystallisation-pathway selection. X-ray diffraction (XRD) should be used to establish crystalline phases, while complementary chemical analysis should determine the major soluble and insoluble constituents. This distinction prevents the process from being designed around an assumed mineral identity that may not correspond to the actual feedstock.

Implications for beneficiation and crystallisation

The available evidence suggests that the most appropriate way to evaluate Yobe potash/trona is to regard it as a composition-dependent mineral resource. The relevant processing question is therefore not simply how much K or Na is present in the raw material, but which phases are present, which components are soluble, and which species enter the crystallisation liquor.
This leads to a simple process principle: Resource identity → mineralogical composition → soluble-ion chemistry → crystallisation behaviour → product specification
Under this framework, materials with predominantly sodium-containing phases may be considered for sodium-based products, while K-rich materials may provide a basis for potassium-containing products. Mixed Na–K materials are potentially more challenging because selective recovery depends on the relative solubilities and phase relationships of the dissolved components. Calcium, magnesium, chloride, sulfate and insoluble matter may further influence phase formation and product purity[16].

Importantly, these classifications should be regarded as process descriptors rather than established mineralogical classes. Final pathway selection should be based on measured feed-liquor chemistry and experimentally verified phase behaviour rather than indigenous terminology alone. This feed-specific approach is particularly important when the recovered products are intended for petroleum applications, where chemical composition, impurity levels and phase consistency must satisfy defined performance requirements.

[15] Golovin A V. Unusual Mineralogy of Kimberlites: Alkali Carbonates, Sulfates, and Chlorides Among Groundmass Minerals from Unserpentinized Coherent Kimberlite of the Udachnaya-East Pipe, Siberian Craton. Minerals. 2025;15(6):586. doi:10.3390/MIN15060586

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Table 1. Selected indigenous potash/trona resources reported in Yobe State and their processing implications from the work of Sudi et al., 2016[17].

Resource/material	Location	K (mg kg⁻¹)	Na (mg kg⁻¹)	Ca (mg kg⁻¹)	Mg (mg kg⁻¹)	Processing implication
Potash	Madukuri, Yusufari	9,625–18,035	3,751–5,368	554–885	53.4–58.5	K-rich feed; verify mineral phase and assess Na/Ca/Mg before selective K recovery
Potash	Kotufa, Yusufari	8,855–12,493	3,625–3,912	448–933	57.4–58.8	Variable K–Na feed; feed-specific dissolution, impurity removal and crystallisation required
Potash	Kirba, Yusufari	11,472–25,436	3,535–5,535	603–828	54.4–58.3	Relatively K-rich feed; suitable for investigation of K-selective crystallisation after phase verification
Trona/kanwa	Nguru, Geidam and Gashua	—	—	—	—	Reported in different physical forms; pretreatment and Na_2CO_3 recovery should be adapted to feed characteristics

From Raw Mineral to Defined Crystallisation Feed

Crystallisation behaviour is governed by the composition of the solution entering the crystalliser rather than by the bulk composition of the original mineral alone[18]. For heterogeneous Yobe potash/trona resources, feed preparation is therefore an essential link between resource characterisation and crystallisation-process design. The objective is to convert the natural material into a clarified liquor of sufficiently known composition for controlled crystallisation while avoiding unnecessary dilution, contamination and uncontrolled precipitation.

A practical preparation sequence is:
Representative sampling → size reduction and homogenisation → dissolution → clarification → feed-specific pretreatment, where required → feed-liquor characterisation → crystallisation
The extent of pretreatment should depend on the feed composition and target product rather than follow a fixed sequence.

[17] Sudi P, Maina H, Bello H, Hauwa S, Modibbo A. Studies of Some Essential Elements Composition of Potash Deposits Found in Yusufari Local Government Area of Yobe State, Nigeria. American Chemical Science Journal. 2016;10(1):1–6. doi:10.9734/acsj/2016/20198

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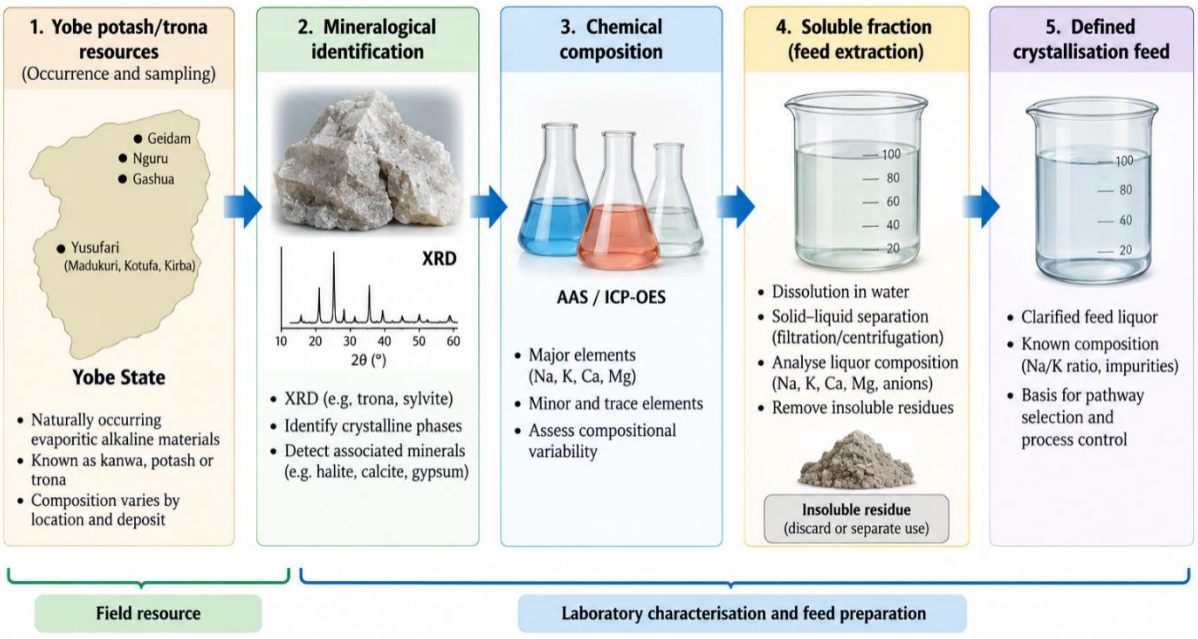


Figure 1. Yobe resource and feed-characterisation framework: From raw material to defined crystallisation feed.

Representative sampling and feed characterisation

Because Yobe potash/trona resources can vary among locations and batches, representative sampling is essential. Differences in crystallisation performance should not be attributed to process conditions when they may originate from variations in the starting material. Initial characterisation should establish the principal mineralogical and chemical characteristics of the feed, including:

- Mineralogical phases: XRD
- Major elemental composition: XRF, AAS or ICP-OES
- Major soluble anions: carbonate/bicarbonate, chloride and sulfate
- Moisture content
- Water-soluble fraction
- Insoluble matter
- Relevant trace constituents

XRD is particularly important because elemental analysis identifies the elements present but does not establish their crystalline phase. Determination of the water-soluble fraction is also important because only the soluble portion can directly enter the crystallisation liquor, whereas insoluble gangue can be removed during clarification. For comparative investigations, sample preparation, including drying, grinding and storage, should be standardised to minimise variability introduced during handling. For petroleum applications, particular attention should be given to Ca, Mg, insoluble matter and trace contaminants that may affect product performance and qualification.

Dissolution and clarification

Dissolution transfers the soluble components of the raw mineral into an aqueous phase suitable for subsequent crystallisation. The principal operating variables are the solid-to-liquid ratio, dissolution temperature, mixing intensity and contact time.

The solid-to-liquid ratio requires particular attention. Excessive water produces unnecessary dilution and increases the subsequent concentration duty, whereas insufficient water may limit dissolution and soluble-component recovery. The selected condition should therefore provide adequate extraction without excessive dilution.

Following dissolution, insoluble material should be removed by sedimentation, filtration, centrifugation or an appropriate combination of these operations. Clarification removes suspended gangue and particles that could contaminate the product or interfere with controlled crystallisation[19]. The resulting clarified liquor should be treated as the actual crystallisation feed. This distinction is fundamental: Raw-mineral composition ≠ crystallisation-feed composition. Selective dissolution, incomplete extraction and removal of insoluble matter can substantially change the composition of the solution entering the crystalliser.

Impurity management and thermal pretreatment

Pretreatment should be feed-specific rather than automatic. Calcium, magnesium, iron-bearing species, suspended solids and competing anions may influence crystallisation through competing precipitation, modification of nucleation and crystal growth, or incorporation into the recovered product. Their control is particularly important where the recovered alkaline product is intended for oilfield use, since residual impurities may affect fluid performance and downstream compatibility[20].

Impurity management

Where necessary, impurity management may involve controlled precipitation, pH adjustment, filtration, washing or recrystallisation. However, unnecessary purification should be avoided because additional treatment increases reagent and energy requirements, generates secondary waste and may remove potentially valuable components. A practical rule is therefore:
Identify impurity → determine its effect on crystallisation/product quality → remove only when justified.

Thermal pretreatment

Calcination may be appropriate for selected carbonate/bicarbonate feedstocks when thermal conversion produces a more suitable phase for dissolution or subsequent product manufacture. It should not, however, be regarded as a universal pretreatment for Yobe materials.

Thermal treatment can alter hydration state, carbonate/bicarbonate speciation and mineral phase while increasing energy demand. Therefore, where calcination is proposed, the sequence should be:
Phase identification → thermal analysis → controlled calcination → phase verification → dissolution/crystallisation testing.
TGA/DSC combined with XRD before and after heating can be used to verify the intended transformation.

[19] Chai L, Li Q, Wang Q, Yan X. Solid-liquid separation: an emerging issue in heavy metal wastewater treatment. Environmental Science and Pollution Research. 2018;25(18):17250–67. doi:10.1007/S11356-018-2135-7/

[20] Zhang H, Dong M, Ni J, Li B, Mao H, Chen G, et al. A comprehensive study of impact of solid particles on scaling formation and inhibition in oilfield water. Desalination Water Treat. 2025;323:101331. doi:10.1016/J.DWT.2025.101331

Feed-liquor characterisation and specification

Characterisation of the clarified liquor is the most important analytical step immediately before crystallisation because it defines the chemical system on which phase behaviour and separation performance depend.

At minimum, the feed liquor should be characterised for: K^+, Na^+, Ca^{2+}, Mg^{2+},CO_3^{2-}/HCO_3^-, Cl^-, SO_4^{2-}, pH, total dissolved solids or conductivity, temperature, density, where relevant, and selected trace constituents where required by the intended product specification.
For mixed Na–K feedstocks, the K/Na ratio should also be reported because changes in this ratio between the feed, recovered crystals and mother liquor provide a useful indication of separation behaviour.

A minimum reporting framework for crystallisation experiments is therefore:
Raw mineral → soluble fraction → clarified feed liquor → crystalline product/phase → mother liquor. This provides a stronger basis for evaluating separation than reporting crystal yield alone. The ultimate objective is not necessarily to produce the purest possible feed liquor, but a reproducible and chemically defined liquor whose composition is known sufficiently well to permit controlled crystallisation. Once established, the feed can be subjected to evaporation, cooling, fractional or other crystallisation pathways according to its phase behaviour and target product.

Solution Chemistry and Crystallisation Fundamentals

Crystallisation of indigenous potash/trona liquors is governed by the interaction of solution composition, solid–liquid equilibrium, supersaturation, nucleation and crystal growth. These factors determine the crystalline phase that forms, the extent of recovery and the physical characteristics of the product[21]. For compositionally variable Yobe resources, understanding these relationships is essential for selecting an appropriate crystallisation pathway and ultimately determine the suitability of the recovered product for its intended oilfield application.

Phase equilibrium, solubility and supersaturation

Solubility defines the equilibrium concentration of a dissolved component under specified conditions. If $C$is the actual concentration and $C^*$is the equilibrium solubility, the solution may be classified as:
$C < C^*$ — undersaturated
$C = C^*$ — saturated
$C > C^*$ — supersaturated

Crystallisation becomes thermodynamically possible when the solution enters the supersaturated region. A convenient measure is the supersaturation ratio:
$S = C/C^*$

[21] Li J, Deepak FL. In Situ Kinetic Observations on Crystal Nucleation and Growth. Chem Rev. 2022;122(23):16911–82. doi:10.1021/ACS.CHEMREV.1C01067

where $S < 1$, $S = 1$ and $S > 1$ represent undersaturated, saturated and supersaturated conditions, respectively. Supersaturation may be generated by evaporation, cooling, chemical reaction or combinations of these approaches[22].
For Yobe potash/trona, however, crystallisation cannot be described adequately using single-salt solubility data because the liquors are multicomponent. Interactions among K^+, Na^+, Ca^{2+}, Mg^{2+}, CO_3^{2-}/HCO_3^-, Cl^- and SO_4^{2-}, together with temperature and ionic composition, can modify phase stability and precipitation behaviour.

The ternary K_2CO_3–Na_2CO_3–H_2O system provides an important comparator. Yin et al. (2017) identified several crystallisation regions and phase boundaries at different temperatures, demonstrating that both solution composition and temperature influence the stable crystalline phase[23]. Importantly, this model system should be treated as transferable thermodynamic evidence rather than as a direct phase diagram for Yobe liquors.

Supersaturation also defines the metastable zone, in which existing crystals can grow while spontaneous primary nucleation remains limited. The width of this region is influenced by solution composition, temperature history, agitation and the presence of seed crystals [30]. It is therefore an important experimental parameter for controlling crystallisation rather than allowing uncontrolled nucleation. The key implication is that crystallisation selectivity depends on where the feed liquor lies within the relevant phase-equilibrium space, not simply on its total K or Na concentration.

Nucleation, crystal growth and seeding
Once sufficient supersaturation is established, crystals form through nucleation followed by crystal growth. Primary nucleation generates new crystals in the absence of pre-existing target crystals, whereas secondary nucleation occurs in the presence of crystals. Subsequent growth incorporates dissolved species onto crystal surfaces.

The balance between nucleation and growth strongly affects crystal size distribution. High nucleation with limited growth tends to produce numerous fine particles, whereas controlled nucleation followed by sustained growth favours fewer and larger crystals[24]. Crystal size and morphology are not merely physical descriptors; they influence filtration, washing, drying, handling and product recovery.

This becomes particularly important in multicomponent systems because different phases may nucleate simultaneously or sequentially. Penha et al. (2018), although working with the

[22] Ma H, Wang H, Tian C, Wang L, Yuan W, Qi Y, et al. An integrated membrane- and thermal-based system for coal chemical wastewater treatment with near-zero liquid discharge. J Clean Prod. 2021;291:125842. doi:10.1016/J.JCLEPRO.2021.125842; Guo P, Rao S, Hao L, Wang J. Fault diagnosis of a semi-batch crystallization process through deep learning method. Comput Chem Eng. 2022;164:107807. doi:10.1016/j.compchemeng.2022.107807

[23] Yin C, Liu M, Yang J, Ma H, Luo Z. (Solid + liquid) phase equilibrium for the ternary system (Na_2CO_3–K_2CO_3-H_2O) at T = (323.15, 343.15, and 363.15) K. J Chem Thermodyn. 2017;108:1–6. doi:10.1016/J.JCT.2017.01.001

[24] Zhang F, Shan B, Wang Y, Zhu Z, Yu ZQ, Ma CY. Progress and Opportunities for Utilizing Seeding Techniques in Crystallization Processes. Org Process Res Dev. 2021;25(7):1496–511. doi:10.1021/acs.oprd.1C00103

chemically different NaCl–KCl system, demonstrated that operating conditions influenced nucleation, growth, agglomeration and final product characteristics. This provides transferable evidence that product composition and particle quality are coupled to crystallisation kinetics[25]. Seeding introduces crystals of a known phase into a supersaturated solution to provide controlled growth surfaces. Proper seeding can reduce uncontrolled primary nucleation, improve reproducibility and influence crystal-size distribution and product composition. Important variables include seed phase identity, seed loading, particle size and the conditions at which seeds are introduced.

Penha et al. demonstrated the effect of KCl seeding on product segregation in simultaneous NaCl/KCl crystallisation[26]. For Yobe systems, the transferable lesson is more important than the specific salt system: seeding should be applied only after the target crystalline phase has been established. An inadequately characterised indigenous solid may introduce unwanted phases or uncontrolled heterogeneous nucleation. Thus, seed identity should be verified by XRD before optimisation.

Temperature, impurities and thermodynamics–kinetics integration

Temperature: Temperature affects crystallisation through changes in solubility, phase stability, supersaturation and nucleation/growth rates. In cooling crystallisation, supersaturation develops as solubility decreases with decreasing temperature, whereas in evaporative crystallisation temperature affects both solvent removal and phase equilibrium. In multicomponent carbonate systems, temperature can also change the identity of the stable solid phase.

Impurities: Natural Yobe liquors also contain non-target ions that may influence crystallisation. Ca^{2+}, Mg^{2+}, Cl^{-} and SO_4^{2-} may modify phase equilibrium, promote competing precipitation, affect nucleation and growth, adsorb onto growing crystal surfaces or become incorporated through mother-liquor entrainment. However, the mere presence of an impurity does not mean that it must be removed. Its significance should be judged according to its concentration, effect on phase behaviour and influence on the specification of the intended product.

Thermodynamics versus kinetics: A useful distinction for process design is that thermodynamics determines which phases can form; kinetics determines when and how rapidly they form. Phase-equilibrium information identifies the saturation boundaries and potentially stable solid phases, whereas nucleation and growth kinetics determine how reproducibly those phases can be formed under practical operating conditions.

For indigenous Yobe resources, the overall sequence can therefore be represented as:

> Feed-liquor composition → phase equilibrium → supersaturation → nucleation and growth → crystal phase/size/morphology → recovery, purity and selectivity.

Crystallisation Pathways for Indigenous Potash/Trona

Selection of a crystallisation pathway for Yobe potash/trona should be governed by feed-liquor composition, phase behaviour, impurity burden and target-product specification, rather than

[25] Penha FM, Zago GP, Nariyoshi YN, Bernardo A, Seckler MM. Simultaneous Crystallization of NaCl and KCl from Aqueous Solution: Elementary Phenomena and Product Characterization. Cryst Growth Des. 2018;18(3):1645–56. doi:10.1021/acs.cgd.7b01603

[26] Penha FM, Zago GP, Seckler MM. Strategies To Control Product Characteristics in Simultaneous Crystallization of NaCl and KCl from Aqueous Solution: Seeding

indigenous nomenclature alone. The objective is not simply to maximise the mass of recovered solids, but to direct the liquor through appropriate phase boundaries so that the desired component is recovered with acceptable purity, selectivity and product quality. For petroleum applications, pathway selection must ultimately provide products with the composition, purity and phase characteristics required for the intended oilfield use.

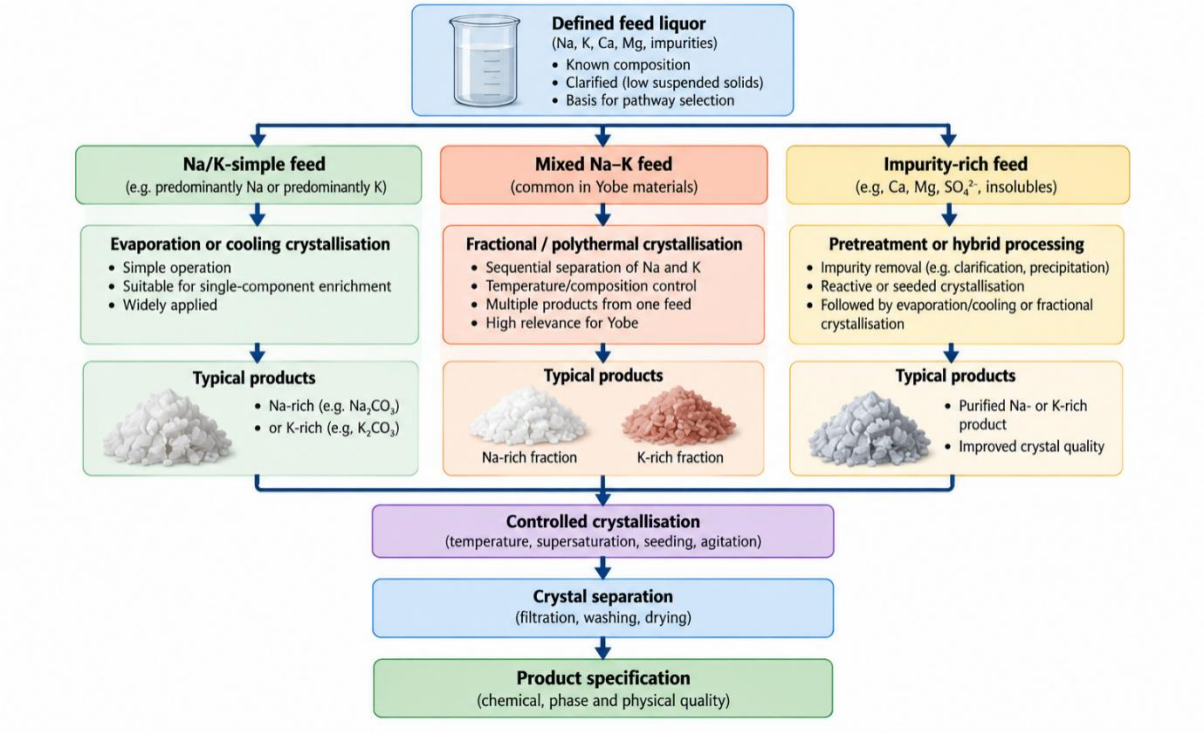


Figure 2. Crystallisation pathway-selection framework for Yobe potash/trona feeds.

Evaporative and cooling crystallisation

Evaporative crystallisation: Evaporative crystallisation generates supersaturation through solvent removal and increasing dissolved-solids concentration. Its relatively simple operation makes it attractive for indigenous mineral processing, particularly for feeds dominated by a recoverable Na- or K-containing component. Its main limitation is reduced selectivity as solution complexity increases. In mixed Na–K systems, progressive evaporation can cross multiple phase boundaries and produce sequential or simultaneous precipitation of competing, hydrated or double salts. The K_2CO_3–Na_2CO_3–H_2O system demonstrates why complete evaporation cannot automatically be assumed to achieve selective Na/K separation[27].

For Yobe materials, evaporative crystallisation is therefore most appropriate for relatively simple feeds, preliminary concentration before a subsequent separation stage, or applications where moderate selectivity is acceptable. Dilute feeds should be avoided where possible because unnecessary water removal increases energy demand.

Cooling crystallisation: Cooling crystallisation generates supersaturation through a decrease in solubility with temperature. It can reduce the amount of water that must be removed and may provide better control of supersaturation than complete evaporation. However, its

[27] Yin C, Liu M, Yang J, Ma H, Luo Z. (Solid + liquid) phase equilibrium for the ternary system (Na_2CO_3–K_2CO_3-H_2O) at T = (323.15, 343.15, and 363.15) K. J Chem Thermodyn. 2017;108:1–6. doi:10.1016/J.JCT.2017.01.001

suitability is strongly phase-dependent because temperature can affect solubility, hydration state and the identity of the crystallising phase[28].
Cooling may therefore be applied directly or after evaporative pre-concentration:

Feed liquor → pre-concentration → controlled cooling → crystallisation

The cooling trajectory should be selected to maintain manageable supersaturation and controlled crystal growth rather than excessive fine-particle formation. Consequently, cooling should be considered a phase-specific option requiring experimental solubility–temperature data, rather than a universally preferred route.

Fractional and polythermal crystallisation

Among the available pathways, fractional/polythermal crystallisation is the most promising for investigating selective separation of mixed Na–K Yobe feedstocks. Rather than allowing the entire dissolved-solids inventory to precipitate together, the process aims to recover different components sequentially as the liquor moves through changing composition and temperature conditions.
The basic sequence is:

> Mixed feed liquor → controlled concentration/temperature change → first phase crystallisation → crystal separation → altered mother liquor → second crystallisation stage

The approach depends on differences in phase stability and solubility. Removing the first crystal fraction changes the composition of the remaining liquor and can create conditions for subsequent recovery of another phase.
The K_2CO_3–Na_2CO_3–H_2O phase relationships reported by Yin et al. (2017) provide a useful thermodynamic basis for this concept. They show that the phase recovered depends on both composition and temperature, indicating that the trajectory through composition–temperature space is an important process variable[29].
For a Yobe mixed liquor, a representative strategy is:

> Characterised feed → controlled evaporation/cooling → first saturation boundary → crystal separation → mother-liquor analysis → second operating condition → secondary recovery

This approach is particularly attractive because it offers the possibility of converting mixed feed chemistry into sequentially recoverable products rather than treating the residual mother liquor as waste.

However, no specific Na/K fractional-crystallisation sequence should presently be prescribed for Yobe resources. Real liquors may contain carbonate/bicarbonate, chloride, sulfate, Ca^{2+} and Mg^{2+} in addition to Na^+ and K^+, and these components may substantially modify phase equilibria. Therefore, experimental solid–liquid equilibrium mapping of representative Yobe liquors is a prerequisite for rational fractional/polythermal process design.

Recrystallisation and seeded crystallisation

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[28] Guo P, Rao S, Hao L, Wang J. Fault diagnosis of a semi-batch crystallization process through deep learning method. Comput Chem Eng. 2022;164:107807. doi:10.1016/j.compchemeng.2022.107807; Wang S, Feng M, Du H, Weigand JJ, Zhang Y, Wang X. Determination of metastable zone width, induction time and primary nucleation kinetics for cooling crystallization of sodium orthovanadate from NaOH solution. J Cryst Growth. 2020;545:125721. doi:10.1016/j.jcrysgro.2020.125721

[29] ibid

Recrystallisation: Recrystallisation is primarily a product-upgrading operation. It is useful when an initial crystalline product has the desired major component but insufficient purity. A simplified sequence is:

Crude crystal → redissolution → clarification → controlled recrystallisation → separation → washing → drying

Purification may arise from removal of insoluble matter during clarification, preferential retention of soluble impurities in the mother liquor and removal of adhering liquor during crystal washing. Gärtner and Witkamp (2007) demonstrated the conversion of trona to anhydrous sodium carbonate through reactive recrystallisation, illustrating how recrystallisation can combine phase transformation and purification[30].

For Yobe resources, conventional aqueous recrystallisation should generally be considered before more complex solvent-based approaches because solvent recovery, cost, safety and process simplicity will be important in eventual local implementation.

Seeded crystallisation: Seeded crystallisation should be regarded primarily as a crystal-control strategy within another crystallisation pathway rather than as an independent Na/K separation method. Introducing crystals of a verified phase into a supersaturated liquor can promote controlled growth, reduce uncontrolled primary nucleation and improve crystal-size distribution and reproducibility. Penha et al. (2019) demonstrated the influence of seed characteristics on product segregation in the NaCl–KCl system; although chemically different from Yobe carbonate systems, the result illustrates the broader importance of seed policy[31]. For Yobe applications: Seeding should follow phase identification, not substitute for it. The seed phase should therefore be confirmed by XRD before seed loading, particle size and addition conditions are optimised.

Reactive and hybrid crystallisation pathways

Reactive crystallisation generates supersaturation through chemical reaction or deliberate modification of solution speciation. In carbonate-containing systems, this may involve pH adjustment, carbonation/decarbonation or selective precipitation of interfering components[32]. For Yobe resources, its principal value is likely to be impurity removal, phase conversion or feed conditioning before a subsequent crystallisation stage.

The main disadvantage is that reagent addition can introduce new ions, increase waste generation and raise operating costs. Reactive treatment should therefore be used only where the resulting improvement in selectivity or product quality justifies the additional complexity. For compositionally complex resources, a hybrid pathway may be more appropriate than a single-unit operation. A representative sequence is:

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[30] Gärtner RS, Witkamp GJ. Mixed solvent reactive recrystallization of trona (sodium sesqui-carbonate) into soda (sodium carbonate anhydrate). Hydrometallurgy. 2007;88(1–4):75–91. doi:10.1016/j.hydromet.2007.03.006

[31] Penha FM, Zago GP, Seckler MM. Strategies To Control Product Characteristics in Simultaneous Crystallization of NaCl and KCl from Aqueous Solution: Seeding with KCl. Cryst Growth Des. 2019;19(2):1257–67. doi:10.1021/acs.cgd.8B01670

[32] Rao AR, Tide PS, George BK, Mathew J. Quasi-dynamic model for dissolution coupled with reaction and precipitation of sodium bicarbonate in fed-batch reactive crystallization. Chemical Engineering Journal Advances. 2023;15:100504. doi:10.1016/j.ceja.2023.100504

Feed characterisation → impurity management → pre-concentration → fractional crystallisation → crystal separation → recrystallisation/seeding, where required → washing and drying

Each operation should have a clearly defined role. This is preferable to adding process steps simply because they are technically available. Processing intensity should remain proportional to the required product quality and economic value.

Pathway selection for Yobe feedstocks

No single crystallisation pathway can presently be designated as universally optimal for Yobe potash/trona because the mineralogical and feed-liquor compositions vary among resources. The pathway should therefore be selected according to the feed chemistry and desired product. A practical selection sequence is:

Characterise feed → identify target phase/product → establish relevant phase behaviour → select pathway → control crystallisation → validate product

In general, relatively simple Na- or K-dominant feeds may be suitable for evaporative or cooling crystallisation; mixed Na–K feeds warrant particular attention to fractional/polythermal crystallisation; crude products may require recrystallisation for upgrading; verified seed crystals can be incorporated to control crystal growth and particle properties; and impurity-rich feeds may require reactive or hybrid treatment.

The overall feed–process–product relationship can be summarised as:

Feed composition → appropriate crystallisation pathway → controlled phase separation → product specification

Thus, crystallisation-route selection should follow verified feed chemistry and phase behaviour rather than indigenous nomenclature alone.

Table 2. Comparative suitability of crystallisation pathways for Yobe potash/trona feedstocks.

Pathway	Best-matched feed	Main advantage	Main limitation	Yobe priority
Evaporative	Simple Na-/K-dominant feed	Simple operation	Energy demand; co-crystallisation	Moderate
Cooling	Feed with favourable solubility–temperature behaviour	Lower water-removal duty	Requires phase/solubility data	Conditional
Fractional/polythermal	Mixed Na–K feed	Sequential separation potential	Requires SLE data	Critical
Recrystallisation	Crude crystalline product	Product purification	Product loss to mother liquor	High
Seeded	Verified target phase	Crystal-growth control	Requires verified seed phase	High
Reactive/hybrid	Complex or impurity-rich feed	Targeted conditioning/separation	Reagents and process complexity	Case-specific

Process Control and Optimisation of Indigenous Potash/Trona Crystallisation

Selection of an appropriate crystallisation pathway does not by itself guarantee satisfactory separation or product quality. Process performance depends on the interaction of feed composition, temperature, supersaturation generation, mixing, residence time and, where

applicable, seeding. For heterogeneous Yobe potash/trona liquors, process control should therefore aim to balance recovery, selectivity, purity, crystal quality and process efficiency rather than maximise crystal yield alone. For petroleum applications, these objectives must be considered together with the performance requirements of the intended oilfield chemical.

Process-performance criteria

A useful optimisation framework should distinguish between recovery and purity. High recovery indicates that a large fraction of the target component has been transferred from the feed liquor to the crystalline product, whereas high purity indicates that the recovered solid contains a high proportion of the desired component. These responses may not increase simultaneously.

For mixed Na–K systems, process performance should therefore consider: target-component recovery, chemical purity, phase selectivity/purity, K/Na or Na/K enrichment, impurity rejection, crystal-size and handling characteristics, and processing time and energy/resource demand. The operating objective can consequently be expressed simply as: Maximise recovery, purity, selectivity and desirable crystal characteristics. Minimise impurity incorporation, unnecessary processing time and energy demand subject to the required crystalline phase and product specification. Where products are intended for drilling fluids or other oilfield applications, impurity rejection and composition consistency become particularly important because they directly affect product qualification and application performance.

This approach avoids the important error of treating maximum solid recovery as equivalent to optimum crystallisation. High recovery may be accompanied by poor purity, whereas high purity may occur with unacceptable target loss to the mother liquor.

Key operating variables

Feed composition and initial concentration: Feed composition is the primary constraint on crystallisation because the concentrations of Na^+, K^+ and competing ions determine the phase boundaries approached during evaporation or cooling. For mixed Na–K feeds, the K/Na ratio, total dissolved solids, carbonate/bicarbonate concentration and levels of Ca^{2+}, Mg^{2+}, Cl^- and SO_4^{2-} are particularly important.

Initial concentration also influences process efficiency. Excessive dilution increases the subsequent water-removal requirement, whereas excessive concentration may induce premature nucleation or competing precipitation. The appropriate starting concentration should therefore provide sufficient supersaturation potential without compromising phase control.

Temperature and supersaturation-generation rate: Temperature and the rate of supersaturation generation influence nucleation, crystal growth and phase selection. Rapid evaporation or cooling may generate excessive supersaturation and produce fine crystals, while excessively slow operation may increase processing time and energy demand[33]. For mixed systems, temperature must also be considered together with feed composition because changing

[33] Shaddel S, Ucar S, Andreassen JP, Sterhus SW. Engineering of struvite crystals by regulating supersaturation – Correlation with phosphorus recovery, crystal morphology and process efficiency. J Environ Chem Eng. 2019;7(1):102918. doi:10.1016/j.jece.2019.102918

temperature can alter the stable crystalline phase. Consequently, the desired operating condition is not the fastest possible evaporation or cooling rate, but one that provides:

> controlled supersaturation → manageable nucleation → sustained growth → desired phase and crystal characteristics.

Mixing, time and process endpoint: Adequate mixing promotes uniform temperature and concentration throughout the crystalliser, whereas insufficient mixing can produce local supersaturation gradients. Excessive agitation, however, may increase attrition and secondary nucleation. Agitation speed, vessel geometry and crystallisation time should therefore be reported in experimental work to support reproducibility.

The process endpoint is equally important. Extending crystallisation may increase total solid recovery, but in fractional crystallisation it may also move the liquor into another phase region and promote co-crystallisation. Thus: Maximum crystallisation is not necessarily optimum crystallisation.

The endpoint should instead be judged from the combination of product recovery, product composition/phase and mother-liquor composition.

Seeding: Where seeded crystallisation is used, the principal controllable variables are seed phase, seed loading, seed particle size and the conditions of seed addition. However, as established in Sections 4 and 5, these variables should only be optimised after the target phase has been identified.

Experimental optimisation strategy

A simple strategy is:

> Preliminary experiments → identify feasible operating ranges → screen important variables → evaluate interactions where necessary → select practical operating conditions → experimental validation

Initial one-variable-at-a-time experiments can be used for range finding, particularly for concentration, temperature, crystallisation time and agitation. Once realistic operating ranges have been established, multivariable experimental design may be used where several factors are expected to interact.

The most important point is that optimisation should be experimentally validated, rather than relying solely on a statistically predicted optimum.

Robustness for variable Yobe feedstocks

A major challenge for indigenous Yobe resources is feed variability. An operating condition that performs well for one K/Na ratio or impurity level may not perform equally well for another feed composition. This limitation highlights the risk of relying on a single mathematical optimum for compositionally variable feedstocks.

Future optimisation should therefore consider a representative range of feed compositions rather than one nominal feed. A practical sequence is:

> Define representative feed range → identify influential variables → establish operating conditions → experimentally validate → test across feed variability → define an acceptable operating window

The objective is consequently not a single optimum point, but an operating window within which acceptable recovery, purity and selectivity are maintained despite realistic variation in

feed chemistry. This approach is particularly appropriate for Yobe potash/trona because resource heterogeneity is an intrinsic characteristic of the material rather than simply an experimental inconvenience. For oilfield applications, validation across realistic feed variability is therefore essential to ensure consistent product quality and reliable field performance.

Product Quality and Characterisation

Successful crystallisation should not be judged solely by the mass of solids recovered. For indigenous Yobe potash/trona, product quality depends on whether the recovered crystals possess the required chemical composition, crystalline phase, physical characteristics, stability and application fitness. A product may show high chemical purity while containing an undesirable crystalline phase, or acceptable phase purity while exhibiting poor filtration, washing or storage characteristics. Product assessment should therefore follow a specification-driven approach in which the intended application determines the critical quality attributes and the required level of purification. For petroleum applications, these quality attributes must additionally be assessed against the performance and compatibility requirements of the intended oilfield use.

Chemical and phase quality

Chemical composition is a primary measure of crystallisation performance, but chemical purity and phase purity are not equivalent. Recovery indicates the fraction of the target component transferred from the feed liquor into the crystalline product, whereas purity indicates the proportion of the target component within the recovered solid.

For mixed Na–K feedstocks, product evaluation should therefore determine the distribution of Na, K and relevant impurities among the feed liquor, crystalline product and mother liquor. Useful indicators include: target-component recovery, product chemical purity, K/Na or Na/K enrichment, impurity rejection, and target-component loss to the mother liquor.

However, elemental analysis alone cannot establish the identity of the recovered solid. XRD should be used to confirm the desired crystalline phase and identify competing, hydrated or double-salt phases. This is particularly important for indigenous alkaline materials because a Na-rich or K-rich product cannot automatically be assigned to a specific mineral or salt phase from elemental composition alone.

A practical minimum characterisation package is therefore a quantitative chemical analysis + XRD phase identification. FTIR or Raman spectroscopy may be added where structural confirmation is required.

Physical properties and stability

Crystallisation also determines the physical characteristics of the recovered product. Important properties include crystal-size distribution, morphology, agglomeration, bulk density, filtration

behaviour, washability and flowability because these characteristics directly influence solid–liquid separation, drying, storage and handling[34].

Particle-size distribution may be evaluated by sieving or particle-size analysis, while optical microscopy or SEM can provide information on crystal habit and agglomeration. Fine crystals generated by excessive nucleation may be difficult to filter and wash, whereas controlled crystal growth generally improves downstream handling. Nevertheless, larger crystals are not universally better; the preferred distribution depends on the intended application.
Washing is particularly important because adhering mother liquor can contribute soluble impurities to the final product and distort the apparent chemical purity. Washing conditions should therefore remove residual liquor while minimising dissolution and loss of the target crystals.

On product stability, recovered crystalline products must also retain their required properties during drying and storage. Carbonate-based materials may undergo hydration/dehydration, moisture uptake, caking or phase transformation, depending on their composition and environmental conditions[35]. For petroleum applications, storage stability and reproducible dissolution behaviour are particularly important because the product may be incorporated into formulated drilling or production fluids.

Relevant stability measurements may include moisture content, thermal behaviour, hygroscopicity and storage stability. TGA/DSC can assist in evaluating hydration and thermal transformations, while XRD before and after drying or storage can reveal changes in crystalline phase. Thus, product quality should be considered not only at the point of crystal recovery but also after washing, drying and storage.

Application-specific specifications and integrated quality assessment
There is no single purity value that defines a successful Yobe potash/trona product. The required specification depends on its intended application. Trace metals or other contaminants that may be acceptable in some technical applications may be unacceptable for petroleum operations where tighter control of product composition and compatibility is required. The appropriate logic is therefore:

> Target application → required specification → critical quality attributes → required purification intensity

For petroleum applications, recovered alkaline products should be evaluated against the relevant American Petroleum Institute (API) or equivalent industry specifications applicable

[34] Yu ZQ, Chew JW, Chow PS, Tan RBH. Recent Advances in Crystallization control: An Industrial Perspective. Chemical Engineering Research and Design. 2007;85(7):893–905. doi:10.1205/cherd06234; Fang L, Gao Z, Wu S, Jia S, Wang J, Rohani S, et al. Ultrasound-assisted solution crystallization of fotagliptin benzoate: Process intensification and crystal product optimization. Ultrason Sonochem. 2021;76:105634. doi:10.1016/j.ultsonch.2021.105634

[35] Patel AS, Raudsepp MJ, Wilson S, Harrison AL. Water Activity Controls the Stability of Amorphous Ca–Mg- and Mg-Carbonates. Cryst Growth Des. 2024;24(5):2000–13. doi:10.1021/acs.cgd.3c01283; Elahi B, Salehzadeh D, Vos WM de, Shahidzadeh N, Brem G, Mehrali M. Boosting stability of K_2CO_3 granules for thermochemical heat storage applications through innovative membrane encapsulation. Chemical Engineering Journal. 2024;500:157042. doi:10.1016/j.cej.2024.157042

to the intended use [42]. Particular attention should be given to Ca, Mg, insoluble matter, trace contaminants, phase composition and other properties that may influence fluid performance, compatibility or scaling behaviour in drilling and production operations.

For Yobe potash/trona, integrated product quality can therefore be assessed through five dimensions as presented in Table 3:

Table 3. Quality attributes and characterisation requirements for crystalline products recovered from Yobe potash/trona.

Quality dimension	Key attributes	Main characterisation
Chemical purity	Na/K content, impurities, composition consistency	AAS/ICP-OES, ion analysis, titration
Phase purity	Target phase, hydrates, competing/double salts	XRD; FTIR/Raman where needed
Physical quality	Size, morphology, agglomeration, density, filtration	Particle-size analysis, microscopy/SEM
Stability	Moisture, hydration and thermal/storage behaviour	Moisture analysis, TGA/DSC, XRD
Application fitness	Trace contaminants, application compatibility and oilfield specification compliance	ICP-based trace analysis and application-specific performance tests

Importantly, these attributes can compete. Additional recrystallisation may increase purity but reduce recovery; aggressive washing may reduce impurities while dissolving some product; and conditions that maximise recovery may produce fines that complicate filtration. Product development should therefore seek the best balance between product quality and process performance, rather than the highest possible laboratory purity.

Value Addition and Applications

The value of indigenous Yobe potash/trona resources depends not only on their occurrence or total K and Na contents, but on the ability to convert heterogeneous materials into reproducible products with defined chemical, mineralogical and physical properties. Crystallisation provides an important route for achieving this transition through concentration, separation, purification and control of the recovered crystalline phase.

Value addition should therefore follow a resource-to-product hierarchy, with processing intensity increasing only where the resulting improvement in product quality provides sufficient technical or economic benefit for the intended oilfield application, as illustrated in Figure 3.

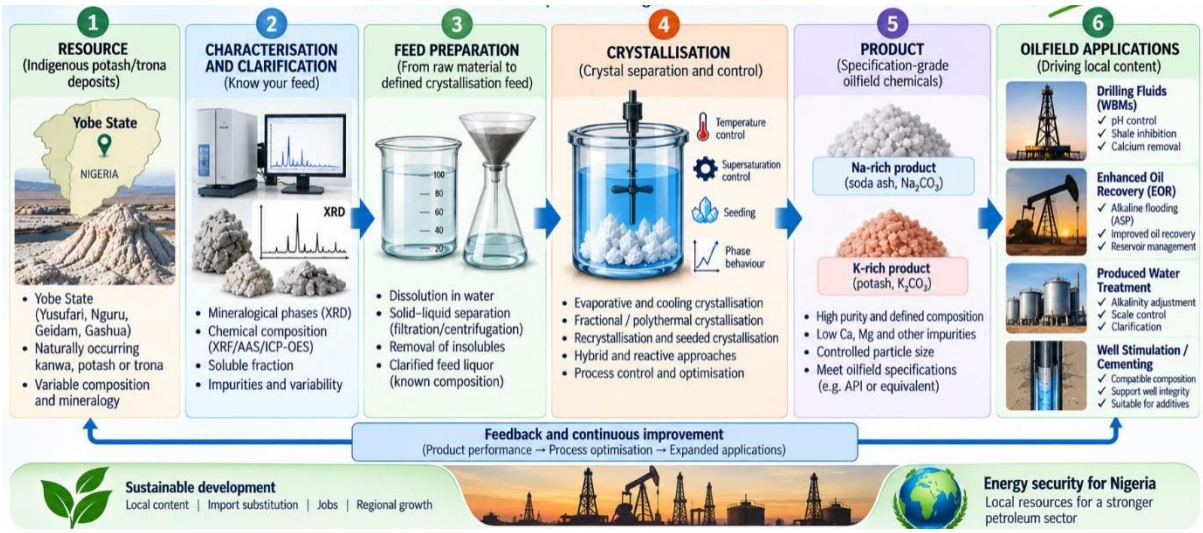


Figure 3. Resource-to-product and value addition roadmap for Yobe potash/trona

Value-addition pathway

A progressive value-addition pathway for Yobe potash/trona can be represented as:

Crude indigenous mineral
↓
Beneficiated and characterised feed
↓
Purified crystalline product
↓
Specification-grade chemical
↓
Application-specific product

Initial beneficiation may involve sorting, washing, size classification and removal of insoluble material. This can improve consistency for relatively low-specification uses but does not by itself produce a defined chemical product. A higher level of value addition is achieved through controlled dissolution and crystallisation to obtain a chemically and mineralogically defined product suitable for further evaluation as an oilfield chemical. Further washing, recrystallisation or fractional crystallisation may then be applied where higher purity, phase control or Na/K selectivity is required. Importantly, not every feedstock should be expected to progress to the highest processing level. The appropriate product grade depends on feed composition, attainable quality, processing cost and market requirements.
This leads to an important reverse-design principle:

> Target market → product specification → required product quality → purification intensity → crystallisation pathway

Application-specific opportunities

The compositional diversity of Yobe potash/trona may support several potential product pathways. However, application suitability should be established from the verified properties of the recovered product, rather than from the name assigned to the raw mineral.

Table 4. Potential petroleum industry applications, product requirements, and processing levels for value-added Yobe potash/trona.

Potential Application	Desired Product Characteristics	Main Quality Concern	Likely Processing Level
Drilling Fluids (WBMs)	Defined Na-rich composition (soda ash); high solubility	Insolubles, calcium/magnesium contamination	Purified / Standardised
Enhanced Oil Recovery (ASP Flooding)	Controlled alkalinity; appropriate Na/K composition	Trace heavy metals, phase consistency	Purified – Specification grade
Produced Water Treatment	Predictable alkalinity for water softening/clarification	Insolubles and organic contaminants	Beneficiated / Purified
Well Stimulation / Cementing	Defined composition; compatible with polymer additives	Moisture, hydration stability	Specification-specific

These applications represent specialised value chains within the upstream and oilfield service sectors. Product qualification should be evaluated against relevant API or equivalent industry requirements, since total elemental K or Na content alone does not establish suitability for a specific drilling or production application.[36]

Matching processing intensity with product value

Greater purification does not automatically produce greater economic value. Additional dissolution, crystallisation, recrystallisation, washing, drying and quality-control operations consume water, energy and other resources and may reduce overall product recovery. The appropriate processing intensity should therefore be determined by the balance between the added value of the upgraded product and the additional cost required to achieve its specification. A simple design principle is that added product value should justify added processing intensity.

For example, a relatively simple Na-rich feed may be more appropriately processed into a standardised technical-grade product, whereas a favourable K-rich fraction may justify additional selective crystallisation if the resulting product meets a higher-value application requirement. Conversely, extensive purification of a low-value product may be technically feasible but economically unattractive.

[36] Nzerem P, Adejoh EJ, Ogolo O, Okafor I, Gimba ASB, Choir TJ, et al. Study of the Potential of Sodium Carbonate Extracted from Trona as a Drilling Fluid Additive. International Journal of Engineering Research in Africa. 2020;49:152–72. doi:10.4028/www.scientific.net/jera.49.152

Feed heterogeneity can therefore become an opportunity for product differentiation:

Na-rich feed → Na-oriented product
K-rich feed → K-oriented product
Mixed Na–K feed → selective/fractional recovery
Impurity-rich feed → targeted pretreatment or application-specific upgrading

The resulting value-addition relationship is:

Resource chemistry → processing intensity → product specification → target application → economic value

Accordingly, the most rational strategy for Yobe is unlikely to be universal purification of all indigenous potash/trona to a single grade. Instead, representative feedstocks should be matched with the highest-value product that can be produced reproducibly at an appropriate processing intensity.

Research gaps, Future directions and Development priorities

Despite evidence of indigenous potash/trona resources in Yobe State, converting them into reproducible, specification-driven products remains constrained by limited information linking resource composition, mineralogical identity, feed-liquor chemistry, crystallisation behaviour, and product performance. Available studies establish resource occurrence and elemental composition, but Yobe-specific information on phase equilibria, crystallisation kinetics, selective Na/K recovery and process scale-up remains limited.

Future work should therefore move from isolated compositional studies toward an integrated resource–process–product framework. The main priority is to establish which products can be recovered from representative Yobe feedstocks, under what crystallisation conditions, and to what level of recovery, purity and selectivity. For petroleum applications, these questions must ultimately be linked to the specifications, performance requirements and economic constraints of oilfield chemical supply.

Resource and phase-equilibrium definition

The first priority is systematic characterisation of representative Yobe resources. Existing Yusufari data demonstrate significant variation in K, Na, Ca and Mg contents, but elemental composition alone cannot establish mineralogical identity or predict crystallisation behaviour. Future investigations should therefore combine:

Representative sampling → XRD phase identification → major cation/anion analysis → soluble/insoluble fraction determination → trace-contaminant assessment

The resulting resource classification should be process-oriented, linking feed chemistry to likely crystallisation behaviour rather than relying solely on locality or indigenous name. A second priority is determination of solid–liquid equilibrium (SLE) for representative feed liquors. Model studies such as the K_2CO_3–Na_2CO_3–H_2O system demonstrate that mixed carbonate solutions can contain multiple crystallisation regions whose boundaries depend on temperature and composition. However, these simplified systems cannot be assumed to reproduce the behaviour of real Yobe liquors containing additional ions.

Future SLE studies should therefore progress from simplified systems toward representative multicomponent Yobe liquors. The desired outcome is a practical relationship between:

Feed composition → temperature → equilibrium liquor composition → stable crystalline phase

Such information would provide the thermodynamic basis for rational pathway selection.

Selective crystallisation and process validation

Mixed Na–K resources deserve particular attention because fractional/polythermal crystallisation may enable sequential recovery of differentiated products. Rather than evaporating a mixed liquor to dryness, studies should investigate controlled changes in temperature and concentration followed by separation and analysis of successive crystal fractions. A practical research sequence is:

Mixed feed → first crystallisation condition → crystal separation → mother-liquor analysis → second condition → secondary product

The central question is whether representative Yobe feedstocks can produce distinct Na-rich and K-rich product streams with acceptable recovery and purity. Thermodynamic feasibility must be complemented by kinetic information. Priority measurements include metastable-zone behaviour, induction time, nucleation, crystal growth, seeding response, agglomeration and crystal-size distribution.

Process optimisation should then focus on experimentally validated operating ranges rather than a single mathematical optimum. The important responses remain recovery, chemical purity, phase selectivity, crystal characteristics and resource demand, but these should be evaluated across realistic variations in feed composition. This is particularly important for Yobe because feed variability is an inherent characteristic of the resource. The desired outcome is therefore a robust operating window, not merely the optimum condition for one laboratory feed.

Product validation and scale-up

Crystallisation research becomes technologically meaningful only when the recovered solid can be linked to a defined product specification. Future studies should therefore evaluate products using the quality framework developed in Section 7, including chemical composition, phase identity, physical properties, stability and application-specific requirements. For petroleum applications, validation should include application-specific performance testing in addition to chemical, mineralogical and physical characterisation.

Material balances should also track valuable components among the crystalline product, mother liquor, wash liquor and insoluble residue. This is especially important where sequential crystallisation is used because significant Na or K may remain available for secondary recovery. Mother-liquor recycling should therefore be investigated while monitoring possible impurity accumulation and changes in phase behaviour.

At larger scale, particular attention should be given to mixing, heat transfer, residence time, filtration and crystal-size distribution because laboratory performance may not be maintained automatically under different hydrodynamic and thermal conditions. For Yobe, a flexible pilot-scale crystallisation system capable of handling different representative feedstocks would provide a useful bridge between laboratory research and industrial implementation.

9.4 Techno-economic and environmental assessment

Technical performance alone is insufficient to establish the viability of a new crystallisation pathway. Once reliable mass and energy balances are available, alternative process routes should be compared using techno-economic analysis (TEA) and, where appropriate, environmental assessment.

Key considerations include energy and water consumption, reagent demand, product recovery, mother-liquor recycling, and waste generation. Future techno-economic evaluations should

benchmark the projected production cost of Yobe-derived alkaline chemicals against the import-parity price of commercially available soda ash used in relevant Nigerian petroleum operations. The analysis should quantify the potential economic benefit of domestic production, including raw-material availability, processing, energy and water requirements, product recovery, logistics, quality-control costs and potential savings from import substitution. For the Nigerian petroleum sector, the preferred route should therefore combine technical performance, specification compliance, supply reliability and competitive cost.

Where sufficient process data become available, life-cycle assessment can complement TEA in comparing alternative flowsheets. Commercial development will additionally require standardised sampling, analytical methods, product specifications and quality-control procedures.

Conclusion

Indigenous potash/trona resources in Yobe State represent potentially valuable alkaline mineral feedstocks for local beneficiation and the production of specification-grade chemicals for petroleum-related applications. However, their chemical and mineralogical heterogeneity means that indigenous terms such as potash, *kanwa* and trona cannot alone define feed behaviour or product identity. Representative sampling, mineralogical verification and characterisation of the soluble fraction are therefore essential before crystallisation conditions are selected.

Crystallisation provides a practical route for concentrating, separating and upgrading these resources, but the appropriate pathway must be matched to feed chemistry and target-product requirements. Evaporative and cooling crystallisation may suit relatively simple feeds, while fractional/polythermal crystallisation offers an important research direction for mixed Na–K systems. Recrystallisation, seeding and targeted pretreatment can provide additional control of purity and crystal quality. For petroleum applications, these operations must ultimately deliver products with consistent composition, controlled impurities and properties appropriate for the intended oilfield use. The principal research priorities are Yobe-specific phase-equilibrium and kinetic data, selective crystallisation, product validation against oilfield standards, and scale-up. Ultimately, successfully domesticating these alkaline resources depends on matching the crystallisation processes to the rigorous technical and economic standards of the Nigerian petroleum industry, thereby driving local content development and reducing reliance on imported chemical feedstocks.