Original Research Article

Fragmentation of a Corn Starch-Glycerol-Calcium Alginate Bioplastic Composite in a 35 g/L NaCl solution: An Exploratory Experimental Study Across Three Aqueous Environments

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ABSTRACT

Marine plastic pollution represents a pressing global environmental problem, with conventional and most commercial bioplastics persisting in marine environments for decades without any meaningful degradation. Development of materials that fragment in high-salt aqueous conditions while remaining structurally stable in low-salt conditions is still largely unexplored in sustainable materials design. This study presents an exploratory experimental investigation of corn starch-glycerol-calcium alginate composite bioplastic, made from renewable and food-safe materials. The composite was formulated through iterative development and tested for structural stability in three aqueous environments: distilled water, municipal tap water and 35 g/L NaCl solution within 72 h. Results demonstrate that the composite retained full macroscopic structural integrity in distilled and municipal water while undergoing progressive fragmentation into discrete pieces of approximately 2-3 mm in 35 g/L solution (reached VDI score 3 by 72 h). It is hypothesized, based on prior literature rather than measurements made in this study, that this differential response may involve Na^{+}-mediated disruption of Ca^{2+} crosslinks. These findings demonstrate that polysaccharide composites prepared from renewable, food-safe components can exhibit markedly different fragmentation behavior in a 35 g/L NaCl solution compared with distilled and municipal tap water. Quantitative characterisation and comparative testing across multiple alginate-to-starch ratios are identified as priorities for future investigation.

Keywords: Calcium alginate; corn starch; NaCl-induced fragmentation; ionic crosslinking; aqueous-responsive bioplastic; polysaccharide composite

INTRODUCTION

Plastic waste, particularly in marine environments, remains a persistent global challenge due to the long degradation times and environmental stability of conventional polymers. According to the United Nations Environment Program, plastics constitute about 85% of total marine litter, and about 11 million metric tons of plastic enter the oceans annually. This figure is projected to triple by 2040, which intensifies the urgency of finding viable alternatives to conventional plastics (1).

Although bioplastics are often seen as a viable solution to this problem, their degradation behavior in marine environments is often inadequate. PLA (polylactic acid), Polybutylene Succinate (PBS) and other commercially

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Accepted August 20, 2026
https://doi.org/10.70251/HYJR2348.4412751282

available bioplastics are observed to require specific conditions like elevated temperatures, significant UV exposure, and microbial activity to degrade at meaningful levels (2). In marine environments however, these conditions are likely to be absent or inconsistent. This means that the degradation time periods of these bioplastics may be similar or comparable to conventional plastics.

This problem gives rise to the demand for a predictable material, whose environmental degradation can easily be triggered within marine conditions (3).

Prior literature reports that alginate chains crosslinked by Ca^{2+} ions form stable egg-box junction zones and that monovalent Na^{+} ions, being unable to form equivalent bridging structures, may competitively displace Ca^{2+} when present at sufficiently high concentrations (4, 5). On this basis it has been proposed that at NaCl concentrations approaching 35 g/L, such displacement may be sufficient to drive fragmentation without elevated temperatures or enzymatic activity. This pathway is treated throughout the present study as a mechanistic hypothesis drawn from prior work rather than as a mechanism established here.

Previous studies have quantitatively observed this selective disintegration and reported a sevenfold increase in Ca^{2+} ions release from crosslinked films in the 35 g/L NaCl solution compared to distilled water (6). However, their experiment only used pure dehydrated sodium alginate films, isolated from any secondary polymer matrix. The behavior of ionic selectivity with a secondary polymer matrix is still largely unexplored. This study addresses this gap by investigating whether the fragmentation behavior previously reported for pure alginate films is also observed in a corn starch-glycerol-calcium alginate composite formulated from renewable, food-safe materials. Unlike prior work, wherein alginate is isolated as a sole matrix polymer, this study investigates the interaction between a hygroscopic starch phase and an ionically crosslinked network. This study also gives special focus to the role of citric acid as a dual-function crosslinking agent. It is hypothesized that the corn starch-glycerol-calcium alginate composite will exhibit differential macroscopic fragmentation behavior across aqueous environments of varying ionic strength retaining macroscopic integrity in distilled and municipal tap water while undergoing progressive fragmentation in a 35 g/L NaCl solution within 72 h, with the proposed involvement of Na^{+} mediated disruption of Ca^{2+} crosslinks treated as a hypothesis drawn from prior literature rather than tested directly in this study.

MATERIALS AND METHODS

Materials

All materials used in this study were food-safe, commodity-grade and commercially available. The final composite formulation consisted of the following components (Table 1).

Table 1. *Final composite formulation components, quantities (per 150 mL of distilled water), and functional roles.*

Component	Quantity	Function
Corn Starch	7.50 g	Primary thermoplastic polymer matrix
Glycerol	30% of starch mass (2.25 g)	Plasticiser; reported to increase polymer chain mobility in starch films (7)
Citric Acid	0.30 g	Crosslinking agent; enhances starch cohesion and reduces water uptake
Sodium Alginate	2.00 g	Providing ionic crosslinking capacity
Calcium Chloride	2.00 g	Supplies Ca^{2+} for in situ alginate crosslinking
Distilled Water	150 mL	Solvent phase

Bioplastic Synthesis

Synthesis was conducted at the Central Research Facility, Indian Institute of Technology Delhi (IIT Delhi), with institutional support provided through the IIT Delhi Changemakers Bootcamp.

Dry components (corn starch and citric acid) were weighed and mixed until homogeneous. Sodium alginate was dissolved in a portion of the distilled water to form a viscous alginate solution, to which calcium chloride was added and mixed to initiate ionic crosslinking, forming calcium alginate in situ. Glycerol and the remaining distilled water were then incorporated to complete the liquid phase. The dry mixture was gradually added to this liquid phase under continuous manual stirring (approximately two revolutions per second) to prevent agglomeration. The resulting suspension was heated on a laboratory hot plate at 90°C, as monitored using a laboratory thermometer, until a viscous gel-like

consistency was reached, indicating polymer network formation. The hot material was immediately transferred to molds for casting.

Casting and Drying

The viscous material was poured into flat molds of approximate internal dimensions of 25 × 15 cm to a uniform thickness of approximately 3-5 mm as assessed by visual inspection during pouring. Air bubbles were minimized through controlled pouring and gentle leveling. Cast samples were dried in an oven at 50-60 °C for 6-8 h. Drying was continued until solidification was uniform and residual moisture was reduced to a level assessed by the absence of surface moisture and dimensional stability upon handling, as formal gravimetric moisture measurement was not conducted. Samples were cooled to room temperature (25°C) prior to removal and handling.

Iterative Formulation Development

Formation development proceeded through non-systematic iterative testing of candidate additives. The additives tested and the resulting decisions are summarized in Table 2.

The formulation development process described here represents preliminary exploratory development rather than controlled systematic optimization. Complete formulation compositions, tested concentrations, and replicate numbers were not recorded for each iteration, and several variables were adjusted simultaneously across batches. As a result, the independent effects of individual components - including gelatin removal, citric acid concentration, glycerol ratio, and calcium alginate quantity - cannot be determined from the available data. The exclusion of gelatin and the adjustment of citric acid concentration was associated with improved film cohesion and the emergence of ionic-strength-dependent fragmentation behavior in the final formulation; however, these observations are correlational rather than causal. Gelatin's association with indiscriminate water absorption across all conditions is consistent with prior literature on protein-based components in alginate-containing composite systems (8), but was not isolated experimentally in the present study.

Environmental Stability Testing

Three aqueous test environments were prepared: distilled water, municipal tap water, and the 35 g/L NaCl solution. For each condition, one film sample was immersed in 200 mL of test solution in a glass beaker. Samples were maintained at room temperature (25°C) without agitation throughout the observation period. Solution pH was not measured during this study; this represents a parameter for standardization in future work. Initial sample mass was not recorded prior to immersion and gravimetric analysis was therefore not possible in this study. One film specimen was prepared per batch and divided into three portions, one test per solution. Portions were cut manually using scissors without the use of a template or cutting guide; the dimensions, shape, thickness, and mass of individual portions were therefore not standardized or recorded. All three conditions were tested concurrently within the same 72 h observation window and samples were observed at 24 h intervals. Five independent preparation batches were conducted, with each batch treated as the experimental unit. A total of fifteen sample portions were assessed across all conditions and batches (five per condition). Observations at each time point were made by the same observer using the defined VDI below (Table 3).

Table 2. *Summary of preliminary formulation development decisions. Observations are qualitative and non-systematic iterative testing; independent effects of individual components cannot be determined.*

Component tested	Observed effect	Decision
Gelatin	Excessive water absorption; poor dimensional stability (8)	Excluded
Acetic Acid	Weakened film structure	Excluded
Neem-based additive	Interfered with uniform and consistent film formation	Excluded
Citric Acid	Associated with enhanced cohesion and reduced water uptake relative to earlier formulations; effect not isolated experimentally	Retained
Glycerol	Associated with reduced brittleness and improved flexibility, consistent with prior reports of glycerol plasticization in starch films (7); effect not isolated experimentally.	Retained

RESULTS

Film Formation and Characteristics

The final formulation produced films of consistent surface texture and flexibility. Films exhibited a translucent appearance with a slightly yellow tint. Film thickness was about 3-5 mm across all samples. Films were dimensionally stable at room temperature, with no spontaneous cracking or deformation, observed prior to the immersion testing. Prior to immersion, all samples were also assigned a baseline VDI score of 0, confirming structural integrity before environmental testing. Figure 1 presents representative photographs of the composite film prior to immersion testing. Images were taken under non-standardized lighting and background conditions; no scale bar was available for these preliminary photographs. Standardised photographic documentation with scale bars at each VDI score stage represents a priority for future work.

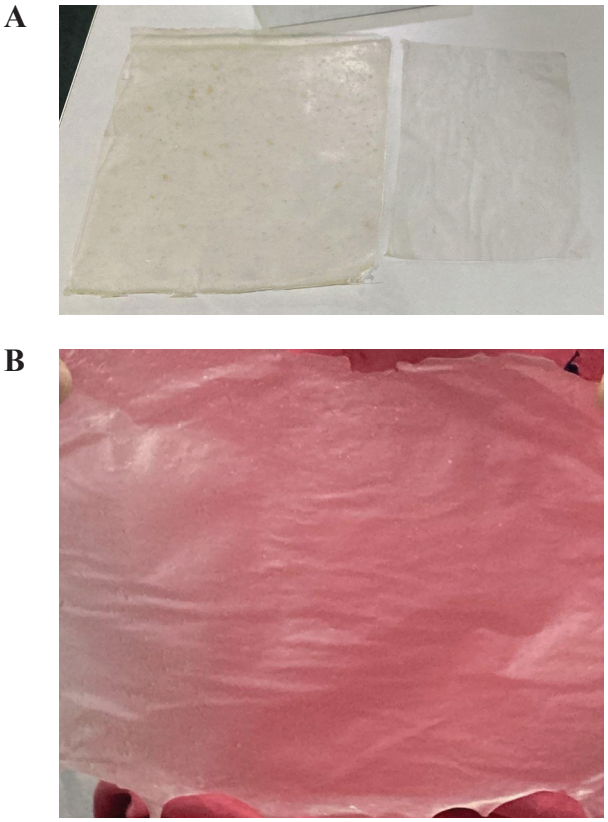

Figure 1. *(A) Corn starch-glycerol-calcium alginate composite bioplastic films following casting and drying (left). (B) Cast films demonstrating uniform surface texture and translucency; (right) film demonstrating flexibility and structural integrity upon handling.*

Environmental Stability Observations

Table 3. *In the absence of gravimetric instrumentation, a four-point Visual Disintegration Index (VDI) was developed to standardize the observation recording.*

Score	Description
0	No visible change; film intact and rigid
1	Surface softening observed; film pliable but structurally continuous
2	Partial fragmentation; visible cracks or separation at edges
3	Complete fragmentation into discrete pieces; no continuous film remaining

Table 4. *Qualitative structural observations of corn starch-glycerol-calcium alginate composite films following complete immersion in three aqueous environments over a 72-hour period. VDI scores represent consistent observations across five independent preparation batches. No variation in score was recorded between batches at any time point.*

Condition	24 h	48 h	72 h
Distilled water	VDI 0 - Film intact. No visible surface change	VDI 0 - Film intact, no visible surface change	VDI 0 - Full structure retained
Municipal tap water	VDI 0 - Film intact. No visible surface change	VDI 0 - film intact, no visible surface change	VDI 0 - Full structure retained
35 g/L NaCl solution	VDI 1 - The surface softened. The film lost rigidity and became pliable.	VDI 2 - Progressive fragmentation initiated	VDI 3 - Progressive fragmentation into discrete pieces of approximately 2-3 mm; no continuous film remaining.

Formulation Development Outcomes

Iterative formulation development identified gelatin as the additive most strongly associated with indiscriminate swelling across all three aqueous environments (Table 2). Formulations containing gelatin exhibited swelling and structural failure across all three aqueous environments,

with no differential fragmentation observed. Gelatin removal and citric acid adjustment were among the formulation changes associated with the emergence of ionic-strength-dependent fragmentation behavior in the 35 g/L NaCl solution across all five preparation batches.

DISCUSSION

The fragmentation of the composite film in 35 g/L NaCl solution, while maintaining a VDI score of 0 in distilled and municipal tap water throughout the 72 h observation period, is consistent with the mechanistic hypothesis outlined above. It is proposed, consistent with prior literature on alginate ionic crosslinking, that Na^+ ions at elevated concentrations may competitively displace Ca^{2+} ions from egg-box junction zones, progressively weakening the crosslinked network and enabling fragmentation (4, 5); however, this mechanism was not directly tested in the present study and would require calcium-release assays, ion analysis, or spectroscopic characterisation to confirm. The proposed mechanism is illustrated schematically in Figure 2.

This mechanistic hypothesis draws on prior quantitative work in which pure dehydrated sodium alginate films crosslinked with Ca^{2+} demonstrated a sevenfold increase in calcium ion release in equivalent NaCl concentrations compared to distilled water (6). However, several important material differences exist between that work and the present study that limit direct mechanistic comparison. The prior study used pure dehydrated sodium alginate as the sole matrix polymer, whereas the present composite incorporates corn starch, glycerol, citric acid, and calcium alginate formed in situ from sodium alginate and calcium chloride, resulting in a substantially more complex polymer network. Additionally, the present films were considerably thicker (approximately 3-5 mm compared to dehydrated films), and processing conditions differed significantly. These differences may alter crosslink density, network architecture, ion diffusion pathways, and fragmentation behavior in ways that are not captured by the present qualitative observations. The fragmentation behavior observed in this study is therefore described as consistent with, rather than confirmatory of, the ionic displacement mechanism characterized in pure alginate systems; direct mechanistic comparison would require equivalent characterisation methods including calcium release assays and spectroscopic analysis.

The persistence, dissolution, or further fragmentation of the 2-3 mm pieces observed at 72 h was not monitored beyond the observation window. Whether these fragments subsequently dissolve or accumulate as secondary particles cannot be determined from the present data. This distinction is environmentally significant - fragmentation without subsequent biodegradation or mineralization may represent a secondary pollution concern rather than a beneficial outcome, and future studies should explicitly assess fragment persistence,

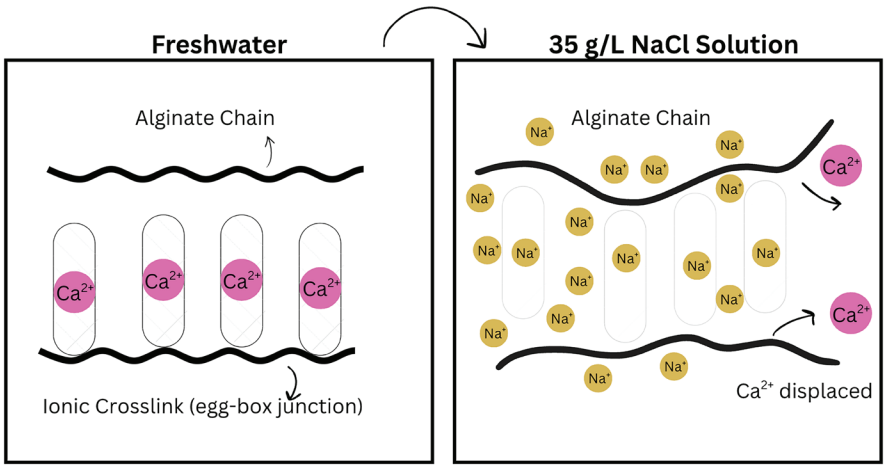

Figure 2. *Schematic representation of the proposed Na^+ mediated fragmentation mechanism, presented as a working hypothesis consistent with prior literature on alginate ionic crosslinking; this mechanism was not directly confirmed by the present experimental data. In freshwater (left), Ca^{2+} ions bridge neighbouring alginate chains at egg-box junction zones, maintaining network integrity. In the 35 g/L NaCl solution (right), excess Na^+ ions competitively displace Ca^{2+} from junction zones, disrupting crosslinks and initiating selective structural disintegration. Schematic created by the author.*

biodegradation, and ecotoxicity before environmental claims are made.

Furthermore, the equivalent structural stability observed in municipal tap water and distilled water suggests that fragmentation depends on NaCl concentration rather than simply on the presence of water. Published values indicate that Municipal tap water typically contains dissolved ionic species at concentrations several orders of magnitude below 35 g/L NaCl (9); The composition of the municipal tap water used in the study was not characterized, and this comparison is therefore based on general literature values rather than direct measurement. At such low concentrations, competitive Na^+ displacement of Ca^{2+} crosslinks would not be expected to proceed at a rate sufficient to produce fragmentation within a 72 h window. However, long-term storage stability, behavior under varying humidity, and response to real marine ionic environments were not assessed and cannot be inferred from the present data.

The hygroscopic nature of starch (due to its abundant hydroxyl (-OH) groups readily forming hydrogen bonds with water molecules) introduces a competing water uptake pathway in composite systems like these, and must be managed through iterative formulations. Early iterations containing gelatin exhibited swelling across all three water environments, consistent with previous studies about the water absorption characteristics of protein-based components in alginate containing systems and showed no selective response (8). Gelatin removal and citric acid adjustment were among the formulation changes associated with the emergence of NaCl-induced fragmentation behavior; however, as multiple variables were modified simultaneously across iterations, no causal attribution to any single component is possible from the present data. Citric acid crosslinks starch chains through esterification of hydroxyl groups, a reaction shown to reduce non-specific water uptake and improve film cohesion (10). No citric-acid-free control was prepared in this study, so this mechanism is not evidenced by the present observations. These observations are consistent with the interpretation that the hygroscopic behavior of non-alginate phases may be a contributing factor in preserving NaCl-induced fragmentation behavior in composite systems, though the causal relationship was not isolated experimentally.

Several limitations of this study should be noted. First, all observations were recorded using a predefined Visual Disintegration Index rather than quantitative instrumentation; mass loss, water uptake, swelling ratio, fragment-size distribution, dimensional change, and mechanical properties were not measured, precluding direct quantitative comparison with prior literature values. As observations were recorded using a visual index rather than quantitative instrumentation, formal statistical analysis of variability across replicates was not possible; future quantitative studies should incorporate replicate-level measurements with appropriate statistical treatment. Second, the iterative formulation development process was not conducted as a controlled experiment; complete formulation compositions, tested concentrations, and replicate numbers were not recorded for each iteration, and as multiple variables were modified simultaneously across batches, the independent contribution of any single component cannot be determined from the available data.

Third, the ionic displacement mechanism proposed to explain the observed fragmentation behavior was not directly tested; calcium release, sodium uptake, crosslink density, and chemical changes in the polymer network were not measured, and confirmation of this mechanistic pathway would require calcium-release assays, ion exchange analysis, or spectroscopic characterisation such as FTIR. Fourth, environmental stability testing employed a single-component 35 g/L NaCl solution rather than natural or artificial seawater; the influence of additional ionic species present in real marine environments - including Mg^{2+}, K^+, sulfate, and bicarbonate - on composite fragmentation behavior remains untested, and the study therefore cannot establish saline selectivity or a sodium-specific mechanism. Ionic strength was not measured in any test condition, and the composition of the municipal tap water used was not characterized; the three conditions are therefore distinguished by NaCl addition rather than by measured ionic strength. Fifth, testing was conducted at room temperature under static immersion conditions, which may not fully replicate the thermal and hydrodynamic variability of real marine environments.

Sixth, solution pH and initial sample mass were not recorded, precluding calculation of the sample-to-solution ratio and limiting reproducibility of the experimental conditions. Seventh, standardized before-and-after photographs with scale bars were not obtained; photographic evidence is therefore limited to pre-immersion film documentation, and visual validation of VDI score assignments at each time point was not possible. Eighth, this study documents physical fragmentation only; biodegradation, mineralization, ecotoxicity, and environmental persistence of fragments were not assessed and cannot be inferred from the

present observations - fragmentation into 2-3 mm pieces may represent a secondary pollution concern rather than an environmentally beneficial outcome unless subsequent biodegradation is demonstrated. Ninth, the formulation development process described here represents preliminary exploratory development; the crosslinking role and concentration optimization of citric acid were not isolated experimentally as no citric-acid-free control or systematic concentration series was tested.

Tenth, specimen preparation was not standardized. Film portions were cut manually without a template, and the dimensions, shape, thickness, and mass of individual portions were not recorded. Variation in specimen geometry between portions may have influenced softening and fragmentation rates, and this represents a source of uncontrolled variability in the observation reported.

Future studies should prioritize quantitative mass loss measurement at systematic time intervals across all three aqueous conditions, as well as comparative testing across multiple alginate-to-starch ratios to establish the relationship between composition and fragmentation behavior. Mechanical characterisation including tensile strength and elongation at break, before and after immersion, should also be conducted. Investigation of a PHBV(Poly(3-hydroxybutyrate-co-3-hydroxyvalerate))-backbone formulation to assess thermal processability for potential extrusion-based applications represents a further direction. Testing across multiple NaCl concentrations, artificial seawater formulations, and alternative ionic controls would determine whether fragmentation is ionic-strength-dependent broadly or Na^+-specific. Calcium-release assays and spectroscopic characterisation such as FTIR are needed to confirm or refute the proposed ionic displacement mechanism. Standardised photographic documentation with scale bars at each VDI score stage should be incorporated in future observation protocols. Finally, assessment of fragment persistence, biodegradation, mineralization, and ecotoxicity is essential before any environmental safety claims can be made regarding this material system.

CONCLUSION

This study demonstrates that the corn starch-glycerol-calcium alginate composite undergoes fragmentation in a 35 g/L NaCl solution within 72 h while retaining macroscopic integrity in distilled and municipal tap water. The proposed involvement of Na^+-mediated displacement of Ca^{2+} crosslinks remains a hypothesis drawn from prior literature and was not tested in this study. The observed fragmentation behavior is consistent with, though does not confirm, the operation of this mechanism in a multi-component starch composite system.

These findings suggest that markedly different fragmentation behavior between high-salt and low-salt aqueous conditions is achievable using simple composite formulations with accessible, renewable, and food-safe feedstock. Formulation decisions, particularly the removal of hygroscopic additives and the optimization of citric acid concentrations, were found to be critical determinants of the emergence of fragmentation behavior observed in the 35 g/L NaCl solution.

It should be noted that the physical fragmentation documented in this study - specifically the formation of approximately 2-3 mm discrete pieces in a 35 g/L NaCl solution does not constitute evidence of biodegradation, mineralization, or environmental safety. Fragment persistence, ecotoxicity, and behavior in natural marine environments with complex ionic composition were not assessed. These represent essential parameters for future investigation before any environmental claims can be made regarding this material system. Moreover, quantitative characterization of mass loss kinetics, comparative multi-ratio formulation testing and investigation of PHBV backbone materials should represent the priorities of advancing this work towards a practically applicable salt-responsive material system.

ACKNOWLEDGEMENTS

The author gratefully acknowledges the Central Research Facility, Indian Institute of Technology Delhi (IIT Delhi), for access to laboratory facilities and infrastructure. Institutional support provided through the IIT Delhi Changemakers Bootcamp is also acknowledged, which enabled material procurement and experimental development.

CONFLICT OF INTEREST

The author declares no conflicts of interest related to this work.

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