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Sweetpotato chip texture and fat content: Effects of enzymatic modification of cell wall polymers

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Abstract: Impacts of cell wall polymers on sweetpotato chip texture and fat content were investigated through enzymatic modification. Covington sweetpotato slices were treated with cellulase, hemicellulase, pectinase, pectin methyl esterase, protease, the enzyme blend Viscozyme, or no enzymes (control) at 40–45°C for 0.5–2 h. Treated slices were blanched, dried, and fried in triplicate per experimental condition. Breaking forces of 20 chips per frying replicate were measured followed by chip fat, moisture, sugar, alcohol insoluble solids, glass transition temperature, and color analyses. Untreated slices from each batch (daily check) were fried and analyzed to account for starting material variability. Viscozyme and protease-treated chips had the greatest reduction in breaking force from untreated chips (−30.9% and −23.7%, respectively), while pectin methyl esterase-treated chips had the lowest reduction in breaking force (−9.0%). Chips treated with Viscozyme for 2 h were 6.7-6.3 percentiles lower in fat than the control. Principal component analysis elucidated that chip breaking force was associated with unfried slice puncture force, alcohol insoluble solids, and chip color, and chip fat content was inversely associated with maltose content and glass transition temperature. Breaking down multiple cell wall polysaccharides or structural proteins weakened chip textures, while strengthening the pectic fraction resulted in harder chips. Chip fat reduction also occurred when multiple cell wall polysaccharides were broken down. Therefore, cell wall polymers impact sweetpotato chip texture and fat contents, and their attributes should be considered when selecting cultivars and processes for sweetpotato chips.

KEYWORDS
chip, enzyme, fat, sweetpotato, texture

Practical Application: Sweetpotato chips are an increasingly popular snack, but there is little understanding how cell wall polymers impact chip textures and fat contents. Raw sweetpotato slices were enzymatically treated to selectively modify cell wall polymers before frying. Chip breaking forces were lowered by protease or Viscozyme (cell wall enzyme blend) treatments, while breaking forces were increased with pectin methyl esterase. In addition, chip fat contents were reduced by the Viscozyme treatment. Since cell wall modifications

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could impact chip texture and fat content, cell wall polymer attributes should be considered in selection and processing of sweetpotatoes for chip manufacturing.

1 | INTRODUCTION

The sweetpotato (*Ipomoea batatas*) is a nutritious crop that can be processed into many value-added products, such as French fries, chips, frozen dices, purees, canned, and dried products (Truong et al., 2018). In the United States, a substantial portion of the sweetpotato crop is processed into sweetpotato chips. For example, 11.2 million kilograms of North Carolina sweetpotatoes were processed into chips in 2019 alone (US Sweet Potato Council, 2020). It is likely sweetpotato chip production will continue to increase with the growing demand for sweetpotatoes.

For efficient development of new sweetpotato chip cultivars and processes, it is important to understand which intrinsic factors influence chip texture and oil uptake. It has been shown that sweetpotato starch or dry matter levels are positively correlated with chip breaking force (Gao et al., 2014) and negatively correlated with oil uptake (Hagenimana et al., 1998); yet, little is known on the influence of cell wall polymers on sweetpotato chip textures and fat contents. Sweetpotato French fry textural attributes have also been associated with starch and dry matter contents (Sato et al., 2018) and starch physicochemical properties (Allan et al., 2021), but sweetpotatoes that were similar in these properties could still have widely varying textures (Sato et al., 2018). It is postulated that the sweetpotato cell wall polymers may have also impacted these textures. For example, modifying pectin chemically or with endogenous pectin methyl esterase impacted blanched, steam cooked, and fried sweetpotato textures (Truong et al., 2006; Walter et al., 1992, 2003); sweetpotato pectin degradation during storage was speculated to decrease sweetpotato product firmness (Walter & Palma, 1996); raw sweetpotato textures were correlated with cell wall composition and gene expression of the cell wall protein expansion (Dong et al., 2020), and fried potato (*Solanum tuberosum*) chip and French fry textures and fat contents were impacted by cell wall polymers (Kita, 2002; Lisińska et al., 2007). Since cell wall polymers influence cooked sweetpotato textures and fried potato textures and fat contents, it is plausible that sweetpotato chip textures and fat contents may also be affected.

The effects of specific cell wall polymers on sweetpotato chip texture and fat contents were explored by targeted enzymatic modifications of the following cell wall polymers: cellulose, hemicellulose, pectin, and structural proteins. Cellulose arranges in crystalline microfibrils and functions mainly as structural scaffolding, while hemicellulose is a broad category of branched polysaccharides (e.g., xyloglucans, arabinoxylans, and galactomannans) that strongly interacts with cellulose as well as interacts with pectin in the middle lamella. The pectic fraction mainly resides in the middle lamella to adhere cells together and has homogalacturonan, rhamnogalacturonan I and II domains with intricate branching and esterified moieties. Structural proteins, such as hydroxyproline-rich glycopoproteins (e.g., expansins), provide unique structural support such as during cell growth, wound response, and can be crosslinked and insolubilized with cell wall polysaccharides (Amos & Mohnen, 2019; Carpita & Gibeaut, 1993; Cassab, 1998; Cosgrove, 2005; Dong et al., 2020; Jarvis et al., 2003; Kieliszewski & Lamport, 1994; Watkins, 2017). The objective of this study was to enzymatically modify these targeted sweetpotato cell wall polymers and observe the effects on sweetpotato chip texture and fat content to better understand which class of cell wall polymers most impact these sweetpotato chip attributes.

2 | MATERIALS AND METHODS

2.1 | Raw materials, enzymes, and chemicals

Covington sweetpotatoes with diameters between ~6 cm and 13 cm were grown at the Horticultural Crops Research Station in Clinton, NC, USA during the 2020 season by the Sweetpotato Breeding and Genetics Program at North Carolina State University, Raleigh, NC, USA. Covington is a uniformly shaped, orange-fleshed variety (Yencho et al., 2008) that is suitable for chipping and frequently used for fried sweetpotato products in the US. Reported Covington sweetpotato composition on a fresh weight basis is 20% dry matter, 9.5% starch, 3.8% sucrose, 0.9% glucose, 0.6% fructose, no maltose detected, 1.8% protein, and 2.2% dietary fiber (Yencho et al., 2008). Sweetpotatoes were cured at 85–90% RH at 30°C for 7 days and then stored at 85–90% RH at 14°C for 6 months. Canola oil with tert-butylhydroquinone (antioxidant) and dimethylpolysiloxane (antifoaming agent) (Swad, Raja Foods, Chicago, IL) was sourced for frying because it was designed for the harsh deep-fat frying conditions.

Enzymes used to treat sweetpotato slices prior to frying were sourced from Novozymes (Copenhagen, Denmark):

Viscozyme L (Viscozyme) is a mixture of pectinases, hemicellulases, xylanases, and β-glucanases (107.1 units per gram); Celluclast 1.5 L (cellulase) is a cellulase (765.5 units per gram), NovoShape (PME) is a pectin methyl esterase (11.4 units per gram), Pectinex Smash XXL (pectinase) is a pectin lyase (21,515 units per gram); Shearzyme 500 L (hemicellulase) is a xylanase (544 units per gram) and was selected because the predominate hemicellulose in sweetpotatoes are xylans (Noda et al., 1994); and Alcalase Pure (protease) is a broad spectrum endo-protease (2.6 units per gram) (Novozymes, 2021). Liquid enzymes were used according to supplier recommendations and stored at 4–6°C until use.

HPLC standards D-sucrose, D-maltose monohydrate, D-glucose, D-galactose, L-fucose, L-arabinose, D-cellobiose, D-xylose, D-mannose, D-galacturonic acid monohydrate, and D-mannitol were sourced from Sigma Aldrich (St. Louis, MO, USA); L-rhamnose and D-maltotriose from Indofine Chemicals (Hillsborough, NJ, USA); and D-fructose from Acros Organics (Geel, Belgium). Food grade acetic acid (99.5%) and sodium acetate (99%) were sourced from Aldrich Chemicals (Milwaukee, WI, USA). Hexanes solvent and 50% sodium hydroxide solution were sourced from Fisher Chemical (Waltham, MA, USA) and 95% ethanol from Decon Labs, Inc. (King of Prussia, PA, USA).

2.2 | Sweetpotato chip processing

Sweetpotatoes were washed, peeled, and sliced 1.5-mm thick latitudinally (perpendicular to its vascular system) using a Hobart FP150 continuous feed food processor (Hobart Corp., Troy, OH, USA). Slices were gently hand tossed to randomly distribute, and then 600 g subbatches were filled into 30 × 60 cm expandable nylon mesh bags for a total of three bags per enzyme treatment. For each day of processing, sweetpotato roots were sliced, mixed, aliquoted, and randomly assigned to the designated treatments, and samples of raw, untreated sweetpotato slices from each day of processing (daily check) were fried and analyzed to account for biological variability in the raw materials (Figure S1).

Enzyme solutions were prepared in 10 L volumes in 18.9 L high-density polyethylene buckets using 45–50°C activated carbon filtered water. All enzymes in this study had optimum activities around pH 5, except the protease which had an optimum pH around 7. Thus, the water was buffered to pH 5 using a 0.01 M acetic acid–sodium acetate buffer or left unbuffered for pH 7, and pH values were verified using a pH meter calibrated with pH 4 and 7 standards before and after enzyme treatments. The buckets of pH-controlled water were held in an incubator at 45°C until the temperature was 45±1°C, and then liquid enzymes were added to make 0.1% enzyme solutions. Shortly after adding the enzyme, three 600 g bags of raw sweetpotato slices were submerged in the enzyme solution followed by 15 s of bobbing to help disperse enzyme solution throughout the slices, and then bags were weighed down to ensure complete submersion during the treatment. The enzyme solutions with sweetpotato slices were held in an incubator at 45°C, but the solution temperature initially dropped to ≈40°C and gradually increased over 2 h. Every 10 min for the first 30 min and one more time after the first hour, the bags were bobbed for 15 s to reposition the slices and mix the enzyme solution. A bag of sweetpotato slices was removed after 0.5, 1, and 2 h of enzyme exposure and was immediately blanched in 100°C water for 10 s while bobbing to stop enzymatic activities. The blanched slices were then spread out in a single layer and dried in an open-faced forced air food dehydrator (The Sausage Maker Inc., Buffalo, NY, USA) at 35°C for 10 min. Treated sweetpotato slices and daily untreated checks (raw slices) were fried in three 150 g subsample batches in canola oil at 149°C for 3 min in a 22 L electric fryer (1ER50 Series, Vulcan-Hart Co., Louisville, KY, USA) (Figure S1). Immediately after frying, chips were vigorously shaken for 2–3 s to remove excess oil, dispensed onto wire racks, and spread out in a single layer to cool. Room temperature chips were bagged in resealable zipper storage bags and stored at −20°C until use.

2.3 | Texture analysis

Chip breaking force was measured on a TA.XT2 Texture Analyzer (Texture Technologies Corp., Hamilton, MA, USA) with a 12.7-mm ball probe and Crisp Fracture Support Rig (Salvador et al., 2009). The peak force of 20 chips from each frying replicate was measured. Breaking force outliers within a treatment condition were identified, and then removed using the Robust Fit Outlier test using the quartile K spread with K Sigma = 3. The remaining values were averaged as the representative frying lot's breaking force. Puncture forces of 20 unfried sweetpotato slices were measured using a 7-mm rounded tipped probe and a plate with a 10=mm round hole. Texture analyzer settings for both force measurements were 0.8 mm/s pretest, 0.5 g trigger force, then 1.0 mm/s test speed for 5 s. Chips used for texture analysis were ground with a mortar and pestle into a puree and used for fat content, moisture content, and color measurements (Figure S2).

2.4 | Moisture content

Moisture contents of ground chips were measured using a modified AOAC 925.45 method (AOAC, 1990). Briefly, ~2 g of chip puree was weighed into 57-mm aluminum pans

and dried until weight loss stopped in an Across International AT09 vacuum oven (Across International, Berkeley Heights, NJ, USA) for > 3 h at 60°C and < 1 kPa with a slow purge of 0% RH air through a Drierite® trap. Moisture content was reported on a wet weight basis.

2.5 | Color measurement

Approximately 3–5 g of ground chip was tamped into a 2-cm thick layer in a 47-mm clear petri dish (Fisherbrand, Waltham, MA, USA), and the D_{65} L^*, a^*, b^* color values were measured using a Konica Minolta CM-700d Spectrophotometer (Osaka, Japan) with an 8-mm aperture with plate. Color was measured in three regions and averaged to obtain representatitive color values for each frying replicate. The ΔE was calculated as color value changes in treated chips to the respective untreated daily check.

2.6 | Chip defatting and Soxhlet-extracted crude fat contents

Chip crude fat analysis was modified from Sato et al. (2018). About 3 g of ground chips was weighed into 33 × 94 mm cellulose thimbles, and then packed with glass wool to prevent sample floating during extraction. Chip lipid fraction was extracted using a Buchi E-816 Soxhlet (BUCHI Corporation, New Castle, DE, USA) with hexanes for 5 h, then rinsed with hexanes for 10 min, and finished with a 2 h distillation step to remove most of the hexane solvent from the lipid extract. The lipid extract was then dried for 1 h at 105°C and cooled at 0% RH. The weight of the dried lipid extract was recorded as the crude fat content as any component soluble in hot hexane was coextracted. The defatted material was saved for glass transition, alcohol insoluble solids, and sugar content measurements (Figure S2).

2.7 | Time domain nuclear magnetic resonance measured fat contents

Fat contents of ground chips were also measured using a time domain nuclear magnetic resonance (TD-NMR) Bruker minispec mq-one seed analyzer (Bruker Corporation, Billerica, MA, USA) that was calibrated with the same, but unused, canola frying oil. A 10 g portion of ground chips was weighed into a glass NMR tube and then a single pulse sequence was used to quantify chip oil content.

2.8 | Differential scanning calorimeter

Glass transition temperatures (T_g) of chips were measured using ~10 mg of defatted, ground chips in 50 µl vented aluminum pans with a Perkin Elmer DSC 6000 (Waltham, MA, USA) that was calibrated with indium and tin. Samples were heated from 30°C to 140°C at 10°C/min, held at 140°C for 5 min to dry samples, cooled to −40°C at 50°C/min, then heated to 140°C at 10°C/min. The thermogram from the second heating scan was used for T_g calculations by the Perkin Elmer Pyris software 13.3.2.0030. The T_g onset, T_g midpoint, T_g end, and heat capacity change (ΔC_p) were recorded.

2.9 | Alcohol insoluble solids

Alcohol insoluble solids (AIS) were prepared from the method according to Sato et al. (2018) with modifications. Briefly, ~1 g of defatted chip powder was weighed into a 50 ml centrifuge tube, ~25 ml of 70°C 95% ethanol was added, vortexed for 1 min, and then centrifuged at 6500*g* for 10 min at 20°C. Supernatant was decanted into a 50 ml volumetric flask, the extraction was repeated, and the volume of the combined supernatants was brought to 50 ml and saved for chip sugar content analysis. After the first extraction process, it was evident that there was incomplete sugar extraction due to high AIS and low sugar quantities when compared to values reported for Covington sweetpotatoes by Sato et al. (2018). To complete the extraction, the partially extracted AIS fractions were milled with a rounded glass rod to fine powders in the original tubes. The sugar extraction was then repeated as previously described and the sugar content calculated as the sum of the two extracts. AIS fractions were dried at 75–80°C for 4 h to remove most of the remaining ethanol and then finish dried in a vacuum oven for > 1 h at 75°C and < 1 kPa until weight change stopped.

2.10 | High-performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD)

Glucose, fructose, sucrose, and maltose contents in chips were quantified with high-performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD). Two hundered microliters of ethanol extract were dried in 1.5 ml centrifuge tubes in a Speed-Vac SPD1030 Integrated Vacuum Concentrator (Savant, ThermoFisher Scientific) for 30 min at 60°C followed by

no heating for another 30 min while at < 1 kPa. The precipitate was reconsituted with 1 ml of water and vortexed till fully dissolved. All mobile phases were filtered through a 47 mm 0.45 μm nylon membrane (GVS Life Sciences, Bologna, Italy) and samples were passed through a 13 mm 0.45 μm polytetrafluoroethylene membrane (ThermoFisher Scientific). Sugars were seperated isocratically with 200 mM NaOH at 1 ml/min on a Dionex™ CarboPac™ PA1 (4 × 250 mm) with guard column (4 × 50 mm) (ThermoFisher Scientific) at 30°C on a Shimadzu Prominence HPLC system (Kyoto, Japan) and detected using an Antec Scientific Decade Elite (Zoeterwoude, Netherlands) PAD with a SenCell 2 mm Au HyRef. External standard curves of gluose, fructose, sucrose, and maltose were used to quantify sugar contents.

Sugars leached from the sweetpotato slices into enzyme solutions were compared on a semiquantitative basis using HPAEC-PAD chromatogram peak areas. Sugars and other PAD active compounds from undiluted enzyme and control solutions were separated using the same apparatus and settings as previously described with modified mobile phase conditions adapted from Widmer (2011). The mobile phase flow rate was 1.1 ml/min with mobile phase A as 18 mM NaOH and mobile phase B as 500 mM NaOH: 0–7 min 100% A; 7–16 min linear gradient to 88.67% A; 16–22 min linear gradient to 0% A (100% B); 22–32 min 0% A; then requilibrated the column with 100% A for 20 min before the next injection. Chromatograms using both 1 μl and 10 μl injections of each treatment solution were generated to compare predominate sugars (e.g., glucose) and trace sugars (e.g., L-fucose), respectively. These data and discussion on unique differences in sugars and cell wall sugars released due to individual enzyme treatments are found in the Supporting Information.

2.11 | Data analysis

Differences in chip properties among experimental conditions and untreated daily checks were compared using one-way ANOVA (Table S1). The impact of treatments, treatment time, and their interaction on a given chip attribute were compared using two-way ANOVA. If the treatment time nor the interaction were significantly associated with a given chip attribute and there were no differences between treatment times within a treatment, then values across all treatment times were pooled for treatment comparisons. Untreated daily check chips varied in chip breaking force (Table S2); therefore, to account for raw material variability on chip breaking force, the percent reduction in breaking force from the untreated daily check chips to the treated chips were reported and used for statistical analyses. All ANOVA analyses used a Tukey's HSD post hoc test with $\alpha = 0.05$ and were performed in JMP Pro 15.2.1 (SAS Inc., Cary, NC, USA).

Associations of sweetpotato chip breaking forces and fat contents with the other measured chip attributes were compared using a linear correlation matrix and principal component analysis (PCA). Untreated check chips were not included in the multivariate methods because they had substantially different textures, fat contents, and compositions that skewed the multivariate analyses, overshadowing differences between treated chips. The α value for the correlation matrix was 0.01 due to the high number of samples ($n = 72$). Both multivariate analyses were conducted in JMP Pro 15.2.1.

3 | RESULTS AND DISCUSSION

3.1 | Chip texture

Enzyme treated and control chip breaking forces ranged from 3.6 N to 4.8 N, and average breaking forces of untreated daily check sweetpotato chips ranged from 4.8 N to 5.4 N (Table S1 and Figure S3). All treated chips had lower breaking forces compared to the untreated daily checks including controls, which were soaked and blanched but not enzymatically treated. The 0.5–2 h soak at 40–45°C would be expected to induce β-amylase activity, converting available glycans (e.g., dextrins and portions of starch) to maltose (Hagenimana et al., 1994; Takahata et al., 1994), and the 100°C blanch would gelatinize starch plus dissolve and break down portions of the pectic fraction (Fraeye et al., 2007; Valetudie et al., 1999). These processes soften sweetpotato tissue (Valetudie et al., 1999) and likely lowered treated chip breaking forces compared to the untreated daily checks. In addition, the spent treatment solutions had varying amounts of cell wall sugars and unknown PAD-active compounds that were released from the sweetpotato slices, indicating unique enzymatic modifications took place among the treatments (Supporting Information, Table S18, and Figures S5–S12).

Daily check chip breaking forces were not the same between processing days (Table S2 and Figure S3), which suggests starting material variability could have impacted treated chip breaking forces. Therefore, the effects of treatments on chip texture were determined in respect to the untreated daily check chip textures and discussed as the percent reduction in chip breaking force from the untreated daily check.

Enzyme type was the only significant factor in the two-way ANOVA model, while treatment time nor the interaction were significant (Table 1). Thus, chip breaking forces across all treatment times were combined. The Viscozyme treatment induced the greatest reduction in chip

TABLE 1 Two-way ANOVA *p*-values

	p		
	Enzyme	**Time**	**Enzyme × time**
Chip peak force	**< 0.0001**	0.5635	0.1214
Breaking force difference from check	**< 0.0001**	0.5873	0.1580
Unfried slice puncture force	**< 0.0001**	0.0826	0.5031
%Crude fat (Soxhlet)	**< 0.0001**	0.4341	**0.0007**
% Fat (NMR)	**0.0029**	**0.0120**	**0.0026**
%MC	**0.0027**	0.5467	**0.0079**
L^*	0.3082	0.9910	0.1648
a^*	**0.009 1**	0.6326	0.7803
b^*	**0.0110**	0.7322	0.7893
ΔE	0.6663	0.1785	0.4973
AIS	**0.0078**	0.1631	0.9052
Chip glucose	**0.0004**	**0.0016**	0.0729
Chip fructose	**0.0295**	**<0.0001**	0.1914
Chip sucrose	0.7606	**0.0020**	0.1458
Chip maltose	**< 0.0001**	**0.0163**	**0.0002**
T_g onset	**< 0.0001** *	**0.0135**	**0.0309**
T_g mid	**< 0.0001**	**0.0208**	0.2874
T_g end	**< 0.0001**	**0.0041**	0.4182
ΔC_p	**0.0086**	0.1442	**0.0115**

Abbreviations: AIS, Alcohol insoluble solids; MC, moisture content; NMR, nuclear magnetic resonance. *P*-Values <0.05 are bolded.

breaking force (−30.9%), while PME-treated chips retained the highest breaking force, only a 9.0% reduction (Figure 1 and Table S3). Breaking force reductions of both Viscozyme and PME treated chips were significantly different than the control pH 5 chips (−19.3%). Viscozyme is a blend of pectinases, hemicellulases, xylanases, and β-glucanases (Novozymes, 2021), and since no single polysaccharidase reduced chip breaking force, this suggests that the breakdown of multiple cell wall polysaccharides was needed to induce significant reductions in chip breaking force (Figure S4). PME demethoxylates the homogalaturonan regions of pectin, which encourages cation crosslinks. This increases pectin strength and decreases pectin solubility and β-elimination during cooking and has been shown to affect sweetpotato and other fruit and vegetable textures (Buescher & Balmoori, 1982; Keijbets & Pilnik, 1974; Ross et al., 2011). For example, endogenous PME activity in sweetpotatoes increased cooked sweetpotato firmness (Walter et al., 2003) and can induce "hardcore," a defect characterized by a sweetpotato that fails to soften when cooked (Buescher & Balmoori, 1982). Similarly, increased PME activity and lower degree of pectin methylation in potato tissue resulted in a firmer cooked texture (Andersson et al., 1994; Ross et al., 2011). The PME treatment in this study also impacted chip breaking force (Figure 1), presumably by increasing the strength of the pectic fraction (Figure S4), which is the stated function by the manufacturer (Novozymes, 2021).

Breaking forces of protease-treated chips were also lower than its control, control pH 7 (Figure 1 and Table S3). This suggests there are proteins that influence sweetpotato chip textures. There are structural proteins in plant cell walls that affect cellular strength, such as hydroxyproline-rich glycosylated proteins (Cassab, 1998; Lamport et al., 2011) and cell wall linked arabinogalactan proteins (Seifert & Roberts, 2007; Tan et al., 2013). There is evidence that proteins interact with cell wall polysaccharides and influence the tensile strength (Cassab, 1998), and modifications to cell wall structural proteins, via mutations, can lead to weakened cell walls, demonstrating their significance in cell structures (Nguema-Ona et al., 2014). Interestingly, microalgaes treated with the same protease used in this study, Alcalase, had greater cell wall disruption compared to cells treated with Viscozyme, also used in this study (Mahdy et al., 2014). Therefore, cell wall proteins are important polymers for plant tissue strength, and these protease-treated chips were weaker likely due to the breakdown of these structural proteins.

Chip breaking forces were correlated with unfried slice puncture forces (Table 2). Gao et al. (2014) reported a similar correlation between unfried puncture and fried chip breaking forces. Even though the frying process greatly changes the material properties from a moist malleable slice to a dry brittle chip, textural changes in the unfried sweetpotato slices from cell wall polymer modifications impacted fried chip breaking forces.

TABLE 2 Correlation coefficient (r) matrix of treated sweetpotato chip attributes (untreated check samples were not included)

	Chip breaking force	**Unfried puncture force**	**%Crude fat (Soxhlet)**	**% Fat (NMR)**	***L**D65**	***a**D65**	***b**D65**	**Δ*E***	**T_g onset**	**T_g mid**	**T_g end**	**ΔC_p**	**Chip moisture**	**% AIS**	**Glucose**	**Fructose**	**Sucrose**
Unfried puncture force	**0.370**																
%Crude fat (Soxhlet)	0.098	0.236															
% Fat (NMR)	−0.011	0.099	**0.857**														
*L**D65	0.041	0.245	−0.295	−0.282													
*a**D65	0.242	**0.327***	0.049	−0.043	**0.731***												
*b**D65	0.255	**0.327**	0.128	0.089	**0.702**	**0.956**											
Δ*E*	−0.140	−0.087	−0.021	−0.004	−0.170	−0.117	−0.060										
T_g onset	−0.221	0.030	−0.095	−0.253	0.166	0.044	0.017	0.073									
T_g mid	0.000	0.171	−0.023	−0.283	0.036	0.103	0.070	0.095	**0.674**								
T_g end	0.029	0.223	0.274	0.099	0.031	0.188	0.149	−0.130	0.146	0.299							
ΔC_p	0.258	0.182	−0.239	−0.218	−0.077	−0.137	−0.132	0.036	−0.159	−0.004	−0.099						
Chip Moisture	0.123	0.020	0.096	0.086	0.224	0.222	0.204	0.046	−0.147	−0.180	0.143	−0.052					
% AIS	0.077	0.209	−0.085	−0.163	0.174	0.234	0.095	−0.283	−0.156	0.071	0.267	0.131	0.129				
Glucose	−0.064	−0.189	−0.086	−0.012	−0.018	−0.167	−0.082	**0.348**	0.158	0.144	**−0.397**	−0.010	−0.030	**−0.596**			
Fructose	−0.119	−0.213	0.091	0.202	−0.141	−0.223	−0.133	**0.335**	−0.053	−0.057	**−0.331**	−0.180	−0.028	**−0.621**	**0.896**		
Sucrose	−0.045	−0.064	0.136	0.241	−0.099	−0.135	−0.068	0.243	−0.104	−0.104	−0.191	−0.252	−0.025	**−0.549**	**0.698**	**0.839**	
Maltose	0.008	−0.079	**−0.389**	**−0.406**	0.193	0.004	0.033	0.178	**0.530**	**0.435**	−0.298	**0.317**	−0.168	**−0.345**	**0.581**	0.247	0.083

Note: Significant correlations with *p*-values < 0.01 are bolded ($n = 72$).

Abbreviations: AIS, Alcohol insoluble solids; MC, moisture content; NMR, nuclear magnetic resonance.

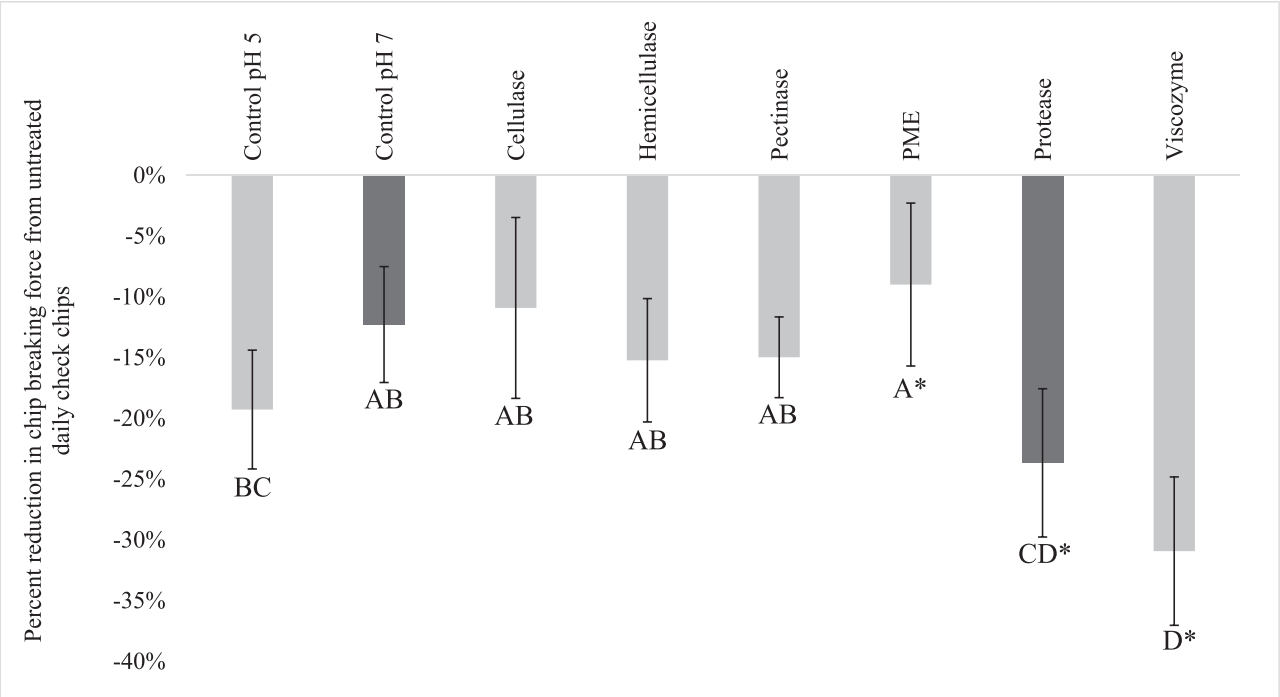

FIGURE 1 Percent reduction in chip breaking force of enzyme-treated chips from untreated daily check chips. The pH 5 treatments are light grey bars, and the pH 7 treatments are dark grey bars. Breaking forces from all treatment times were pooled ($n = 9$, statistical differences from controls are indicated with an asterisk)

3.2 | Chip fat content

Chip crude fat contents ranged from 42.2% to 53.6% using a Soxhlet apparatus and TD-NMR measured fat contents ranged from 38.9% to 50.8% (Table S1). Fat contents of untreated daily checks (39.4–44.1% fat) were comparable to fat contents of commercial sweetpotato chips (28.0–39.3%) (Table S5) and chips made from 6-month Covington sweetpotatoes chips (34.1 ± 1.2% fat) in a previous study (Qiu, 2019). Enzyme treated and control chips generally had higher fat contents than untreated daily check samples (Tables 3 and 4), thus the 40–45°C soak and/or blanch increased fat uptake. In potato chips, blanched slices (85°C for 3.5 min) absorbed more oil than unblanched slices as a result of surface wetting (Pedreschi & Moyano, 2005b), but additional drying and higher frying temperatures could help reduce oil absorption (Pedreschi & Moyano, 2005a). The higher oil contents in treated sweetpotato chips may be a function of surface moisture and reducing oil uptake can be further optimized with processing modifications. Nonetheless, oil uptake between enzyme treatments could be compared as they underwent similar surface wetting and drying procedures.

The Soxhlet-extracted crude fat contents of treated chips were impacted by treatment type and the interaction with time (Table 1) and will be compared within treatment times (Table 3). Crude fat contents were not different from each other except the 2 h Viscozyme-treated chips, which were 6.7 percentiles lower than the control (Table 3). TD-NMR measured chip fat contents were also affected by treatment, treatment time, and the interaction (Table 1). Fat contents of 2 h Viscozyme-treated chips plus 0.5 h hemicellulase, 0.5 h pectinase, and 0.5 h Viscozyme chips were significantly lower than the control (Table 4). However, the lower fat contents of these 0.5 h treatments from the control pH 5 may be inconsequential since they were not different from other 0.5 h treatments, and differences were not found in the Soxhlet crude fat extractions. Whereas the crude fat and fat contents of 2 h Viscozyme-treated chips were lower than most other treated chips and was the only treatment that was not significantly higher than the untreated daily check chips. Thus, there is confidence the 2 h Viscozyme treatment lowered chip fat content compared to the control treatment.

The 2 h Viscozyme treatment likely caused the largest breakdown of cell wall structures, since these chips and unfried slices had the lowest breaking forces in the study (Table S1 and S4), and the spent Viscozyme solution had the highest concentration of cell wall sugars and galacturonic acid (Supporting Information, Table S18 and Figures S5–S6). This cell wall breakdown impacted oil uptake as well. During frying, water evaporates from cells and forms steam channels in the tissue, which then become pathways for oil absorption into the chip by vacuum and capillary

TABLE 3 Sweetpotato chip Soxhlet extracted percent crude fat with respect to treatment time

Soxhlet extracted %Crude Fat												
	0.5 h Treatment				1 h Treatment				2 h Treatment			
Treatment	Mean		Std. dev	Tukey HSD	Mean		Std. dev	Tukey HSD	Mean		Std. dev	Tukey HSD
Untreated	42.5	±	1.9	B	42.5	±	1.9	B	42.5	±	1.9	D
Control pH 5	52.3	±	1.9	A	48.4	±	2.3	A	49.2	±	1.2	AB
Control pH 7	49.3	±	1.8	A	52.1	±	1.8	A	53.6	±	2.6	A
Cellulase	49.0	±	3.1	A	50.9	±	2.5	A	50.0	±	2.5	AB
Hemicellulase	50.8	±	1.0	A	50.0	±	3.0	A	47.0	±	1.0	BC
Pectinase	48.0	±	0.6	A	53.6	±	1.0	A	53.5	±	1.9	A
PME	52.0	±	1.4	A	50.8	±	1.6	A	51.6	±	2.4	AB
Protease	52.1	±	1.2	A	50.0	±	3.0	A	51.0	±	1.9	AB
Viscozyme	47.7	±	1.0	A	49.1	±	2.8	A	42.5	±	0.5	CD

Note: The pH 5 treatments are white, and the pH 7 treatments are grey. Statistical differences between enzymes are indicated by capital letters (Tukey HSD post-hoc test, $\alpha = 0.05$).

TABLE 4 Sweetpotato chip TD-NMR measured percent fat with respect to treatment time

TD-NMR Measured %Fat												
	0.5 h Treatment				1 h Treatment				2 h Treatment			
Treatment	Mean		Std. dev	Tukey HSD	Mean		Std. dev	Tukey HSD	Mean		Std. dev	Tukey HSD
Untreated	40.6	±	1.6	C	40.6	±	1.6	C	40.6	±	1.6	C
Control pH 5	50.8	±	4.0	A	46.4	±	2.3	AB	45.2	±	1.0	AB
Control pH 7	45.8	±	1.6	AB	49.0	±	1.7	A	47.1	±	2.2	AB
Cellulase	45.8	±	3.0	AB	47.6	±	2.3	AB	47.2	±	2.2	AB
Hemicellulase	44.8	±	1.3	B	43.5	±	2.1	BC	43.1	±	1.0	BC
Pectinase	44.9	±	0.9	B	48.4	±	0.6	AB	48.1	±	1.5	A
PME	48.2	±	1.1	AB	47.1	±	1.6	AB	46.3	±	2.1	AB
Protease	47.4	±	0.3	AB	47.8	±	2.1	AB	46.1	±	1.6	AB
Viscozyme	45.1	±	1.1	B	46.5	±	2.5	AB	38.9	±	1.9	C

Note: The pH 5 treatments are white, and the pH 7 treatments are grey. Statistical differences between enzymes are indicated by capital letters (Tukey HSD post-hoc test, $\alpha = 0.05$).

forces after frying (Pedreschi et al., 2018). The 2 h Viscozyme treatment likely impeded these oil absorption channels by collapsing and/or plugging them. Similarly, potato French fries treated with Pectinex SP-L, a combination of hemicellulases, β-glucanases, and pectinases (Novozymes, 2021), had less oil uptake than the control and it was attributed to the enzyme blend weakening the cellular structure. This reduced oil uptake was associated to starch leaching out of the cells and forming a film on the surface and steam channels collapsing after frying (Lisińska et al., 2007).

It is important to note, the crude fat contents measured using the Soxhlet method were generally 1–3% higher than the TD-NMR measured fat contents (Tables 3 and 4). The difference is likely due to the mechanism of fat measurement. Anything soluble in hot hexane could be theoretically extracted during the Soxhlet extraction. This would include phospholipids, carotenoids, and any nonpolar compound. Whereas the TD-NMR is a more selective method that was calibrated with canola oil and uses lipid proton relaxation rates for fat determination (Ellefson, 2017). Similarly, Shin et al. (2013) also reported that Soxhlet-extracted crude fat contents were greater than or equal to fat contents measured by a more selective gas chromatography method.

3.3 | Chip color

Chip a^* and b^* values were affected by treatment type but not treatment time, while L^* and calculated ΔE values were not associated with experimental variables (Table 1). There

were no significant differences between L^*, a^*, b^*, or ΔE values of enzyme-treated chips and the respective controls, but a^* and b^* values of Viscozyme-treated chips were lower (less orange) than some of the other treatments (Table S6). It is worth noting the chips were blanched to stop enzymatic reactions, which this step would remove surface sugars, browning substrates, negating some potential differences.

3.4 | Attributes affecting chip textures and fat contents

Chip compositions and material properties were explored to investigate changes induced by the treatments and any associations with chip breaking force reduction and fat content. Chip thermal properties, moisture, sugar, and alcohol insoluble solids contents were compared.

3.4.1 | Defatted chip glass transition temperatures and heat capacity change

Glass transition temperatures and heat capacity changes were measured in dried defatted chips, and the T_g onset, mid, and end temperatures ranged from 69.9–76.3, 83.1–89.0, and 100.2–103.2°C, respectively, and the heat capacity change ranged from 0.24 J/(g × K) to 0.42 J/(g × K) (Table S1). Untreated daily check chips tended to have lower T_g onset and ΔC_p values (Tables S7 and S10).

Chip T_g temperatures and ΔC_p were impacted by enzyme treatment and treatment times (Table 1). The T_g onsets of the untreated checks and the 0.5 h controls were ≈70.5°C, while 0.5 h cellulase and hemicellulase-treated chip T_g onsets were higher at 76.1 and 75.9°C, respectively (Table S7). The rest of the 0.5 h treatments and the 1 and 2 h treatments were not different from the control. Treated chip T_g mid and end temperatures were also not different from the control (Tables S8 and S9).

The ΔC_p values of treated sweetpotato chips did not differ from the control except for 2 h pectinase and 2 h PME-treated chips were less (Table S10). The ΔC_p of the T_g event of amorphous pectin decreases with decreasing pectin degree of esterification (Iijima et al., 2000); thus these enzymes, particularly PME, may have lowered the ΔC_p by altering the pectic fraction.

Chip T_g and ΔC_p values were not correlated with chip breaking force nor fat content (Table 2); therefore, there is no evidence to suggest changes in the glass transition event impacted these chip attributes. This could be due to the T_g events of these sweetpotato chips being ≈50°C above ambient temperature. This is consistent with previous findings that textures and force-deformation curves of crispy snacks were unaffected by T_g changes until the T_g decreases to near or below ambient temperatures (Roos et al., 1998).

3.4.2 | Moisture contents of chips

Sweetpotato chip moisture contents ranged from 0.86% MC_{wb} to 1.54% MC_{wb}, well within the expected moisture content of a sufficiently fried chip (< 5% moisture) (Kerr, 2017), and chip moisture contents were associated with treatment type and the interaction with time (Table 1). However, there were no differences in moisture content when compared within treatment times (Table S11); thus, chip moisture contents were likely not impacting chip attributes.

3.4.3 | Alcohol insoluble solids of defatted chips

The AIS fraction of plant materials is mainly cellular polymers such as starch, proteins, and cell wall polysaccharides (Fry, 2010). AIS contents were measured to compare the effects enzymatic treatments and possible associations with chip texture and fat content changes. Untreated daily check sweetpotato chips had higher amounts of AIS (83.2%) than enzyme treated and control chips (74.6–78.4%) (Table S12), and AIS was associated with only the treatment type and not treatment time (Table 1). Since even the controls had less AIS than the untreated chips, AIS material was being lost during the treatment process. This loss of AIS, and thus dry matter, may be a factor for the higher fat contents of treated chips compared to the untreated daily check chips (Tables 3 and 4), because sweetpotato chip fat contents were inversely correlated with dry matter (Hagenimana et al., 1998). However, AIS contents of treated chips were not correlated with chip breaking force nor fat contents (Table 2). AIS loss likely occurred, while the sliced chips soaked in warm water for up to 2 h and during blanching, where sugars and water-soluble polymers (e.g., cell wall polysaccharides and proteins) could leach out. In addition, some of the starch would be converted to maltose by β-amylase while incubating at 40–45°C (Hagenimana et al., 1994), which would also lower the AIS fraction. Despite the lower AIS values in treated chips compared to the untreated daily check chips, only the protease-treated chips were significantly different from its control (Table S12). Proteases may have reduced the AIS by increasing protein solubility (Tavano et al., 2018) and/or increasing starch breakdown by removing protein matrixes hindering amylase interactions (Rovalino-Córdova et al., 2018). Interestingly, the protease treatment

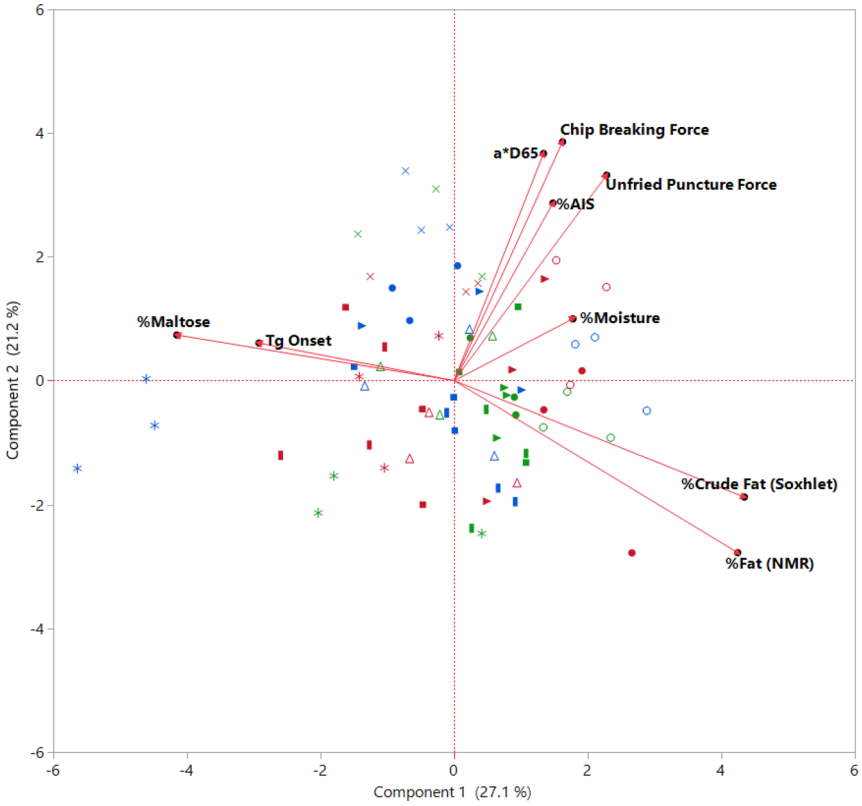

FIGURE 2 Principal component analysis of chip attributes. The data points are control pH 5 (●), control pH 7 (○), cellulase (■), hemicellulase (X), pectinase (▮), PME (►), protease (Δ), and Viscozyme (*) treatments for 0.5 (red), 1 (green), and 2 h (blue) treatment times

also significantly lowered chip breaking forces (Figure 1) and is plausible the lower AIS is associated. However, this relationship is not significant using linear correlations (Table 2), and further investigation is needed.

3.5 | Chip sugar contents

Sweetpotato chip glucose, fructose, sucrose, and maltose contents ranged from 1.65% to 1.99%, 1.72% to 1.91%, 9.83% to 12.23%, and 0.47% to 8.40%, respectively (Table S1), and all sugar contents were affected by treatment and treatment time, except sucrose content was not impacted by treatment type (Table 1).

Treated chips had significantly more maltose (≈10×) than untreated check chips because treated chips were soaked at 40–45°C for up to 2 h, which would have activated β-amylase and produced maltose (Hagenimana et al., 1994). This starch to maltose conversion is not conducive for sweetpotato chip manufacturing, as this could generate more browning and acrylamide (Truong et al., 2014). Therefore, care should be taken to minimize this conversion if sweetpotato chips were to be treated with enzymes.

The glucose, fructose, and sucrose levels in treated chips were not significantly different from their controls (Tables S13–S15), but the maltose contents in various treatments were higher than the control (Table S16). There was no consistent trend in maltose contents other than control pH 7 chips were consistently 2–3% lower than enzyme-treated chips. This may be due to enzymatic cellular damage releasing β-amylase plus substrates and the other treatments were at pH 5, closer to sweetpotato β-amylase optimum pH at 5.3–5.8 (Hagenimana et al., 1994).

There were no major differences in sugar contents between treatments nor was chip sugar contents correlated with chip breaking force or Soxhlet measured crude fat contents (Table 2). However, maltose contents were negatively correlated with TD-NMR measured fat contents, and it is unclear why this occurred. Further data analysis beyond linear correlations was needed.

3.6 | Principal component analysis

Differences in chip attributes between treated chips were also analyzed using PCA to simultaneously account for all measured chip properties and identify changes that are associated with one another (Figure 2). To minimize overemphasis of a single chip attribute in the PCA, only one representative value per analysis method was used. The T_g onset and maltose values were selected for the model because significant differences between treatments were present.

Chip breaking force was clustered with chip color (a*D65), unfried puncture force, and AIS (Figure 2). Chip crude fat and fat contents were clustered together and inversely associated with T_g onset and chip maltose contents. The PCA was similar to the linear correlation matrix where correlations were present between chip breaking and unfried puncture forces; chip color and unfried puncture force; T_g onset and maltose content; as well as maltose and fat contents (Table 2); yet, there were new associations when accounting for all measured chip attributes such as the relationship with AIS and chip breaking force.

Chip AIS content may be affecting chip texture, where more AIS results in harder chips. The association of AIS with chip texture is plausible because AIS is mainly composed of cellular polymers (Fry, 2010) such as cell wall polysaccharides and starch. Chips with higher breaking forces also tended to have higher a* values and likely b* since a* and b* values are highly correlated ($r = 0.956$). Therefore, harder chips tended to be more orange. Clearly, color has no direct impact on texture but may be associated with common factors such as chip thermal conditions during cooking. Interestingly, chip fat contents were negatively associated with T_g onset and maltose content. It is unclear why this occurred, but a speculation is the varying amounts of maltose generated may be altering the space and accessibility for oil absorption. This association could also be driven by the 2 h Viscozyme-treated chips. In Figure 2, these 2 h Viscozyme points are on the far left and these chips had the highest levels of maltose (Table S16), lowest fat contents (Tables 3 and 4), and lowest chip breaking forces (Figure 1). Other than unfried puncture force, there was no chip attribute linearly correlated with chip breaking force (Table 2); however, with PCA, there is evidence to suggest AIS also impacts chip breaking force. Similarly, chip fat contents may be inversely affected by maltose generation. More research with broader ranges of AIS and maltose contents is needed for stronger conclusions. However, since there were no definitive chip composition or thermal property associated with chip texture and fat contents that was consistent throughout all treatment conditions, it is likely the differences in chip texture and fat contents were driven by the enzymatic physicochemical changes to the cell wall polymers.

4 | CONCLUSIONS

The effects of cell wall modifying enzymes on sweetpotato chip textures and oil contents were investigated to elucidate the impact of cell wall polymers on these attributes. Sweetpotato chips treated with a protease or Viscozyme, a blend of multiple cell wall polysaccharide active enzymes, reduced chip breaking force, while strengthening the pectic fraction with PME increased chip breaking force compared to the control. Chips that were treated with Viscozyme for 2 h had lower fat contents than the control, likely a result from the enzyme breaking down multiple cell wall polysaccharides. Accounting for all measured chip attributes, AIS was associated with chip breaking force and is plausible that chips with lower AIS, thus cell wall material, will have lower breaking forces. Sweetpotato chip textures and fat contents were altered by enzymatic cell wall polymer modifications; therefore, sweetpotato chip texture and fat contents are not solely dictated by starch/dry matter and cell wall polymer attributes should be considered when selecting sweetpotatoes, storage conditions, and processing treatments for producing sweetpotato chips.

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AUTHOR CONTRIBUTIONS

Matthew Allan designed the experiment, collected data, performed data analysis and interpretation, and drafted and revised the manuscript. Suzanne Johanningsmeier consulted on experimental plans, contributed to interpretation of results and revised the manuscript.

CONFLICT OF INTEREST

There are no conflicts of interest to declare.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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