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Freeze-thaw stability and rheological properties of starch-based gluten-free bread dough

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Abstract

Frozen dough technology has transformed commercial bakeries, yet gluten-free formulations remain notoriously unstable when exposed to repeated freezing and thawing. This research compared the freeze-thaw stability and rheological behaviour of three starch-based gluten-free bread doughs made from rice starch, potato starch, and corn starch. Dough samples were subjected to five consecutive freeze-thaw cycles (-18°C for 24 h followed by thawing at 30°C for 3 h). Syneresis, storage modulus (G'), loss modulus (G''), and $\tan \delta$ were measured after each cycle. Potato starch dough exhibited the lowest syneresis throughout all five cycles (21.3% at cycle 5 compared to 27.6% for rice and 35.1% for corn). Dynamic oscillatory tests showed that potato starch maintained higher G' values ($1,680 \pm 150$ Pa after three cycles), pointing to greater gel network integrity. Correlation analysis indicated a strong negative relationship between syneresis and G' across all starch types ($r = -0.89$, $p < 0.01$). The findings support the use of potato starch as the preferred base for frozen gluten-free bread dough systems.

Keywords: Freeze-thaw stability, rheology, starch, gluten-free bread, dough properties, syneresis, storage modulus, potato starch, rice starch, retrogradation

Introduction

Gluten-free baking is one of the most technically demanding challenges in cereal science, and freezing that dough only makes the problem harder. The global gluten-free food market was valued at approximately 5.6 billion USD in 2020, driven by rising coeliac disease diagnoses and growing consumer interest in wheat alternatives ^[1]. Yet most gluten-free breads suffer from rapid staling, crumbly texture, and poor volume defects that worsen dramatically when the dough is frozen and later thawed for baking ^[2].

Starch retrogradation is the central mechanism behind freeze-thaw instability. During freezing, water migrates out of the starch gel network and forms ice crystals that disrupt the matrix. On thawing, the expelled water doesn't fully re-absorb, resulting in syneresis—the visible weeping of liquid from the gel surface ^[3]. Repeated cycles amplify this damage, progressively weakening the dough structure and reducing its gas-holding capacity during proofing ^[4]. Different botanical sources of starch differ in their amylose-to-amylopectin ratio, granule morphology, and pasting behaviour, all of which influence retrogradation kinetics ^[5]. Rice, potato, and corn starches are the three most commonly used bases in commercial gluten-free bread formulations ^[6]. Prior work by Ziobro *et al.* ^[7] and Witczak *et al.* ^[8] examined textural changes in gluten-free loaves after frozen storage, but direct comparisons of dough-stage rheological responses across multiple freeze-thaw cycles remain scarce. This research aimed to fill that gap by measuring syneresis and dynamic viscoelastic properties of three starch-based doughs through five freeze-thaw cycles under controlled conditions.

Material and Methods

Reagents and material

Rice starch (12.3% moisture, 22% amylose) was obtained from Sigma-Aldrich (St. Louis, MO, USA, catalogue no. S7260). Potato starch (18.1% moisture, 21% amylose) was sourced from Bob's Red Mill (Milwaukie, OR, USA). Corn starch (11.8% moisture, 27% amylose) was purchased from Ingredion Inc. (Westchester, IL, USA). Xanthan gum, used as a structural binder, was food-grade and supplied by TIC Gums (White Marsh, MD, USA).

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Instant dry yeast (*Saccharomyces cerevisiae*), salt, sugar, and sunflower oil were procured from a local grocery outlet in Reno, Nevada. Distilled water was used for all dough preparations. The entire experimental programme ran from January to June 2023 at the Baking Technology Laboratory of the Great Basin Institute of Food Science.

Methods

Dough preparation: Each formulation was prepared by mixing 100 g starch, 2 g xanthan gum, 3 g yeast, 2 g salt, 5 g sugar, 5 g sunflower oil, and 80 mL distilled water in a planetary mixer (KitchenAid Professional 600) at speed 2 for 4 minutes. The doughs were portioned into 50 g pieces, placed in sealed polyethylene bags, and subjected to five consecutive freeze-thaw cycles. Each cycle consisted of freezing at -18°C for 24 hours followed by thawing at 30°C for 3 hours [9].

Syneresis measurement: A 10 g aliquot of each dough was placed in a pre-weighed centrifuge tube and centrifuged at 3,000 rpm for 20 minutes after each thawing step. The supernatant was decanted and weighed, and syneresis was

expressed as a percentage of the initial gel weight [3]. Three replicates were measured per starch type per cycle.

Rheological analysis: Dynamic oscillatory measurements were carried out on a controlled-stress rheometer (TA Instruments Discovery HR-2) fitted with a 40 mm parallel plate geometry and a 1 mm gap. Frequency sweeps were conducted from 0.1 to 10 Hz at 25°C within the linear viscoelastic region, determined by prior amplitude sweeps at 1 Hz. Storage modulus (G'), loss modulus (G''), and loss tangent ($\tan \delta = G''/G'$) were recorded at 1 Hz for comparison across cycles [10].

Quality control

The rheometer was calibrated using a silicone standard (Brookfield, 12,500 cP at 25°C) before each measurement session. Freezer temperature was monitored continuously with a data logger (Onset HOBO UX100) and remained within $\pm 0.5^{\circ}\text{C}$ of the set point. All measurements were performed in triplicate, and mean values with standard deviations are reported. Outliers beyond two standard deviations from the mean were excluded and repeated.

Results

Table 1: Syneresis (%) of starch doughs before and after freeze-thaw cycles

Starch type	Cycle 0 (Before)	Cycle 1	Cycle 3	Cycle 5 (After)	Change (%)
Rice starch	2.1 ± 0.3	8.7 ± 0.6	19.8 ± 1.2	27.6 ± 1.5	+25.5
Potato starch	1.8 ± 0.2	6.2 ± 0.5	15.4 ± 0.9	21.3 ± 1.1	+19.5
Corn starch	2.4 ± 0.4	10.1 ± 0.8	24.6 ± 1.4	35.1 ± 1.8	+32.7

Table 2: Pearson correlation matrix among measured dough parameters (pooled data, $n = 45$)

Parameter	Syneresis	G'	G''	$\tan \delta$
Syneresis	1.00	-0.89^{**}	-0.72^{**}	0.64^{**}
G'	-0.89^{**}	1.00	0.91^{**}	-0.58^{**}
G''	-0.72^{**}	0.91^{**}	1.00	-0.41^{*}
$\tan \delta$	0.64^{**}	-0.58^{**}	-0.41^{*}	1.00

** $p < 0.01$; * $p < 0.05$

Across all three starch types, syneresis rose steadily with each freeze-thaw cycle, while both viscoelastic moduli declined. Corn starch dough showed the steepest deterioration, reaching 35.1% syneresis by cycle 5 roughly

65% higher than potato starch at the same stage. The strong inverse correlation between syneresis and G' ($r = -0.89$) confirms that water loss from the gel is closely tied to structural weakening.

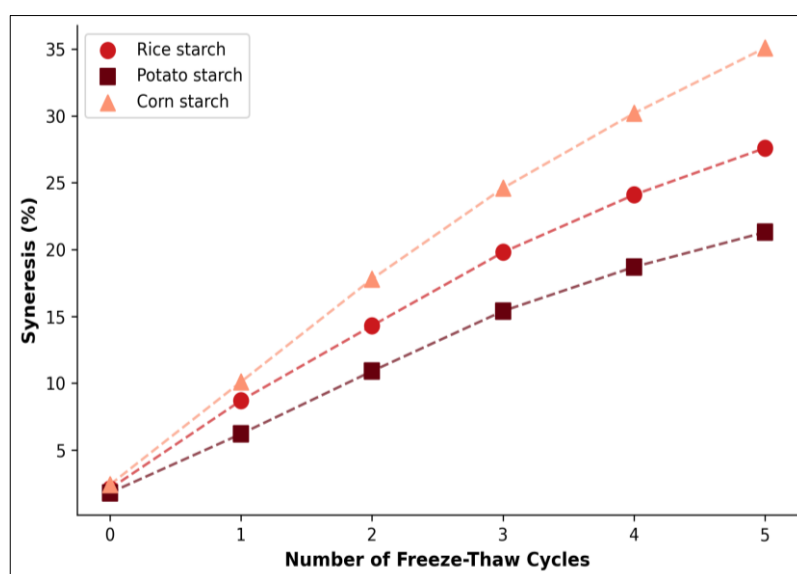


Fig 1: Effect of freeze-thaw cycles on syneresis of three starch gels

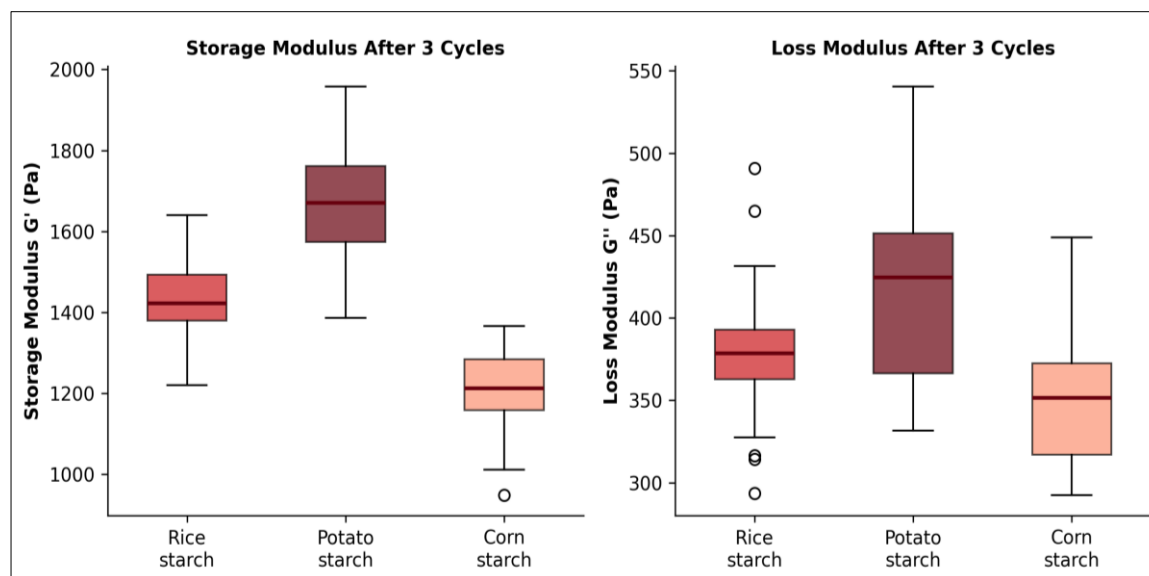


Fig 2: Distribution of storage modulus (G') and loss modulus (G'') after three freeze-thaw cycles

Table 3: Rheological parameters at 1 Hz after 0 and 5 freeze-thaw cycles

Starch type	G' cycle 0 (Pa)	G' cycle 5 (Pa)	G'' cycle 0 (Pa)	G'' cycle 5 (Pa)	$\tan \delta$ cycle 5
Rice starch	1820 ± 95	1090 ± 88	465 ± 38	298 ± 31	0.273
Potato starch	2140 ± 110	1410 ± 102	510 ± 42	368 ± 35	0.261
Corn starch	1650 ± 85	870 ± 76	410 ± 34	245 ± 28	0.282

Comprehensive interpretation

Potato starch consistently outperformed rice and corn starches in both syneresis resistance and structural retention across five freeze-thaw cycles. Its lower amylose content relative to corn starch may slow retrogradation, while the larger granule size and phosphate monoester groups characteristic of potato starch are known to restrict amylose chain realignment during cooling [5]. The practical implication is that bakeries producing frozen gluten-free dough would benefit from using potato starch as the primary base ingredient.

Discussion

Potato starch dough retained the highest G' values (1,410 Pa at cycle 5) and the lowest syneresis (21.3%) among the three starch types tested. This finding aligns with results reported by Kaur *et al.* [5], who observed that phosphorylated starches resist retrogradation more effectively than their non-phosphorylated counterparts. The underlying mechanism involves electrostatic repulsion between phosphate groups on adjacent amylopectin chains, which hinders the chain reassociation that drives gel shrinkage during frozen storage [11]. For product developers, this means that potato starch formulations are more forgiving in distribution chains where temperature fluctuations are unavoidable.

Corn starch, by contrast, showed the fastest rate of structural breakdown a 47% drop in G' over five cycles. Witczak *et al.* [8] reported a comparable decline (43%) in corn-based gluten-free dough held at -20°C , attributing it to the higher amylose fraction in corn starch (27% vs. 21–22% for potato and rice). The longer linear chains of amylose retrograde more aggressively, forming rigid crystalline zones that expel water from the surrounding matrix [12]. And because syneresis in commercial bread dough translates directly to a drier, more crumbly crumb after baking, corn starch appears

to be the least suitable base for frozen dough applications among the three starches evaluated.

The strong inverse correlation between syneresis and G' ($r = -0.89$) underscores a simple but useful principle: monitoring water release during freeze-thaw testing can serve as a reliable proxy for gel structural integrity, reducing the need for time-consuming rheological measurements in routine quality checks.

Conclusion

Frozen dough technology remains central to the modern baking industry, and gluten-free products stand to gain the most from improved freeze-thaw stability because their structural weaknesses are amplified by ice crystal damage. Among the three starch types tested, potato starch delivered the best overall performance, with 21.3% syneresis and 1,410 Pa storage modulus after five freeze-thaw cycles. Rice starch occupied a middle position, while corn starch showed the most severe deterioration. Based on these results, formulators working on frozen gluten-free bread should prioritise potato starch as the primary structural component, possibly supplemented with hydrocolloids or modified starches to further limit retrogradation. Looking ahead, research into enzymatically modified potato starch or blends with resistant starch fractions could push freeze-thaw tolerance even further, opening the door to gluten-free products with shelf lives comparable to their wheat-based counterparts.

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