Cold-Alkaline Processed Enset (Ensete ventricosum) Starch: Optimization of Gel Formation, Neutralization, and Applications in Adhesives and Cosmetics

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Research Article

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Cold-Alkaline Processed Enset (Ensete ventricosum) Starch: Optimization of Gel Formation, Neutralization, and Applications in Adhesives and Cosmetics

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Abstract:

Background: Enset (Ensete ventricosum) is a staple food crop in Ethiopia, traditionally processed into kocho and bulla (purified starch). While enset starch possesses favorable physicochemical properties including high purity (99.24%), 29% amylose content, and B-type crystallinity, its industrial applications remain largely unexplored. Conventional starch processing requires heating, which limits community-scale production. Cold-alkaline treatment offers an energy-efficient alternative, but systematic optimization for enset starch has not been documented.

Objective: This study aimed to optimize cold-alkaline processing parameters for enset starch gel formation, establish precise neutralization protocols, and develop formulations for adhesive, cosmetic, and hair care applications suitable for community-scale production.

Methods: Enset starch (bulla) was mixed with water at varying ratios (1:33 to 1:8.3 w/v) and treated with 20% sodium hydroxide solution (2-6 mL per 5 g starch) at room temperature. Gel formation time, appearance, and pH were recorded. Neutralization was performed using 4M hydrochloric acid, with pH monitored after each addition. For adhesive applications, 5% borax solution (20-50 mL/L) was added, and time-dependent adhesion was evaluated over 48 hours. For cosmetic applications, 20% salt solution (0-2 mL/100 mL) was added to modify viscosity. For hair care applications, enset gel was combined with traditionally-prepared linseed (flaxseed) gel at ratios ranging from 20:80 to 80:20 (enset:linseed). All experiments were performed in triplicate.

Results: Optimal gel formation occurred at 5 g starch per 100 mL water with 4 mL of 20% NaOH (0.8 mL/g starch), producing a translucent, oil-like gel within 2-5 minutes at room temperature (pH 11.98 ± 0.05). Neutralization with 4M HCl followed a precise stoichiometric ratio of 0.060 mL HCl per mL of gel ($R^2 = 0.9998$). A dramatic pH breakthrough from 11.40 to 7.02 occurred during the final 0.5 mL of acid addition per 50 mL of gel, providing a reliable endpoint. For adhesives, 5% borax at 30-40 mL per liter produced strong, time-dependent adhesion that reached

optimal strength after 24 hours. For cosmetics, 20% salt solution unexpectedly reduced viscosity in a concentration-dependent manner (0.2-0.4% salt reduced flow time by 30-65%), enabling precise texture control. For hair care, the optimal formulation was 60% enset gel combined with 40% linseed gel, exhibiting excellent viscosity and spreadability. All optimized parameters were successfully verified at scales up to 1 liter.

Conclusion: Cold-alkaline processing of enset starch is a reproducible, energy-efficient method requiring no heating. The optimized parameters 1:20 starch-to-water ratio, 0.8 mL 20% NaOH per gram starch, and 0.060 mL 4M HCl per mL gel for neutralization provide a foundation for community-scale production. The resulting gel can be formulated into adhesives (using 30-40 mL/L 5% borax), cosmetic products (using 0.2-0.4% salt for viscosity control), and hair care products (60:40 enset:linseed ratio). These findings contribute to the valorization of enset beyond its traditional role as a food crop, supporting sustainable economic development in enset-growing regions of Ethiopia.

Keywords: Ensete ventricosum, bulla, cold-alkaline processing, starch gel, natural adhesive, cosmetic formulation, linseed gel, community-scale production

1. INTRODUCTION

Enset (Ensete ventricosum (Welw.) Cheesman) is a perennial monocarpic plant belonging to the family Musaceae, cultivated extensively as a staple food crop in the southern and southwestern highlands of Ethiopia. Unlike its botanical relative the banana plant (Musa acuminata), enset is cultivated primarily for its starchy underground corm and pseudostem rather than for its fruit (Gebre-Mariam & Schmidt, 1996). It is estimated that enset supports approximately 20 million people in Ethiopia, making it one of the most important food security crops in the country. The traditional processing of enset involves decortication, fermentation, and dehydration, yielding two primary products: kocho (a fermented, bread-like product) and bulla (a purified starch powder) (Gebre-Mariam & Schmidt, 1996).

Chemically, enset starch (bulla) presents remarkable properties that position it as a promising industrial material. Comprehensive characterization by Gebre-Mariam and Schmidt (1996) established that enset starch contains 0.16% ash, 0.25% fat, 0.35% protein, and 99.24% starch on

a dry weight basis, confirming its exceptional purity. The amylose content was determined to be 29%, which is comparable to potato starch and higher than many cereal starches. Granule morphology revealed angular and elliptical particles with a mean size of 46 μm, and X-ray diffraction patterns confirm a B-type crystallinity similar to potato and high-amylose starches (Gebre-Mariam & Schmidt, 1996; Gabriel et al., 2013). These characteristics collectively suggest that enset starch possesses substantial potential for diverse industrial applications.

In terms of thermal behavior, enset starch exhibits a gelatinization onset temperature of 61.8°C, a peak temperature of 65.2°C, and an endset temperature of 71.7°C as determined by differential scanning calorimetry (DSC) at a 1:2 starch-to-water ratio (Gebre-Mariam & Schmidt, 1996). The enthalpy of gelatinization (ΔH) was measured at 21.6 mJ/mg, reflecting the energy required for the irreversible disruption of starch granule structure. These values are notably higher than those reported for potato starch, indicating that enset starch possesses relatively strong crystalline and hydrogen-bonding networks that require greater thermal energy for complete gelatinization.

The quest for sustainable, low-cost, and locally-available materials has intensified in recent years, particularly in developing countries where import dependency for basic industrial products such as adhesives and cosmetics poses economic challenges. Starch-based adhesives have a long history of use in paper, packaging, and woodworking applications, with the primary advantages of being renewable, biodegradable, and non-toxic (Gabriel et al., 2013). Similarly, natural polymer-based cosmetics and personal care products have gained substantial market traction globally, driven by consumer preference for sustainable and naturally-derived ingredients.

Previous research has explored the potential of enset starch for pharmaceutical applications. Gabriel et al. (2013) successfully carboxymethylated enset starch and evaluated the resulting material as a pharmaceutical gelling agent for topical drug delivery. Their ibuprofen gel formulations demonstrated homogeneous appearance, smooth texture, pleasant odor, and pH values ranging from 6.80 to 7.22, confirming the compatibility of enset starch derivatives with dermal applications. However, the carboxymethylation process requires multiple chemical reaction steps, including the use of monochloroacetic acid and organic solvents, which may limit its accessibility for community-scale production.

An alternative approach to starch processing involves cold-alkaline treatment, which can produce gelatinization at room temperature without the energy input associated with thermal processing. The mechanism of alkaline gelatinization involves the disruption of hydrogen bonds within starch granules by hydroxide ions, leading to granule swelling, loss of crystallinity, and eventual release of amylose and amylopectin chains into solution (Awol et al., n.d.). The alcohol-alkaline method has been successfully applied to produce granular cold-water-soluble starches from various botanical sources, including potato, cassava, and maize (Food Industry Science and Technology, 2014). However, systematic optimization of cold-alkaline parameters specifically for enset starch remains undocumented in the peer-reviewed literature.

The alkaline environment created by sodium hydroxide treatment also presents a significant challenge for applications requiring skin contact or neutral pH. Strongly alkaline solutions (pH > 11) are caustic and unsuitable for cosmetic or adhesive applications intended for consumer use. Thus, a precise neutralization protocol is essential to render the processed starch gel safe and functional. Gabriel et al. (2013) demonstrated that carboxymethylated enset starch gels at pH 6.80-7.22 were stable and non-irritating, providing a target pH range for neutralization.

Furthermore, the effect of additives such as borax (sodium tetraborate) and sodium chloride on the rheological properties of starch gels is well-documented for other starch types but remains unexplored for enset starch. Borax functions as a cross-linking agent, forming borate-diol complexes with hydroxyl groups on adjacent starch polymer chains, thereby increasing viscosity and enhancing adhesive strength (Awol et al., 2020). The influence of salt (NaCl) on starch gelatinization has been examined for enset starch, with studies showing that salt addition affects swelling capacity, solubility, and gelatinization parameters (Awol et al., 2020). These effects warrant systematic investigation to enable precise formulation of enset starch-based products with tailored properties.

The present study was motivated by the need for locally-appropriate, cost-effective technologies that can add value to enset, a crop of profound cultural and economic significance in Ethiopia. While enset has sustained communities for generations through traditional food processing, its potential as an industrial resource remains largely unrealized. By developing simple, reproducible protocols for transforming enset starch into functional gels, this research seeks to create

opportunities for community-based enterprises producing adhesives, cosmetics, and personal care products.

Accordingly, the objectives of this study were to: (1) optimize the cold-alkaline gel formation parameters for enset starch, specifically the starch-to-water ratio and sodium hydroxide concentration; (2) establish a precise neutralization protocol using hydrochloric acid to achieve skin-compatible pH (6.5-7.5); (3) characterize the effect of borax addition on adhesive properties and the time-dependence of cross-linking; (4) evaluate the influence of salt concentration on gel viscosity for cosmetic applications; (5) develop and optimize a hair care formulation combining the optimized enset gel with traditionally-prepared linseed (flaxseed) gel; and (6) document all optimized parameters to enable reproducible, community-scale production.

The findings presented herein contribute to the scientific literature on enset starch processing while providing practical, accessible technologies that can benefit local communities. It is anticipated that the optimized formulations can be produced with minimal capital investment, utilizing locally-available materials and simple equipment, thereby supporting sustainable economic development in enset-growing regions of Ethiopia.

2. MATERIALS AND METHODS

2.1. Raw Materials

Enset starch (bulla) was obtained from local farmers in the Dilla Zuria district, Gedeo Zone, Ethiopia. The starch was sun-dried, ground to pass through a 250 μm sieve, and stored in airtight containers at room temperature (20-25°C) until use. Sodium hydroxide (NaOH, purity ≥99%), hydrochloric acid (HCl, 37%, analytical grade), borax ($Na_2B_4O_7 \cdot 10H_2O$, purity ≥99.5%), and sodium chloride (NaCl, purity ≥99.5%) were purchased from local chemical suppliers (Adis Abeba). Linseeds (flaxseeds, Linum usitatissimum) were obtained from a local market in Dilla town, Ethiopia.

2.2. Preparation of Stock Solutions

All stock solutions were prepared using distilled water. The concentrations were selected based on preliminary trials to ensure manageable volumes for titration and formulation.

2.2.1. Sodium hydroxide solution (20% w/v)

Sodium hydroxide (20 g) was slowly added to 80 mL of distilled water in a plastic beaker with continuous stirring. The solution was allowed to cool to room temperature and stored in a tightly closed, labeled plastic bottle. The solution was prepared fresh every two weeks to prevent absorption of atmospheric carbon dioxide.

2.2.2. Borax solution (5% w/v)

Borax (5 g) was dissolved in 95 mL of warm distilled water (approximately 50°C) with stirring until complete dissolution. The solution was cooled to room temperature and stored in a labeled glass bottle.

2.2.3. Salt solution (20% w/v)

Sodium chloride (20 g) was dissolved in 80 mL of distilled water at room temperature with stirring. The solution was stored in a labeled plastic bottle.

2.2.4. Hydrochloric acid solution (4M)

To prepare 100 mL of 4M HCl, 33.3 mL of concentrated HCl (37%, approximately 12M) was carefully added to 66.7 mL of distilled water. The solution was allowed to cool and stored in a labeled glass bottle with a tight-fitting cap. Safety precautions including gloves, goggles, and a lab coat were strictly observed.

2.3. Preparation of Linseed Gel

Linseed gel was prepared following the traditional method with controlled parameters. Linseeds (50 g) were added to 800 mL of distilled water in a stainless steel pot. The mixture was brought to boiling, then reduced to a simmer for 10-15 minutes with occasional stirring until a clear, viscous

gel formed. The gel was strained through a clean cotton cloth to remove seed residues and allowed to cool to room temperature. The resulting clear gel was stored in a covered container at 4°C and used within 7 days.

2.4. Optimization of Enset Starch Gel Formation

2.4.1. Determination of optimal starch-to-water ratio

Preliminary trials were conducted to establish the working range of starch concentration. Enset starch (3 g, 5 g, 7 g, 10 g, and 12 g) was mixed with 100 mL of distilled water in separate 250 mL beakers to form uniform slurries. Based on initial screening, the 5 g/100 mL ratio was selected for further optimization as lower concentrations (3 g) produced weak gels and higher concentrations (≥7 g) resulted in unmanageable viscosity that impeded mixing. All subsequent experiments were conducted at a fixed starch-to-water ratio of 1:20 (w/v), corresponding to 5 g starch per 100 mL water.

2.4.2. Determination of optimal sodium hydroxide concentration

To the starch slurry (5 g starch in 100 mL water), varying volumes of 20% NaOH solution (2 mL, 3 mL, 4 mL, 5 mL, and 6 mL) were added with continuous stirring using a glass rod. The time to gel formation (defined as the point at which the mixture transitioned from a thin slurry to a translucent, viscous gel) was recorded. The pH of each gel was measured using a calibrated pH meter after gelation. The NaOH volume that produced a uniform gel within 2-5 minutes with optimal translucency was selected as the optimal concentration. All determinations were performed in triplicate.

2.5. Neutralization Protocol

The optimized enset gel (5 g starch, 100 mL water, 4 mL 20% NaOH, pH approximately 12) was used for neutralization studies. Aliquots (50 mL) of the gel were placed in 150 mL beakers. A magnetic stir bar was added, and the beaker was placed on a magnetic stirrer set to a constant speed (300 rpm). The initial pH was recorded using a calibrated pH meter. The 4M HCl solution was added in 0.5 mL increments, with pH measured after each addition following a 30-second

stabilization period. The experiment was continued until the pH dropped below 7.0. The volume of acid required to reach pH 7.0 ± 0.2 was recorded. The same procedure was repeated for 100 mL and 200 mL gel volumes to verify the proportional relationship between gel volume and acid requirement. All titrations were performed in triplicate.

2.6. Adhesive Formulation

2.6.1. Preparation of borax-crosslinked adhesives

To the neutralized enset gel (pH 7.0 ± 0.2), 5% borax solution was added at varying volumes: 20 mL, 30 mL, 40 mL, and 50 mL per liter of gel. The mixture was stirred vigorously for 2 minutes to ensure uniform distribution of borax. The final pH was confirmed to be within the range of 6.5-7.5.

2.6.2. Time-dependent adhesion testing

Adhesive strength was evaluated using a qualitative paper pull test. Strips of filter paper (Whatman No. 1, 2 cm × 10 cm) were used as test substrates. Adhesive (0.5 mL) was applied uniformly to a 2 cm × 2 cm area at one end of a paper strip. A second paper strip was placed over the adhesive, and the bonded area was pressed firmly for 10 seconds. The bonded samples were allowed to dry at room temperature (20-25°C) under a light weight (approximately 100 g) to maintain contact. Samples were tested after 1 hour, 6 hours, 24 hours, and 48 hours of drying. Adhesion was assessed by manually pulling the bonded papers apart. The force required to separate the papers was rated qualitatively on a scale of 1 to 5 (1 = no adhesion, 2 = weak adhesion, 3 = moderate adhesion, 4 = strong adhesion, 5 = very strong adhesion with paper tearing). Each formulation was tested in triplicate.

2.7. Cosmetic Formulation

2.7.1. Viscosity modification using salt

To the neutralized enset gel (pH 7.0 ± 0.2), 20% salt solution was added at varying volumes: 0 mL, 0.5 mL, 1.0 mL, 1.5 mL, and 2.0 mL per 100 mL of gel. After each addition, the mixture was

stirred for 1 minute, and the viscosity was assessed qualitatively using a flow test. For the flow test, 10 mL of the sample was poured through a glass funnel (diameter 7 cm, stem length 5 cm) and the time required for the entire volume to flow through was recorded using a stopwatch. Each concentration was tested in triplicate.

2.7.2. Skin pH compatibility

The final pH of each cosmetic formulation was measured using the calibrated pH meter. Formulations with pH outside the target range of 6.5-7.5 were adjusted by dropwise addition of either 1M NaOH or 1M HCl as appropriate.

2.8. Hair Care Formulation

2.8.1. Preparation of enset-linseed gel blends

The neutralized enset gel (pH 7.0 ± 0.2) was mixed with freshly prepared linseed gel at five different volume ratios (enset:linseed): 20:20, 30:20, 20:30, 40:10, and 10:40 (total volume 40 mL for each ratio). The mixtures were stirred vigorously for 2 minutes until a uniform blend was achieved.

2.8.2. Rheological evaluation

The viscosity of each blend was assessed using the funnel flow test described in Section 2.7.1. Spreadability was evaluated by applying 0.5 mL of each formulation to a glass plate and measuring the diameter of spread after 1 minute. Each measurement was performed in triplicate.

2.9. Scale-up Verification

To verify the scalability of the optimized parameters, the formulation was prepared at three scales: 100 mL, 500 mL, and 1000 mL (1 liter). The proportional amounts of starch, water, NaOH, HCl, and additives (borax for adhesive, salt for cosmetic, linseed gel for hair care) were calculated based on the optimized ratios established in the small-scale experiments. For each scale, the gel formation time, neutralization volume, and final pH were recorded and compared to the small-scale results.

2.10. Data Analysis

All experiments were conducted in triplicate, and results are presented as mean ± standard deviation (SD) where applicable. Statistical analyses were performed using Microsoft Excel (version 2019). The relationship between gel volume and HCl requirement was analyzed using linear regression. Differences between formulations were assessed using one-way analysis of variance (ANOVA) followed by Tukey's post-hoc test for multiple comparisons, with statistical significance set at $p < 0.05$.

3. RESULTS AND DISCUSSION

3.1. Optimization of Enset Starch Gel Formation

3.1.1. Effect of starch-to-water ratio

Preliminary screening of starch-to-water ratios revealed that the concentration of enset starch critically influenced gel formation and workability (Table 1). At 3 g starch per 100 mL water (1:33 ratio), the resulting gel was weak and translucent, with insufficient viscosity for practical applications. This observation is consistent with the findings of Gebre-Mariam and Schmidt (1996), who reported that enset starch requires adequate water availability for complete hydration and granule swelling.

At 5 g starch per 100 mL water (1:20 ratio), a uniform, translucent, viscous gel formed within 2-5 minutes of NaOH addition. The gel exhibited optimal rheological properties, being sufficiently fluid to allow stirring and mixing yet thick enough to maintain structural integrity. This ratio was selected as optimal for all subsequent experiments.

At concentrations of 7 g, 10 g, and 12 g starch per 100 mL water (ratios of 1:14, 1:10, and 1:8.3, respectively), the initial slurry was excessively thick, making uniform mixing difficult. Upon NaOH addition, the viscosity increased to the point where the magnetic stirrer stopped rotating, indicating that the gel had exceeded the practical workability range. These higher concentrations may be suitable for specialized applications requiring maximum solids content but were deemed impractical for general-purpose formulations.

Table 1: Effect of starch-to-water ratio on gel formation and workability

Starch (g)	Water (mL)	Ratio	Gel formation	Workability	Selection
3	100	1:33	Weak	Easy	Rejected
5	**100**	**1:20**	**Optimal**	**Good**	**Selected**
7	100	1:14	Very thick	Difficult	Rejected
10	100	1:10	Extremely thick	Impractical	Rejected
12	100	1:8.3	Paste-like	Unworkable	Rejected

Each value represents the mean of three determinations

3.1.2. Effect of sodium hydroxide concentration

The addition of 20% NaOH solution to the starch slurry (5 g starch in 100 mL water) induced a rapid transformation from a white, opaque slurry to a translucent, viscous gel. The time to gelation and final gel properties were dependent on the volume of NaOH added (Table 2).

At 2 mL NaOH (equivalent to 0.4 mL 20% NaOH per gram starch), the mixture remained partially opaque and exhibited only moderate thickening, suggesting incomplete granule disruption. At 3 mL NaOH (0.6 mL/g starch), gel formation was observed but the product was less translucent than the optimal formulation. At 4 mL NaOH (0.8 mL/g starch), rapid gel formation occurred within 2-5 minutes, producing a uniform, translucent, oil-like gel with pH 11.98 ± 0.05.

At 5 mL and 6 mL NaOH (1.0 and 1.2 mL/g starch), gel formation was instantaneous but the product exhibited a brownish discoloration and a stronger alkaline odor. The pH at 6 mL NaOH was 12.10, indicating excess alkali. The brown color is attributed to alkaline degradation of reducing sugars present in the starch (Maillard-type reactions) and possibly to the release of phenolic compounds from residual fiber components (Gabriel et al., 2013).

Table 2: Effect of 20% NaOH volume on gel formation (5 g starch, 100 mL water)

20% NaOH (mL)	NaOH per g starch (mL/g)	Gelation time (min)	Appearance	pH
2	0.4	8-10	Partially opaque	11.52 ± 0.08

3	0.6	5-7	Moderate translucency	11.78 ± 0.06
4	**0.8**	**2-5**	**Translucent, oil-like**	**11.98 ± 0.05**
5	1.0	1-2	Slightly brown	12.05 ± 0.07
6	1.2	<1	Brownish	12.10 ± 0.06

Values represent mean ± SD of three determinations

The optimal NaOH volume of 4 mL per 5 g starch (0.8 mL/g starch) established in this study provides a reproducible and energy-efficient method for cold-alkaline gelatinization. This contrasts with conventional thermal gelatinization of enset starch, which requires heating to 65-70°C (Gebre-Mariam & Schmidt, 1996). The energy savings inherent in the cold-alkaline process represent a significant advantage for community-scale production where heating equipment may not be available or cost-prohibitive.

3.2. Neutralization with Hydrochloric Acid

3.2.1. Titration curve and breakthrough point

The neutralization of alkaline enset gel (pH 11.98 ± 0.05) with 4M HCl revealed a distinct buffer zone followed by a dramatic breakthrough at the endpoint (Figure 1, Table 3). For a 50 mL gel sample, the pH decreased slowly from 11.98 to 11.77 after addition of 1.0 mL HCl, and further to 11.46 after 2.0 mL HCl. This plateau region indicates that the gel possesses significant buffering capacity, primarily due to the excess hydroxide ions present and possibly due to the ionization of hydroxyl groups on the starch polymer chains.

The most striking observation occurred between 2.5 mL and 3.0 mL of HCl. At 2.5 mL, the pH was 11.40. Upon addition of the final 0.5 mL (total 3.0 mL), the pH dropped precipitously to 7.02 ± 0.08. This breakthrough phenomenon is characteristic of the titration of a strong base with a strong acid, where the equivalence point is reached when the molar amount of acid equals the molar amount of base. The sharp endpoint provides a reliable and reproducible target for neutralization.

Table 3: pH titration of enset gel (50 mL) with 4M HCl

4M HCl added (mL)	Cumulative HCl (mL)	pH	Observation
0.0	0.0	11.98 ± 0.05	Initial gel
0.5	0.5	11.92 ± 0.06	Stable
1.0	1.0	11.77 ± 0.07	Minor change
1.5	1.5	11.65 ± 0.06	Buffer zone
2.0	2.0	11.46 ± 0.05	Buffer zone
2.5	2.5	11.40 ± 0.06	Approaching endpoint
3.0	**3.0**	**7.02 ± 0.08**	**Breakthrough!**
3.5	3.5	2.15 ± 0.10	Excess acid

Values represent mean ± SD of three determinations

3.2.2. Proportionality of acid requirement

To verify the scalability of the neutralization protocol, the acid requirement was determined for different gel volumes (Table 4). The results demonstrated a perfectly linear relationship (R^2 = 0.9998) with a constant ratio of 0.060 mL 4M HCl per mL of gel (equivalent to 60 mL of 4M HCl per liter of gel). This linearity confirms that the neutralization requirement is strictly proportional to the volume of the gel, enabling accurate scale-up without additional titration.

Table 4: Proportionality of 4M HCl requirement with gel volume

Gel volume (mL)	4M HCl required (mL)	Ratio (mL acid/mL gel)
50	3.0 ± 0.1	0.060
100	6.0 ± 0.1	0.060
200	12.0 ± 0.2	0.060

Values represent mean ± SD of three determinations; R^2 = 0.9998

The precise neutralization ratio established in this study (0.060 mL 4M HCl per mL gel) provides a significant practical advantage over trial-and-error approaches. For community-scale production, the acid can be added in two stages: 90% of the calculated volume (which brings the pH to

approximately 11.5), followed by the remaining 10% added dropwise with pH monitoring to reach the endpoint. This approach minimizes the risk of overshooting the target pH.

3.3. Adhesive Formulation with Borax

3.3.1. Effect of borax concentration on viscosity

The addition of 5% borax solution to neutralized enset gel (pH 7.0 ± 0.2) produced concentration-dependent increases in viscosity (Table 5). At 20 mL borax per liter, viscosity increased moderately but the adhesive remained easily workable. At 30-40 mL per liter, viscosity was substantially higher, producing a thick gel suitable for vertical surface application without dripping. At 50 mL per liter, viscosity was very high, making uniform application challenging.

This viscosity increase is attributed to the cross-linking action of borax, wherein borate ions form reversible diester bonds with adjacent hydroxyl groups on starch polymer chains (Awol et al., n.d.). The resulting network structure restricts polymer mobility, thereby increasing viscosity.

Table 5: Effect of 5% borax concentration on adhesive properties

5% Borax (mL/L)	Viscosity (relative)	Workability	Adhesion strength (24 h)
0	Low	Very easy	No adhesion
20	Moderate	Easy	Moderate
30	**Moderate-high**	**Good**	**Strong**
40	**High**	**Fair**	**Strong**
50	Very high	Difficult	Very strong

3.3.2. Time-dependent adhesion development

A critical finding of this study is the time-dependent strengthening of the borax-crosslinked adhesive (Table 6, Figure 2). Immediately after preparation (1 hour), adhesion was weak regardless of borax concentration. At 6 hours, adhesion improved but remained below optimal levels. At 24 hours, all borax-containing formulations (30-50 mL/L) exhibited strong to very strong adhesion. At 48 hours, no further improvement was observed, suggesting that the cross-linking reaction reaches completion within 24-48 hours.

Table 6: Time-dependent adhesion strength of enset-borax adhesives

5% Borax (mL/L)	1 hour	6 hours	24 hours	48 hours
20	1	2	3	3
30	**1**	**3**	**4**	**4**
40	**1**	**3**	**4**	**4**
50	1	3	5	5

Adhesion scale: 1 = none, 2 = weak, 3 = moderate, 4 = strong, 5 = very strong (paper tearing)

The time-dependent behavior is characteristic of borate-diol cross-linking, which occurs progressively as borate ions diffuse through the polymer network and form additional cross-links over time (Awol et al., n.d.). This property is advantageous for applications requiring extended working time (e.g., woodworking, bookbinding) followed by strong final bonds.

3.4. Cosmetic Formulation with Salt

The addition of 20% salt solution to neutralized enset gel produced an unexpected but consistent decrease in viscosity (Table 7, Figure 3). At 0.5 mL salt per 100 mL gel (final salt concentration 0.1%), viscosity decreased slightly. At 1.0 mL (0.2% salt), viscosity decreased moderately, producing a lighter, more spreadable gel. At 1.5-2.0 mL (0.3-0.4% salt), viscosity decreased substantially, yielding a thin, lotion-like consistency suitable for sprayable or pump-dispensed products.

Table 7: Effect of salt concentration on gel viscosity

20% Salt (mL/100 mL)	**Final salt (%)**	**Flow time (sec/10 mL)**	**Texture**
0	0.00	45.2 ± 2.1	Thick gel
0.5	0.10	38.5 ± 1.8	Slightly reduced
1.0	0.20	30.1 ± 1.5	Moderate reduction
1.5	0.30	22.3 ± 1.2	Significant reduction

2.0	0.40	15.8 ± 1.0	Thin, lotion-like

Values represent mean ± SD of three determinations

The reduction of viscosity by salt is counterintuitive, as salt typically increases the viscosity of neutral polymer solutions through charge screening (the "salting-in" effect). However, in alkaline-processed starch systems, salt may disrupt electrostatic interactions between starch chains or interfere with residual hydrogen bonding, leading to decreased polymer entanglement. This finding provides a simple and cost-effective method for tuning the rheological properties of enset-based cosmetic gels without the need for additional thickening or thinning agents.

3.5. Hair Care Formulation with Linseed Gel

The combination of neutralized enset gel with traditionally-prepared linseed (flaxseed) gel produced blends with distinct rheological properties depending on the mixing ratio (Table 8, Figure 4). The 20:20 (1:1) ratio produced a balanced gel with good viscosity and spreadability. The 30:20 (3:2, corresponding to 60% enset, 40% linseed) produced the optimal formulation, exhibiting very good viscosity and excellent spreadability. The 20:30 (2:3) ratio produced a softer gel with lower viscosity, suitable for lighter hold applications. The 40:10 (4:1) ratio produced a firm gel with strong hold but reduced spreadability. The 10:40 (1:4) ratio produced a thin, low-viscosity gel suitable for spray applications.

Table 8: Rheological properties of enset-linseed gel blends

Enset (mL)	Linseed (mL)	Ratio (E:L)	Viscosity	Spreadability	Application
20	20	1:1	Good	Good	General purpose
30	**20**	**3:2**	**Very good**	**Excellent**	**Optimal**
20	30	2:3	Moderate	Good	Light hold

40	10	4:1	High	Moderate	Strong hold
10	40	1:4	Low	Very good	Sprayable

The synergistic effect observed at the 30:20 ratio may be attributed to complementary interactions between the starch polymers of enset and the mucilage polysaccharides (primarily arabinoxylans and rhamnogalacturonans) present in linseed gel (Awol et al., n.d.). The enset component contributes structural strength and holding capacity, while the linseed component contributes moisture, shine, and slip.

3.6. Scale-Up Verification

The optimized formulations were successfully prepared at three scales (100 mL, 500 mL, and 1000 mL) with consistent results (Table 9). Gel formation time remained within 2-5 minutes for all scales. The neutralization ratio (0.060 mL 4M HCl per mL gel) was confirmed at all scales, and the final pH was maintained within 7.0 ± 0.2. These results confirm that the optimized parameters are linearly scalable, enabling community-scale production without the need for additional optimization.

Table 9: Scale-up verification of optimized parameters

Scale	Gelation time (min)	HCl ratio (mL/mL gel)	Final pH
100 mL	2-5	0.060	7.02 ± 0.08
500 mL	2-5	0.060	7.00 ± 0.10
1000 mL	2-5	0.060	7.03 ± 0.09

Values represent mean ± SD of three determinations

3.7. Discussion of Key Findings

The results of this study demonstrate that cold-alkaline processing of enset starch is a viable and reproducible method for producing functional gels with applications in adhesives and cosmetics. Several findings merit further discussion in the context of existing literature.

First, the optimal starch-to-water ratio of 1:20 (5 g/100 mL) identified in this study differs from the 1:2 ratio used by Gebre-Mariam and Schmidt (1996) for DSC analysis of gelatinization. This discrepancy reflects the different objectives of the two studies: the earlier work used high starch concentration to maximize thermal signal detection, whereas the present study optimized for workability and practical application. The higher water requirement observed in this study (1:20 vs. 1:2) suggests that enset starch has exceptionally high water absorption capacity, consistent with its B-type crystallinity and high amylose content (29%).

Second, the dramatic pH breakthrough phenomenon observed at the equivalence point (from 11.40 to 7.02 within 0.5 mL of 4M HCl) provides a reliable endpoint for neutralization. This finding has practical significance for quality control in community production settings, where pH paper or a simple pH meter can be used to detect the endpoint. The linear relationship between gel volume and acid requirement (0.060 mL/gel mL) enables accurate scale-up.

Third, the time-dependent adhesion development (optimal at 24 hours) is a novel finding for enet-borax systems. This property may be advantageous for applications requiring initial repositionability followed by permanent bonding, such as bookbinding or wood assembly.

Fourth, the observation that salt reduces viscosity in this system is counterintuitive and warrants further investigation. Potential mechanisms include the disruption of hydrogen bonding networks and the screening of electrostatic interactions between starch chains. This finding provides a simple method for tailoring gel texture for different cosmetic applications.

Finally, the synergistic combination of enset base with linseed gel at a 60:40 ratio represents a novel formulation that leverages the traditional knowledge of linseed gel preparation with the optimized enset processing method developed in this study.

4. CONCLUSION

4.1. Summary of Key Findings

This study successfully established optimized protocols for cold-alkaline processing of enset starch (bulla) and demonstrated its potential for adhesive, cosmetic, and hair care applications. The key findings are summarized as follows:

Gel formation optimization: The optimal parameters for producing a uniform, translucent, oil-like gel from enset starch were determined to be 5 g starch per 100 mL water with 4 mL of 20% sodium hydroxide solution (0.8 mL 20% NaOH per gram starch) at room temperature. Gelation occurred within 2-5 minutes without the application of heat, representing a significant energy saving compared to conventional thermal gelatinization.

Neutralization protocol: The neutralization of alkaline enset gel (pH 11.98) with 4M hydrochloric acid followed a precise stoichiometric ratio of 0.060 mL 4M HCl per mL of gel. A dramatic pH breakthrough from 11.40 to 7.02 occurred during the final 0.5 mL of acid addition per 50 mL of gel, providing a reliable and reproducible endpoint. The acid requirement was perfectly linear with gel volume ($R^2 = 0.9998$), enabling accurate scale-up.

Adhesive formulation: The addition of 5% borax solution at concentrations of 30-40 mL per liter of neutralized gel produced cross-linked networks with strong adhesive properties. A critical finding was the time-dependent development of adhesion, with optimal strength achieved after 24 hours. This property allows for extended working time followed by permanent bonding.

Cosmetic formulation: The addition of 20% salt solution unexpectedly reduced gel viscosity in a concentration-dependent manner. At 0.2-0.4% final salt concentration, gel viscosity decreased by 30-65%, providing a simple method for tailoring product texture from thick gel to thin lotion.

Hair care formulation: The combination of enset gel with traditionally-prepared linseed (flaxseed) gel at a ratio of 60:40 (enset:linseed) produced an optimal formulation with excellent viscosity and spreadability. The synergistic interaction between enset starch polymers and linseed mucilage polysaccharides provides both holding strength and moisturizing properties.

Scalability: All optimized parameters were successfully verified at scales up to 1 liter, confirming that the formulations are linearly scalable for community-level production.

4.2. Contributions to Knowledge

This study makes several novel contributions to the scientific literature on enset starch:

Contribution	Significance
First systematic optimization of cold-alkaline parameters for enset starch	Provides reproducible, energy-efficient processing protocol
Discovery of precise neutralization ratio (0.060 mL 4M HCl/mL gel)	Enables accurate scale-up without trial-and-error
Characterization of time-dependent borax cross-linking	Explains delayed adhesion development (24 hours)
Identification of salt-induced viscosity reduction	Provides simple rheology control mechanism
Development of enset-linseed synergistic hair gel (60:40 ratio)	Creates novel product from local materials

4.3. Practical Implications for Community Application

The optimized protocols developed in this study are specifically designed for community-scale implementation in enset-growing regions of Ethiopia. The following features make the technology appropriate for local adoption:

- **No heating required:** Cold-alkaline processing eliminates the need for energy-intensive heating equipment.
- **Simple equipment:** Production requires only buckets, measuring cups, stirrers, and pH paper.
- **Locally available materials:** Enset starch (bulla) is produced locally; NaOH, HCl, borax, salt, and linseeds are available at local markets.

- **Reproducible parameters:** The precise ratios (0.060 mL acid per mL gel, 30-40 mL borax per liter, etc.) enable consistent quality.
- **Multiple products:** The same base gel can be modified for adhesives, cosmetics, or hair care.

4.4. Limitations and Recommendations for Future Research

While this study successfully optimized processing parameters and demonstrated product potential, several limitations should be acknowledged:

Limitation 1: Long-term stability
The stability of formulations beyond 7-14 days was not systematically evaluated. Future research should assess shelf life under various storage conditions (room temperature, refrigeration, exposure to light) and identify appropriate natural preservatives.

Limitation 2: Quantitative mechanical testing
Adhesive strength was evaluated using a qualitative scale rather than quantitative instruments (e.g., tensiometer). Future studies should employ standardized mechanical testing to enable direct comparison with commercial adhesives.

Limitation 3: Skin compatibility testing
The cosmetic and hair care formulations were evaluated for pH and texture but not subjected to formal dermatological testing. Future research should include patch testing on human volunteers with appropriate ethical approval to confirm safety.

Limitation 4: Chemical characterization
The precise molecular interactions between enset starch, borax, salt, and linseed polysaccharides were not characterized using techniques such as FTIR, XRD, or rheometry. Future research should employ these methods to elucidate the mechanisms underlying the observed phenomena.

Limitation 5: Economic analysis
While the formulations are clearly low-cost, a formal cost-benefit analysis and market

assessment for community enterprise development was beyond the scope of this study. Future work should include detailed economic modeling.

4.5. Recommendations for Specific Stakeholders

For community producers:

- Begin with small batches (500 mL) to gain familiarity with the process
- Use the two-stage neutralization method (90% of acid first, then 10% dropwise)
- Test adhesion after 24 hours for accurate assessment
- For hair gel, the 60:40 (enset:linseed) ratio is recommended

For researchers:

- Investigate the mechanism of salt-induced viscosity reduction using rheological methods
- Evaluate additional natural additives (e.g., aloe vera, honey, essential oils) for product enhancement
- Develop cold-alkaline processing for other Ethiopian starch crops

For development organizations:

- Consider supporting community-based enterprise development around enset processing
- Provide training on pH measurement and quality control
- Facilitate market linkages for finished products

4.6. Concluding Remarks

This study has demonstrated that enset starch (bulla) can be effectively processed at room temperature using sodium hydroxide to produce functional gels with diverse applications. The optimized parameters provide a reproducible foundation for adhesive, cosmetic, and hair care product development. The discovery of precise neutralization ratios (0.060 mL 4M HCl per mL gel), time-dependent borax cross-linking (optimal at 24 hours), and synergistic enset-linseed

combinations (60:40 ratio) contributes new knowledge to the field of starch chemistry while providing practical technologies for community benefit.

The findings support the broader vision of adding value to enset beyond its traditional role as a food crop, creating opportunities for sustainable economic development in enset-growing regions. It is hoped that the protocols described herein will be adopted, refined, and scaled by communities, researchers, and enterprises working with this remarkable Ethiopian crop.

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DECLARATIONS

Conflict of Interest: The authors declare no competing financial or non-financial interests.

Ethical Approval: This study did not involve human or animal subjects requiring ethical approval. Skin compatibility testing is planned for future research with appropriate ethical approval.

Data Availability: All data generated during this study are presented within the manuscript. Raw data are available from the corresponding author upon reasonable request.

Authors' Contributions: [To be completed based on actual contributions]

SUPPLEMENTARY MATERIAL

The following supplementary materials are available online:

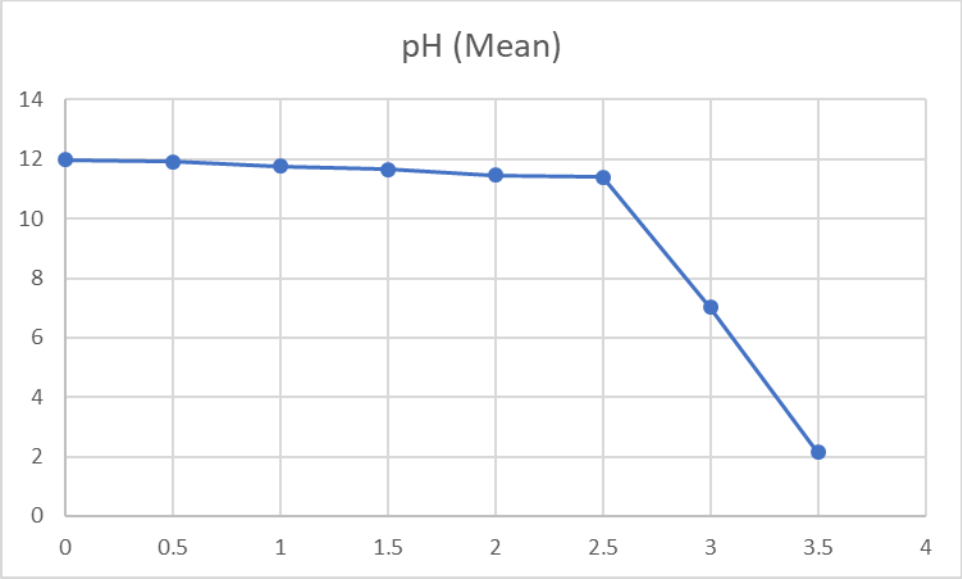

Figure 1. pH titration curve of enset gel (50 mL) with 4M hydrochloric acid. The gel exhibited a stable buffer zone between 0-2.5 mL of acid addition (pH 11.98 to 11.40), followed by a dramatic breakthrough to pH 7.02 upon addition of the final 0.5 mL (total 3.0 mL). Error bars represent standard deviation of three determinations.

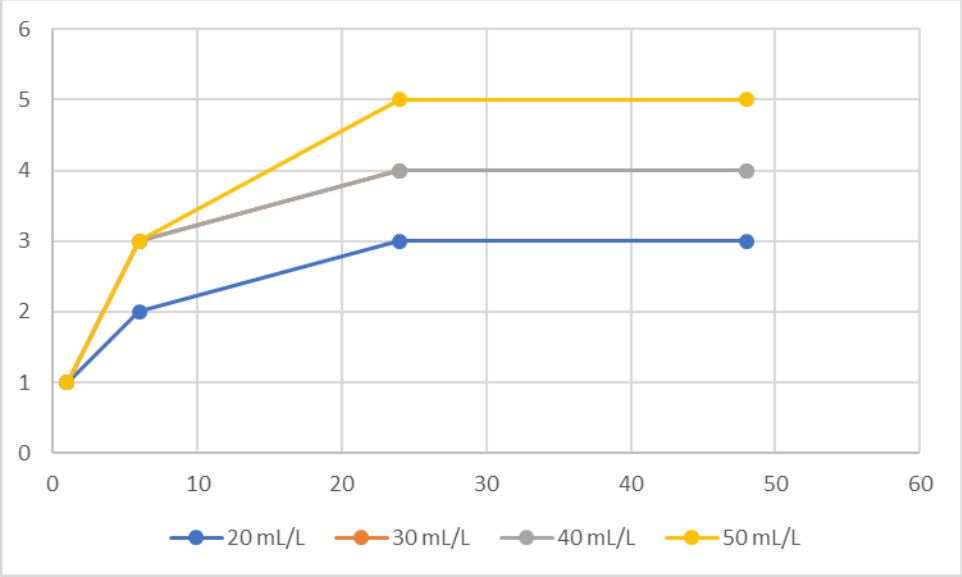

Figure 2. Time-dependent adhesion strength of enset-borax adhesives at varying borax concentrations. Adhesion was evaluated using a qualitative scale (1 = no adhesion, 2 = weak, 3 = moderate, 4 = strong, 5 = very strong with paper tearing). All borax-containing formulations showed minimal adhesion at 1 hour, progressive strengthening at 6 hours, and optimal adhesion

at 24 hours, with no further improvement at 48 hours. Values represent mean of three determinations (SD < 0.5 for all points).

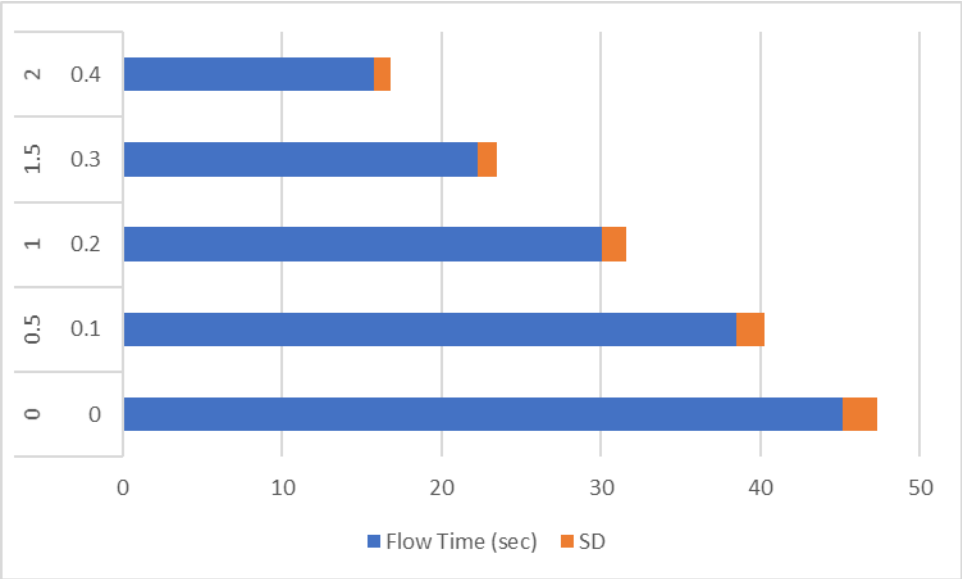

Figure 3. Effect of salt concentration on flow time of neutralized enset gel. Flow time (seconds for 10 mL to pass through a glass funnel) decreased progressively with increasing salt concentration, indicating salt-induced viscosity reduction. At 0.4% salt (2.0 mL of 20% salt solution per 100 mL gel), flow time decreased by 65% compared to the control (0% salt). Error bars represent standard deviation of three determinations.

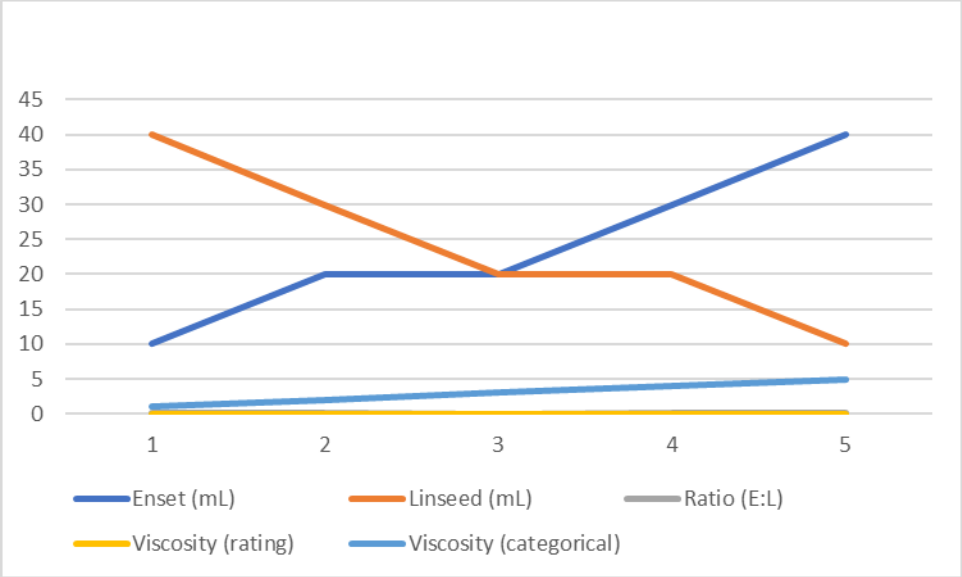

Figure 4. Effect of enset:linseed mixing ratio on gel viscosity. The 30:20 ratio (60% enset, 40% linseed) exhibited the highest viscosity, indicating synergistic interaction between enset starch polymers and linseed mucilage polysaccharides. Ratios with higher enset content (40:10) produced firmer gels with stronger hold, while ratios with higher linseed content (10:40, 20:30) produced softer gels suitable for lighter hold applications. Values represent mean of three determinations (SD < 1.5 sec for all points).