

The Salty Science of Brinicle Formation: Impact of Salt Composition on Brinicle Growth and Morphology

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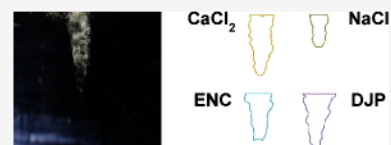


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ABSTRACT: Brinicles are hollow ice structures that grow under sea ice. These structures form when supercooled brine is released into the surrounding ocean, causing ice to form around it. These structures have recently garnered interest for their possible existence on the ocean moons of Enceladus and Europa. This work investigated the impacts of specific salt compositions on brinicle growth and morphology. Brinicles were grown in a subzero facility for 2 h with four different brine solutions: sodium chloride (NaCl), calcium chloride (CaCl_2), Enceladus analog containing NaCl (henceforth labeled as "ENC"), and a Don Juan Pond analog containing CaCl_2 (henceforth labeled as "DJP") utilizing elemental chemistry data from Don Juan Pond (Wright Valley, South Fork, Victoria Land, Antarctica). The brinicles were imaged during growth and extracted for analysis of ice grain size and inner column dimensions. This research concludes that varying salt compositions had a significant impact on the growth, stability, and morphology of brinicle formation. The CaCl_2 -based solutions resulted in brinicles that were more diverse and structurally complex than those grown in NaCl-based solutions, potentially driven by differences in heat capacities and solute properties. This investigation highlights how salt composition influences the physical characteristics of the brinicle, impacting the microenvironments in and around the brinicle and thus affecting their potential habitability in both terrestrial and extraterrestrial frozen environments. The salinity and rapid formation of brinicles would allow for the capture and entombment of halophilic microorganisms. While the stability of organics and biogenic products is not explored in this study, this investigation sets the stage for where and why these frozen water features should be a priority target for future astrobiological and geomicrobiological studies.



INTRODUCTION

Brinicles are tubular formations extending from the underside of sea ice. These structures have been observed in the Arctic and Antarctic oceans.^{1–3} Brinicles form when concentrated brine is released from sea ice at ~15% salinity.³ The supercooled dense brines flow downward, while the seawater freezes around it, resulting in the development of hollow ice tubular structures.^{1,4} Brinicles have been recorded at varying lengths from centimeters to meters, with some reaching down to the ocean floor (Figure 1a,b).^{1–4} These structures originally gained scientific interest due to their hypothesized role in transferring heat and salt throughout the ocean and ice shelf, potentially impacting microenvironments.^{3,5,6}

Brinicles were first recorded in Antarctica during the 1970s.^{1,2} During this time, brinicles were referred to as "ice stalactites" under the same category as icicles.^{1,2} However, recent literature has argued that brinicles have a different growth mechanism than icicles and thus should be referred to under the separate name of brinicles.⁶ The formation processes observed in this investigation would also support the design of brinicles as their own entities.

The first research related to brinicles focused on their formation, structure, and behavior. The earliest studies of these structures consisted of field observations from Antarctica, which laid the groundwork for the field.^{1–5} In 1974, Martin

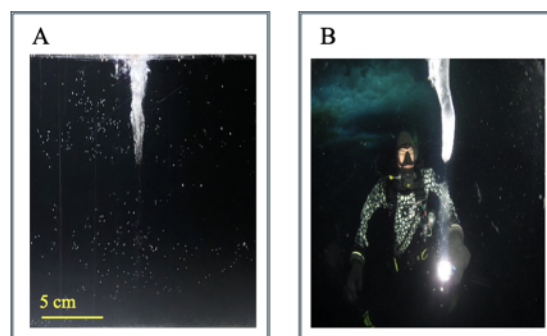


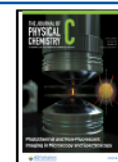
Figure 1. Images of brinicles grown in the laboratory versus field. (A) Brinicle grown in a subzero facility. (B) Brinicle imaged near McMurdo Station in Antarctica. Image courtesy of Rob Robbins, US Antarctic Program.

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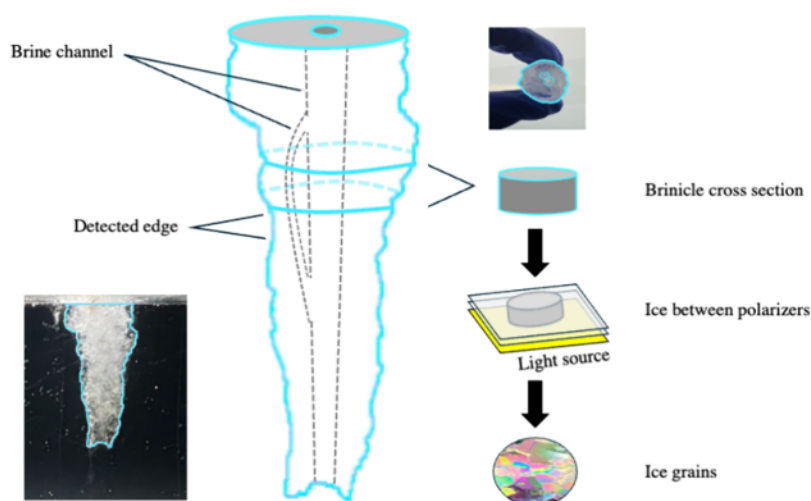


Figure 2. Experimental setup for brinicle formation and analysis. Schematic showing the process of extracting brinicles for ice grain and brine column analysis. Detected edges are outlined in blue. Note the inclusion of multiple brine channels in the drawing on the left and in the image on the top right.

Following the 2 h growth period, a hand saw was used to cut and section around the brinicle, allowing for its careful extraction from the tank. The dimensions of the length and base circumference were measured. Following this, five brinicle structures were formed for each solution, with each brinicle used for a different type of measurement. One brinicle was melted down and the conductivity was measured, one was used for grain size measurements, and three were used for internal column area measurements. For the brinicles used for grain size and column area measurements, the brinicle was sectioned at 25%, 50%, and 75% of its max length (see Table 2 for total

brine channel measurements, cross sections were taken at 0%, 25%, 50%, and 75% of their maximum length (Figure 2).

For postprocessing of the data, a threshold was applied to the images, and edge detection was performed in MATLAB based on the color difference. From these edges, the width at any given length and the height were recorded for each time point.⁷ This was used to analyze the width as a function of both time and height.

By assessing the brinicle's deviation from a linear best fit line, the roughness was quantified. A MATLAB polyfit function was used to establish the best-fit line along the edge of the brinicle. Subsequently, we determined the root-mean-squared value of the horizontal distance between this line and the brinicle's detected edge.⁷ This process was repeated along the entire length for both the right and left edges, providing a numerical representation of the "roughness" for each brinicle formed.⁷

The thin section images were imported to ImageJ for further analysis.²⁰ The images were calibrated using a tape measure to the known scale within the image. The ice grain boundaries were manually outlined, and their areas were measured using ImageJ. The average area of the ice grains for the triplicate measurements was then recorded.

The four cross-section images taken at 0%, 25%, 50%, and 75% of the brinicle's length were utilized to measure both the brine channel area and the total brinicle area. These images were also processed by using ImageJ software. The internal area of the brine channel was manually outlined and measured alongside the edge of the brine channel.

In this study, statistical analyses were conducted to assess the impacts of salt composition on the physical parameters of brinicles. One-way analysis of variance (ANOVA) tests were performed for maximum length, maximum width, average area, and root-mean-square error (RMSE) for both the left and right sides of the brinicle, subsequently termed combined RMSE.

RESULTS

Visual analysis of the detected edges across treatments showed distinct differences in the growth rate, width, and shape. The NaCl treatment brinicles displayed a more constant width along the length of the brinicle. Conversely, the CaCl_2 and DJP

Table 2. Experimental Results of Brinicle Formation^a

composition	time (min)	max length (cm)	max width (cm)	area (cm ²)	RMSE combined (cm)
CaCl_2 200 PPT	30	6.61	2.38	4.03	0.39
CaCl_2 200 PPT	120	8.92	4.15	8.83	0.65
DJP 200 PPT	30	6.63	2.83	4.23	0.47
DJP 200 PPT	120	8.47	4.08	8.21	0.62
Enceladus 200 PPT	30	2.04	2.87	3.6	0.41
Enceladus 200 PPT	120	7.57	3.86	6.71	0.54
NaCl 200 PPT	30	3.92	2.09	2.56	0.37
NaCl 200 PPT	120	6.67	2.7	4.27	0.41

^aRecorded values were averaged across five treatments. RMSE = root mean square error and is the value for roughness. 200 PPT = 200 parts per thousand.

brinicle lengths). For grain size measurements, a cross section was taken out at ~50% of the maximum length for cross-polarized light imaging (Figure 2). Sectioned pieces were attached to a glass slide and microtomed to less than 0.5 mm for grain size imaging.^{20,21} The thin sections were then backlit between two polarizers for grain measurements.^{21,22} The polarizers were adjusted until the grains appeared bright against a dark background, revealing their boundaries. For

treatments exhibited alternating thick and thin regions (Figure 3). To better visualize this, we plotted the width as a function

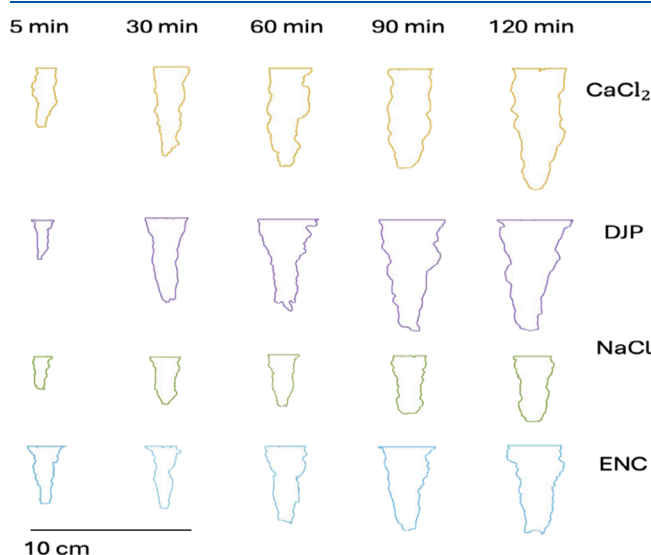


Figure 3. Edge detection between treatments over time. The detected edges of brinicle treatment groups plotted at different time intervals from 5 to 120 min. Note the 10 cm scale bar with respect to each individual brinicle.

of height and time (Figure 4). Figure 4 shows that the CaCl_2 and DJP treatments had the overall widest dimensions and were the most consistently wide across the length of the entire brinicle. The observed banding of colors indicates roughness along the brinicle's length.

CaCl_2 and DJP brinicles exhibited the fastest rates of growth within the full 120 min time scale (see Figures 4 and 5). The data were fit to the square root of time (Figure 5). Within the first five min, similar growth rates were observed between ENC and NaCl solutions (Figure 5b). However, NaCl further separated from all treatments under the full experimental time scale (Figure 5a). For all treatments, an accelerated growth rate was noted during the first 30 min. Good agreement was seen with the fit after 120 min, with CaCl_2 , DJP, NaCl, and ENC having R -squared values of 0.74, 0.75, 0.74, and 0.75, respectively (Figure 5a). While lower R -squared values were observed within the first 5 min of growth, there was still good agreement with the predicted growth for the treatments CaCl_2 , DJP, and NaCl with R -squared values of 0.66, 0.63, and 0.66 respectively.

Figure 6 shows the vertical profiles of the cross-sectional area of the brine channel and the total brinicle. For all treatment groups, the brine channels shifted locations, changed shape, and changed size. For both CaCl_2 and NaCl treatments, the channels also split into multiple channels. For all treatments, there was a trend of the channel size increasing with depth (Figure 6). CaCl_2 had the largest average brine channel area at 0.97 cm^2 , but NaCl had the lowest average area

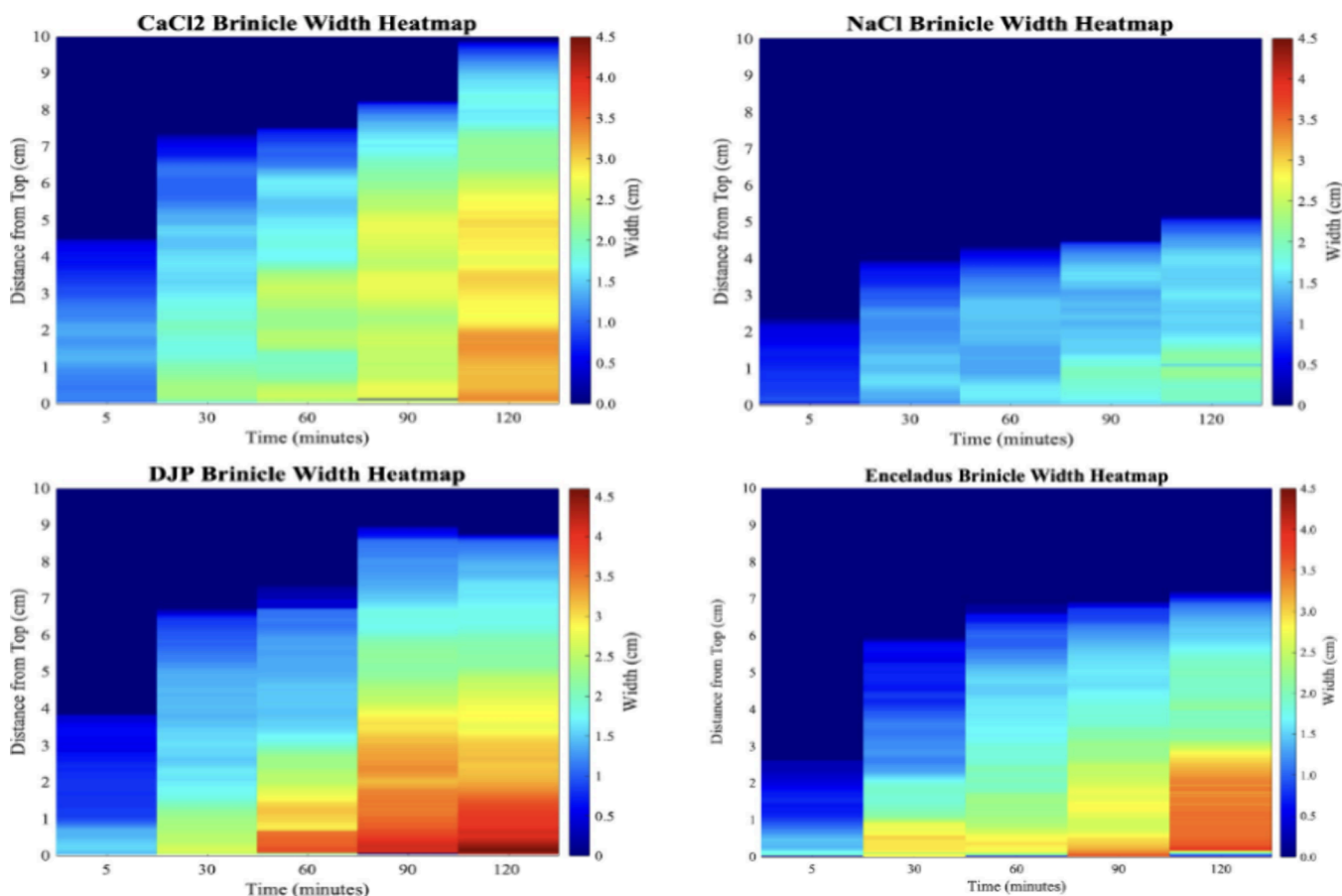


Figure 4. Temporal evolution of brinicle width and length across treatments. Heatmaps showing the effect of salt composition on brinicle width variations at selected distances from the top at each time point.

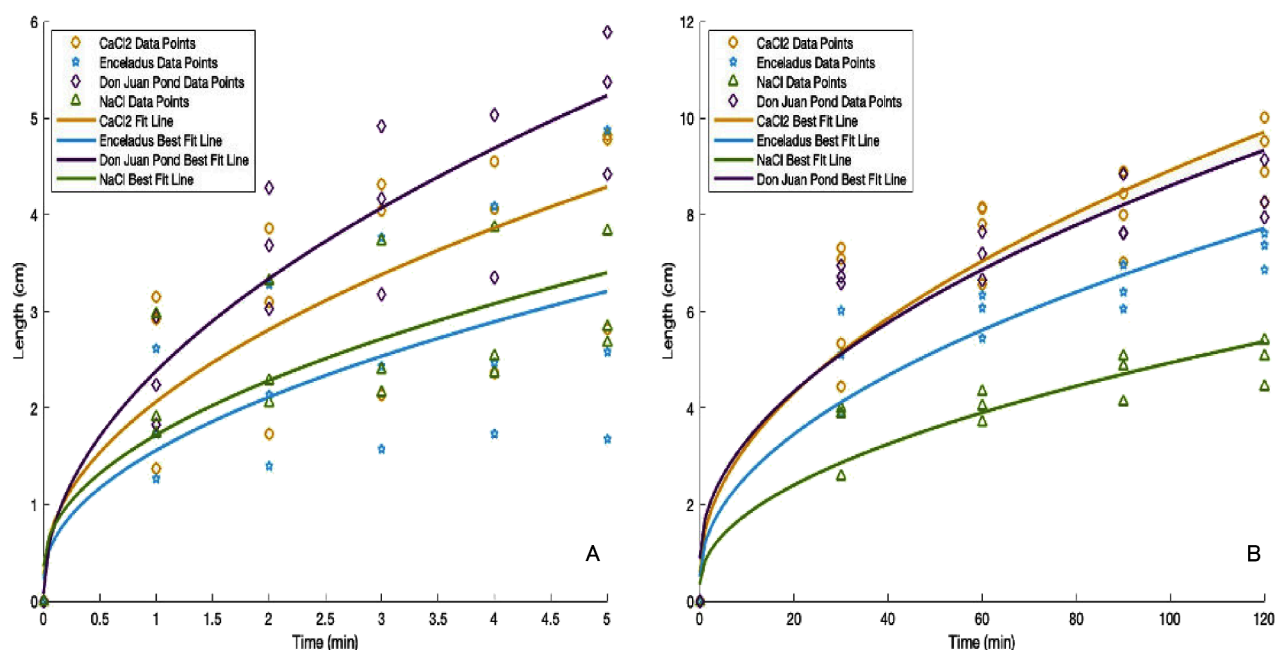


Figure 5. Brinicle growth analysis. (A) Brinicle growth over the full experimental time scale (120 min). (B) Brinicle growth during the first 5 min. All points were fitted to the predicted growth of the square root of time.¹

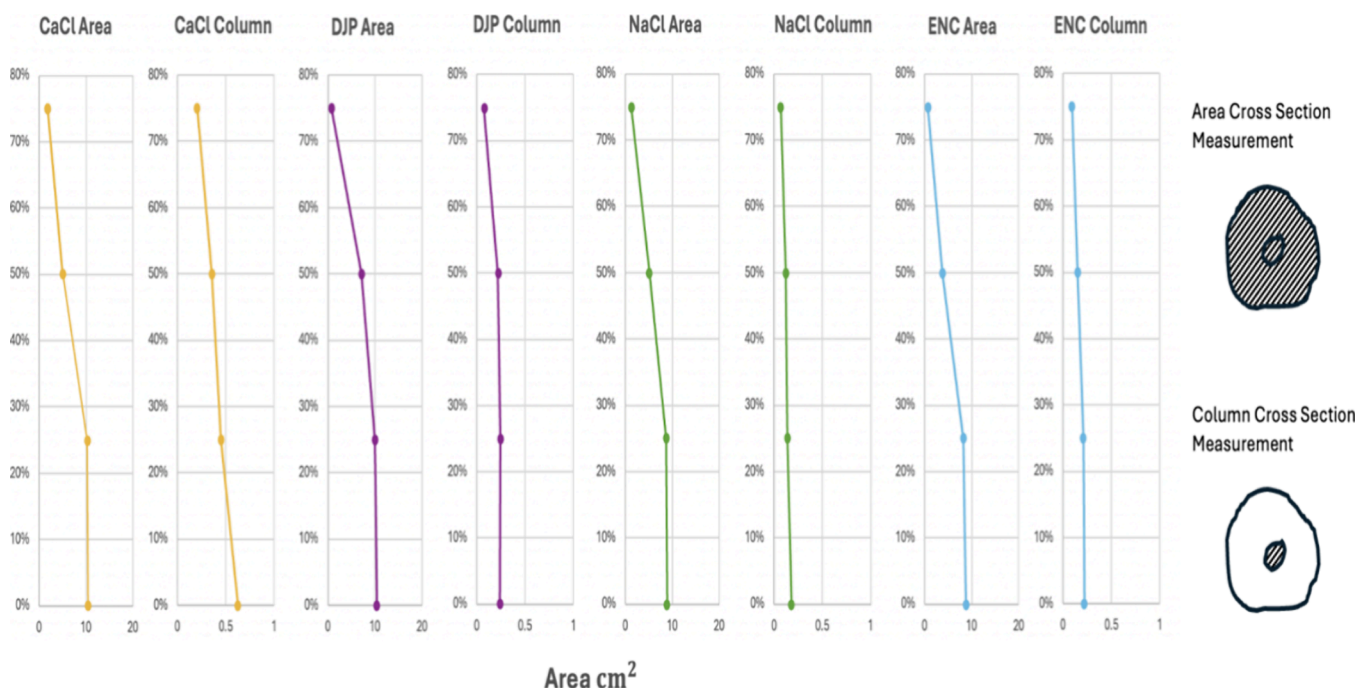


Figure 6. Comparison of cross-sectional and inner brine column areas across treatments. Cross-sectional measurements along the length of brinicles were taken for four different treatment groups: CaCl_2 , DJP, NaCl, and ENC. Cross sections were taken at four positions from 0% (the base at the ice sheet) to 75% (the brinicle tip). At each point, two measurements were taken: (1) the internal brine column area and (2) the total cross-section area.

of 0.51 cm^2 . DJP and ENC brinicles had average brine channel areas of 0.84 and 0.56 cm^2 , respectively. For average brinicle thin section area, CaCl_2 , DJP, ENC, and NaCl had values of 10.21 , 6.01 , 6.86 , and 5.44 cm^2 . For all treatment groups, the ice grains appeared to be made up of frazil ice with random smaller crystals and less defined grain boundaries (Figure S1) as compared to columnar ice.^{21,22} The CaCl_2 and DJP treatments had the smallest and least defined grain boundaries with an average grain size of 29.35 and 26.84 mm^2 ,

respectively. ENC had a comparable average grain size at 29.61 mm^2 . NaCl had the largest average grain size at 33.68 mm^2 .

The analysis of brinicles found that the composition significantly impacted growth at 30 and 120 min, while no significant difference was seen at 5 min. The P -values observed at 5, 30, and 120 were 0.12 , 0.00 , and 0.00 , respectively. At 30 min, DJP 200 PPT and CaCl_2 200 PPT showed the highest mean values in length at 6.63 and 6.61 cm , respectively, and

were not significantly different from each other. Enceladus 200 PPT had the lowest mean of 2.04 cm and was significantly different from all other treatment groups. The NaCl 200 PPT treatment value was intermediate at 3.92 cm, significantly different from all treatments. By 120 min, CaCl_2 200 PPT demonstrated a clearer separation as the highest group with a mean of 8.92 cm, with NaCl 200 PPT being the lowest at 6.67 cm, with both groups being significantly different from all other treatment groups. DJP 200 PPT and Enceladus 200 PPT showed intermediate values at 8.47 and 7.57 cm, with each only being significantly different from NaCl and CaCl_2 , respectively. These results found that the effects of different compositions on brinicle growth become more pronounced over time, with CaCl_2 200 PPT showing a significantly higher mean at 120 min.

The analysis of brinicle width under different brine compositions at 5, 30, and 120 min indicates potential effects over time. At 5 and 30 min, there was no significant impact of composition on brinicle width, with all compositions showing similar means. However, by 120 min, composition becomes a significant factor influencing growth, as indicated by a significant ANOVA result (P -value = 0.04). Pairwise comparisons using Tukey's post hoc test did not reveal significant differences between individual groups, although the largest difference was observed between NaCl and CaCl_2 brines, with average means of 2.7 and 4.14 cm, respectively. While brine composition may play a role in influencing brinicle width over time, the variability within groups limits the ability to discern separation between individual treatments.

The comparison of brinicle area at 5, 30, and 120 min reveals an important compositional influence on area. In the first 5 min of growth, statistical analyses using an ANOVA found that composition significantly impacted area (P -value = 0.04). However, pairwise tests found no significant differences among individual groups. At 30 min, brine composition showed no significant effect on area (P -value = 0.21), with all compositions demonstrating similar areas. At 120 min, composition significantly affected brinicle growth (P -value = 0.00), with DJP 200 PPT and CaCl_2 200 PPT exhibiting significantly higher means at 8.83 and 8.21 cm compared to NaCl 200 PPT at 4.27 cm. The Enceladus 200 PPT treatment mean area was 6.71 cm and was not significantly different from that of any other treatment group.

The comparison between RMSE combined at 120 and 30 min reveals a contrast in the influence of composition on prediction accuracy in brinicle growth modeling. At 30 min, there was no significant effect of composition on RMSE combined (P -value = 0.21). After 120 min, the composition significantly affected RMSE combined (P -value = 0.00), indicating distinct variations in prediction accuracy among compositions, with CaCl_2 200 PPT and DJP 200 PPT showing higher RMSE combined values at 8.83 and 8.21 cm, respectively, compared to NaCl 200 PPT at 4.27 cm. The Enceladus 200 PPT treatment was not significantly different from any treatment, with a mean RSME value of 6.71 cm.

DISCUSSION

Despite current and past interest in brinicles, analog brinicle growth experiments have posed significant challenge for researchers.^{1,6,7} Maintaining longer growth cycles and stable structures seen in environmental samples has not yet been documented in the literature. This work expands on past experimental studies by developing a methodology for brinicle

growth that allows for the long-term study of these structures and extraction capabilities in diverse salt solutions. This methodology development has allowed for further analysis of growth, stability, and morphological analyses of these structures. This also allows for the ideal geochemical endmembers (not including DJP or ENC) to be integrated as a function of time, showing depth and roughness. These salinity endmembers help bound brinicle topography and volume for in situ comparison in terrestrial extreme environments. Moreover, being able to apply our terrestrial ionic endmembers allows us to categorize mineral/ice environments in the future where similar processes would likely yield these formations, should they be observed in situ in frozen ocean worlds.

In this study, we investigated the growth rate of brinicles formed under various salt solutions, examining their evolution over short (5 min) and longer (120 min) times scales (Figure 5). This research found that brinicles grown in CaCl_2 -based solutions (i.e., CaCl_2 and DJP) exhibited faster growth rates than those grown in NaCl. This was most pronounced at the longer time scales where both 30- and 120 min time scales showed significant difference in length between treatments. Interestingly, within the initial 5 min, ENC and NaCl solutions displayed similar growth rates, suggesting comparable initial conditions despite their divergent growth trajectories over the full experimental duration. In all time scales and treatments, initial rapid growth occurred and gradually stabilized. This is concurrent with theoretical models that show that brinicle growth is proportional to the square root of time. As the brinicle grows, it takes longer for the brine solution to reach the tip. As the brine receives heat from its surroundings, the temperature of the brine solution increases as it travels. This makes further freezing and brinicle growth more difficult the longer it becomes.^{1,6} The R -squared values obtained for the square root fit underscore the robustness of this model in predicting brinicle growth across different temporal scales.

The observed differences in growth rates may be partially attributed to differences in the brine solution densities.^{23,24} The higher density of CaCl_2 and DJP solutions may allow the brine solutions to sink more quickly. This could allow the colder denser brine to reach the tip of the brinicle faster, allowing for continual growth.¹ Conversely, NaCl and ENC solutions, with their lower densities, may sink more slowly, leading to less efficient transport of the brine to the tip of the brinicle (Table S1). This slower descent could result in slower overall growth rates for brinicles in NaCl solutions compared to those in CaCl_2 solutions.

The visual examination of the four brinicle treatments revealed distinct growth patterns, width variations, and structural features that may be influenced by both brine and heat flux dynamics. NaCl-treated brinicles maintained a uniform width along their length, suggesting a stable growth pattern within the experimental conditions. In contrast, brinicles treated with CaCl_2 exhibit notable variations in width, alternating between thick and thin regions (Figure 3). This was observed in past experimental research where it was hypothesized that this change forms from instability.⁷ In addition, we noted that the CaCl_2 and DJP treatments appeared to have a larger width and area than NaCl. This variability is further highlighted in Figure 4.

The identification of changing roughness along the outer edge of brinicles, observed through RMSE and width measurements, underscores the complexity of their formation.

Posthoc Tukey tests highlighted notable differences in roughness, with RMSE values for NaCl showing lower values compared to CaCl_2 and DJP. This change in roughness may indicate growth instabilities influenced by fluctuating brine flux and heat flux dynamics, although not directly measured here. Heat flux is known to affect brinicle growth by influencing brine flow patterns, contributing to observed irregularities in growth patterns and structural irregularities.^{1,7}

Comparable to previous research on an extracted brinicle from Antarctica, our laboratory-grown brinicles inner channels did not grow as one singular channel along the middle of the brinicle.⁵ Instead, the inner channels would change size and positions and, at times, split into multiple channels (Figure 2). For all treatment groups, there was a trend of increasing size with depth (Figure 6). Differences in the size, shape, and the number of brine channels along with the presence of frazil ice crystals imply that the fluid flow with the brinicle experienced convective instability.³ While further studies should be conducted on grain size, preliminary results found smaller grain sizes in CaCl_2 and DJP grown brinicles. This could be attributed to the faster growth rates of ice in these CaCl_2 and DJP-dominated salt treatment brinicles.²⁵

The specific heat capacity of the individual solutions may partially contribute to the observed growth patterns and structural variations influenced by brine and heat flux dynamics. CaCl_2 -based solutions, with their lower specific heat capacity, may facilitate faster heat transfer within the brinicle structure (Table S1).²⁴ This accelerated heat transfer can lead to more rapid adjustments in temperature and brine concentration flux within the brinicle.¹ This may potentially contribute to the observed structural irregularities such as the alternating thick and thin regions observed in CaCl_2 -treated brinicles. Conversely, NaCl solutions, with higher specific heat capacities, exhibit slower heat transfer rates, which may result in more stable and uniform growth under similar experimental conditions.^{23,26} As a result, this change in brine chemistry could influence the brinicle growth and morphological features. While not studied here, future work should assess these dynamics to determine the habitability and preservation potential of these systems.

This work demonstrates the ability for brinicle formation in diverse salt solutions relevant to both on and off Earth. The possible growth of these structures on Europa has been modeled, finding that ice shell thickness impacted the predicted longevity of brinicles on Europa.^{13,14} Additionally, it has been hypothesized that brinicles could form on Enceladus.⁷ However, the different gravitational conditions on icy moons may affect the growth of brinicles. Past work has found that the reduced gravity on Europa could lead to slower brinicle growth rate due to decreases in buoyancy-driven flow.⁹

This research found that salt composition may impact heat and brine flux within the brinicle, ultimately impacting potential habitability. The supercooled brine within the brinicles remains in a liquid state, possibly providing a stable and nutrient-rich habitat for microorganisms near or in the brinicle.^{6,8} Past research has found that organisms can exist in similar cryobrine systems.^{10–12} However, the presence of freeze thaw cycles and chaotropic brines has been found to stress organisms and impact survivability.^{27–30} Despite this, past research has found that the microbial survival rate is improved in subzero Cl^- brines compared to warmer temperatures. This work found that the Cl^- brines also enhanced survivability during freeze thaw cycles.²⁸ These past

studies, combined with these investigations' insights into brinicle dynamics, suggest that variations in salt composition and temperature could create unique localized habitats within or around brinicle structures. These environments could create isolated niches in which liquid water and nutrients are available for life. As astrobiologists investigate icy worlds, brinicles may provide habitable environments at ocean–ice interfaces for chemical reactions and the presence of microbial life. Understanding the dynamics underlying brinicle formation may lead to better predictions of where these microenvironments are most likely to be found.

Further research should explore the long-term growth dynamics of brinicles. Specifically, studies should examine brinicle growth and stability under different environmental conditions, such as varying temperatures, pressures, currents, and brine compositions, to simulate terrestrial and extraterrestrial environments. Additionally, efforts should be made to measure the internal heat and brine flux within the brinicles, providing a more comprehensive understanding of factors influencing their development and initial formation. Future work should also explore the potential habitability of brinicles within and around these structures under simulated terrestrial and extraterrestrial conditions. By integrating these approaches, future research can enhance our understanding of brinicle formation and stability and their implications for the search for cellular life beyond Earth.

CONCLUSIONS

This research advances our understanding of brinicle formation by developing a robust methodology for their growth under extended controlled laboratory conditions, providing a foundational methodology for further research into the long-term growth under terrestrial and extraterrestrial environmental conditions of brinicles. This study's findings highlight significant differences in growth rates and structural features of brinicles formed in various salt solutions, particularly noting the faster growth facilitated by calcium chloride-based solutions compared to sodium chloride. In addition, differences in width, roughness, and internal brine column area suggest differences in brine and heat flux between the salt treatments. These observations underscore the need for considering salt compositions' influence on brine dynamics and what factors it may play in the stability and potential habitability of brinicles. Insights gained from this research provide critical and novel information for both terrestrial and solar system ocean–ice environments, suggesting that brinicles can form in a diverse range of salt compositions.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.jpcc.5c02403>.

Images of ice grains from each of the four treatments and treatment density and specific heat capacity (PDF)

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<https://pubs.acs.org/10.1021/acs.jpcc.5c02403>

Author Contributions

M.G.G. and C.M.F. conceived the project and secured the funding. M.G.G., S.M.P., and C.M.F. coordinated research efforts. M.G.G. performed experiments and data analysis. M.G.G., S.M.P., and C.M.F. wrote the paper with input from all authors.

Notes

The authors declare no competing financial interest.

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