Effect of Relative Humidity, Storage Days and Packaging on Pecan Kernel Texture: Analyses and Modeling

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Abstract

The studies expounding effects of storage conditions on texture changes is limited. The researchers have been proposing methods to measure pecan texture instrumentally. But current protocols and/or attribute fail to address huge variability during experimentation. Additionally, there are no predictive model to estimate changes in pecan texture during storage. This study addresses all the above concerns and investigate effects of different relative humidity (RH, 30 to 90%) and packaging material (PE, PP, LDPE and metallic laminates or ML) on pecan texture, introducing a *rift* ratio (F/H or fracturability to hardness ratio) to address variability in the data and predictive model to estimate changes in textural attribute of pecans during storage. The textural analysis was conducted on pecans cores and intact pecans to measure area under curve, fracturability, hardness, cohesiveness, chewiness, springiness, and *rift* ratio. It was observed that values for *rift* ratio obtained using intact pecan method had high R^2 (0.72) as compared to rest of textural attribute. A 3-parameter logistic model was employed to predict pecan texture during strage.. The pecans stored at 75%, 80% and 90% lost half of initial fracturability at approx. 115, 3, and 0.15 days (~ 4 hours), respectively. The pecans stored in LDPE, PP and PE packs at 80% lost half of initial fracturability at approx. 26, 57, and 78 days, respectively. The presence of any kind of package delayed the fracturability loss by at least 8 folds at 80% RH. The pecans stored in ML did not experience significant change in textural attributes.

1. Introduction

Pecan is among the few US native crops with an annual crop value of 560 to 700 million USD (NASS 2020). The Pecan trees are alternate bearing and take anywhere between 5 to 10 years before producing nuts (Zhang et al. 2015). The yield of pecan trees is often diminished by factors such as excessive rain, drought, winds, sunlight exposure, or damage inflicted by insects, rodents, birds, or molds (Erickson et al. 1994). One strategy used by pecan growers and processors is to store nuts for extended periods to ensure a buffer to meet production demand both within and outside the US (NASS 2020). Along with Color and aroma(Kays 1979a; Prabhakar et al. 2022), pecan texture is an important indicator of pecan quality. Absence of crispiness and/or brittleness can discourage buyers from consuming pecans, and might a (Prabhakar et al. 2020)discourage them from purchasing future products. Several researchers have investigated the effects of different conditions encountered during harvesting, storage and transportation on texture of pecans including drying (Shult and Brusewitz 1998), freezing, thawing, and freeze/thaw cycles (Anzaldúa-Morales et al. 1999b; Surjadinata et al. 2001), oil removal (Shult and Brusewitz 1998; Zhang et al. 1995), and moisture restoration (Anzaldua-Morales et al. 1998).

Pecan kernels are non-uniform in size and have irregular surface structure which make it challenging to conduct instrumental measures of texture. The asymmetrical surface makes it difficult to attain a repeatable contact area between probe and nut, thus introducing variations that cause the test method to be inaccurate (Bourne 2002). Thus, researchers have relied on sensory panelists for texture evaluation of (Ocòn et al. 1995; Resurreccion and Heaton 1987; Taipina et al. 2009). Resurreccion and Heaton (1987) developed an objective texture method for distinguishing differences between early and traditionally harvested pecans. The authors conducted a puncture test and calculated the shear force required to cut the pecans halves using a blunt blade attachment. The proposed method did not reflect situations where pecans experience mechanical deformation during handling and storage. to address issues with sample non-the uniformity criteria, Ocòn et al. (1995) proposed a method where samples are prepared by driving a cork borer perpendicularly through the pecan kernel, and taking out

cylinders of uniform dimensions (Prabhakar et al. 2020). The core method is the most adopted textural analysis method for pecan texture (Surjadinata et al. 2001; Shult and Brusewitz 1998; Anzaldúa-Morales et al. 1999a). However, these researchers found very low correlation between sensory and instrumental analysis for texture determination, indicating a need for more accurate ways to determine textural attributes.

There are many reports of the effect of processing methods (roasting, drying, dehydration, etc.) on texture of walnuts (Kita and Figiel 2007), pistachio (Mohammadi Moghaddam et al. 2016; Farahnaky and Kamali 2015), macadamia (Domı et al. 2007; Tu et al. 2021), pecans (Zhang et al. 2019) and pecan shells (Littlefield et al. 2011). However, there are few studies investigating the effects of moisture migration (from kernels to environment and vice versa) on pecan kernel texture and tree-nuts in general, with and/or without use of commonly available packaging materials such as PE, PP, cellophane, etc. (Prabhakar et al. 2020). Furthermore, ability to predict changes in texture under a given set of conditions is valuable for the industry to maximize quality of kernels during storage, or to maximize shelf life once in a store for consumers. Probabilistic models can be used for to predict shelf life based on a specified set of conditions. However, there are no probabilistic model(s) available for predicting changes in pecan kernel texture with changing storage/distribution conditions.

The objectives of this research were to investigate changes in pecan kernel texture due to environmental conditions (RH and packaging type), and to develop a predictive model suitable to estimate change in texture of pecan kernels attributes as storage progressed under different environments.

2. Material & Methods

2.1 Pecan production, source of nutmeat and storage experiment

Three cultivars of pecan (*Carya illinoinensis* 'Stuart', 'Pawnee' and 'Desirable') were harvested from orchards located at the USDA-Agriculture Research Service (ARS) Fruit and Tree Nut Research Laboratory, Byron, Georgia (U.S.A.), (+ 32.6650 N, + 83.7419 W, elevation of ≈ 156 m, 240 d freeze-free growing period, annual precipitation of 118 cm). Orchards received standard tree management practice for the state of Georgia (Wells et al. 2019). The experiment was performed twice, with pecans harvested in November 2018 and December 2019, respectively. In each season, the pecans were processed within 1 week of harvesting. The harvested pecans were conditioned prior to shelling by immersing in 85°C water for 3 min, followed by drying at room temperature for 20–25 min and shelling via mechanical sheller (Modern Electronics, Mansfield, LA) (Forbus Jr and Senter 1976). After shelling, pecans were dried at 20°C and 45% RH overnight to a moisture content of 4–5% moisture content (AOAC 2016) and stored at − 20°C in a commercial freezer until use in the experiments. Information on the different grades of pecans has been provided by Prabhakar et al. (2022).

2.2 Experiment treatments

The pecans were stored in different RH conditions. The desired RH was achieved by using 200 mL saturated salt solutions placed in a static humidity chamber (STC) consisting of a 1-L glass jar with a rubber gasket to seal the lid. More detailed information on construction of the STCs has been provided by Prabhakar et al. (2022). The saturated salt solutions included magnesium chloride (30–32% RH), magnesium nitrate (50–52% RH), sodium chloride (75% RH), ammonium sulfate (80–81% RH) and potassium nitrate (89–93% RH) (Certified ACS, Fisher

Chemical, Waltham, MA) (Rockland 1960). For the sake of simplicity, the RHs will be denoted as 30%, 50%, 75% 80%, and 90%, respectively. The STCs containing pecans from three cultivars were placed in temperature-controlled chambers at 20, 30 and 40°C. For each temperature × humidity treatment (n = 2 jars for each combination), 50 g of whole pecans (25 to 40 pecan halves) were placed in a nylon bag suspended above the saturated solutions on an aluminum mesh disc in the STC. To simulate a real storage environment and corresponding air composition, the jars were opened periodically (every 1–2 weeks) for 30 s to allow fresh air into the container. In addition, the pecan kernels were packaged in materials typically available to small scale pecan producers and packers. The packaging materials used were low-density polypropylene (LDPE), polypropylene (PP), polyethylene (PE) and laminates with an aluminum layer. The packaged samples of pecan kernels were stored at 58% and 80% RH and at a temperature range similar to the pecan kernels in the STCs. The kernel samples were drawn at predetermined intervals based on previous reports of pecan quality changes in the literature (Blackmon 1932; Brison 1945; Kays 1979b; Magnuson et al. 2015; Mexis et al. 2009; Senter and Wilson 1983). The storage time ranged from 15 to 450 days, depending on the treatment. The mold growth assessment was performed visually and samples with mold growth were discarded. In addition, some pecans were placed in packages available to pecan producers and packers viz. low-density polypropylene (LDPE, 50–54 μm), polypropylene (PP, 45–50 μm), polyethylene (PE, 53–57 μm) and metallic laminates (ML, 105–110 μm). The packages were obtained from *OpenTip.com* and sealed using American International Electric Heat Impulse sealer (City of Industry, CA) The packaged samples were stored at 58% and 80% RH at temperature range similar to the unpacked STC pecans. The samples were drawn at predetermined intervals based on previous reports of pecan quality changes. The storage time ranged from 15 to 450 days, depending on the treatment. Any mold growth was assessed visually and samples with mold growth were discarded.

2.3 Sample preparation

2.3.1 Pecan core method

The samples were prepared according to method published by Ocòn et al. (1995). To obtain uniform samples for texture analysis, a cork borer was inserted perpendicularly through the pecan kernels to obtain cylindrical specimens 3mm in diameter and 5mm in length. The cored samples were analyzed using single compression method as the cores were not strong enough to sustain second compression. The textural attributes studied included first peak (hardness) and area under curve (AUC).

2.3.2 Intact pecan-halve method

The intact pecan kernels or halves were compressed under a flat probe for texture profile analysis (TPA, double compression). The textural attributes studied included fracturability, hardness, cohesiveness, springiness, and chewiness. In TPA, these textural attributes can be defined as follow; *fracturability* is the first break in the curve force vs extension/time curve, *hardness* is the highest force on the first compression cycle (always followed by fracturability), *cohesiveness* is the ratio of (positive) first and second force areas, *springiness* is the recovery distance between the end of first and start of second compression, chewiness is product of hardness x cohesiveness x springiness and can be defined as force required to chew the food product. In addition to these textural attributes, fracturability/hardness ratio (F/H, referred to as *rift ratio* from this point onwards) was also studied.

The cored and intact pecan samples were compressed up to 50% of strain under a 55mm compression probe using a TA.XT2 texture analyzer (Texture Technologies Corporation, Scarsdale, New York/Stable Micro Systems, Haslemere, Surrey, UK). The test parameters were as follow: pre-test speed – 1mm/s, test speed – 5 mm/s, post-test speed – 5 mm/s. A total of 10 measurements were taken from unpacked and packed pecans. The packed pecans were only analyzed using intact pecan method.

2.4 Predictive model

A three-parameter logistic (3PL) model, a type of sigmoid model, was used to predict the changes in pecan textural attributes over time. The 3PL model is a type of logistic model prominently used in immunoassays research (Herman et al. 2008) such as ELISA, microbial growth prediction (Fujikawa 2010), dose-response relationships (Andrade-Mogrovejo et al. 2022; ElHarouni et al. 2022; Carøe et al. 2018), and geological phenomena (Chen et al. 2019; Joshuva et al. 2019). The parameters give unique information such as maximum value to response achieved (asymptote), growth rate or slope, and the value of a predictor variable for median response (inflection point) (Fig. 1). The 3PL model equation can be denoted as:

$\hat{y} = \frac{c}{1+exp^{(-ax+ab)}}$ - (1)

Where a is the growth rate or slope, b is the inflection point, c is the asymptote and ŷ is the predicted response. The logistic model was built using a non-linear function in JMP®, Version 16 Pro (SAS Institute Inc., Cary, NC).

2.5 Experimental design and statistical analyses

The design of the experiment was a generalized randomized complete block design (GRCBD) where storage days and RH were experimental factors and cultivars were treated as a block. The whole experiment was repeated twice, indicating replication within each block. To avoid complexity and simplify the interpretation of the statistical output, interactions of block and treatment with other factors were omitted. The preliminary experiment indicated no significant effect of temperature on any of textural attribute of pecans ($p > 0.05$). Thus, readings from all temperature conditions were pooled for the analysis. The outliers were determined and removed using the “jacknife distance” method.

$$J_i = \sqrt{\frac{(n-2)\,n^2}{(n-1)^3} \times \frac{{M_i}^2}{1 - \frac{nM_i^2}{(n-1)^2}}}$$

2

Where n = number of observations, p = number of variables and M_i = Mahalanobis distance for the ith observation. The upper critical line (UCL) is the limit beyond which the J_i values are considered outliers and could be omitted from the analysis. Penny (1996) has provided a detailed account on calculating UCL for jacknife analysis. Subsequently, a mixed model analysis was performed on refined data. The experiment data was normally distributed, and the model residual plots did not indicate heteroskedasticity. The model fixed effects were RH and storage days whereas cultivar was considered a random effect. The storage days were nested within RH. The dependent variables for core method were hardness and AUC and for intact pecan method were fracturability, hardness, F/H, cohesiveness, chewiness and springiness. The 3PL model parameters were

statistically analyzed using One-way ANOVA. The main effects and their interactions (where applicable) were studied and their interactions with blocks were omitted. The fit for the statistical models (mixed (mixed model, ANOVA and 3PL) were assessed based on the adjusted coefficient of determination (adj. R^2). A Tukey's HSD post hoc test (confidence level, = 95%) was performed to explore differences among means for the different treatments. Multivariate correlation analysis was conducted to understand relationship among multiple dependent factors (textural attributes). All statistical analyses were performed using JMP®, Version 16 Pro (SAS Institute Inc., Cary, NC).

3. Results

3.1 Pecan core method

The change in AUC and hardness with storage time is illustrated in Fig. 2. The total work done significantly decreased with increase in RH and storage time (Table 1). The change in AUC during storage was small among pecans stored between 30 to 75% RH. The change in AUC was greatest for pecans kept at 80% RH. The hardness of the cored pecans was significantly affected by RH and storage time (Table 1). The hardness value increased with greater RH. At higher humidity conditions (≧ 75%), the hardness increased as storage progressed. The pecans stored at and below 50% experienced a significant decrease in hardness with storage time (Fig. 2). Even though the goodness of fit for AUC and hardness was low (0.15 and 0.24, respectively), statistical significance of main effects and interactions do indicate that the independent variables were affected by the predictors. The detailed tabulation of change in AUC and hardness with respect to RH and storage days can be found in supplementary table S1 and S2.

Table 1
Mixed model analysis and means for the effects of relative humidity (RH) and storage duration (SD, days) on textural attributes of nonpackaged pecans (cores or kernels). For the analysis SD was nested within RH (SD[RH]).

Method	Variable	Source	F Ratio	Prob > F	Adj. R^2 (Model)	% RH	Least Sq mean[a]		95% Confidence limits
Core method	**Total area under curve**	**SD[RH]**	2.88	0.02	0.14	**30**	18.87	A	16.97–18.64
		RH	8.66	< .0001		**50**	19.88	A	19.25–20.54
						75	17.74	B	17.17–18.30
						80	18.99	A	18.30–19.70
	Hardness (N[b])	**SD [RH]**	7.80	< .0001	0.24	**30**	15.81	C	15.21–16.41
		RH	10.06	< .0001		**50**	16.06	C	15.59–16.52
						70	17.15	B	16.75–17.55
						80	19.79	A	17.9-21.68
Intact Pecan-halve method	**Fracturability (N[b])**	**SD [RH]**	123.34	< .0001	0.67	**30**	33.51	C	26.94–40.09
		RH	349.88	< .0001		**50**	45.40	C	39.03–51.78
						70	167.92	B	160.98-174.85
						80	604.37	A	511.88-696.86
	Cohesiveness	**SD [RH]**	5.64	0.0002	0.12	**30**	0.25	B	0.24–0.26
		RH	27.94	< .0001		**50**	0.26	B	0.25–0.27
						70	0.30	A	0.29–0.31
						80	0.34	C	0.32–0.36
	Springiness	**SD [RH]**	22.20	< .0001	0.27	**30**	0.39	D	0.38–0.4
		RH	63.49	< .0001		**50**	0.41	C	0.4–0.42

[a]Different letters for the means in each RH group indicate significant difference between the means based on Tukey's HSD (α = 0.05).

[b]N-newton to indicate force.

Method	Variable	Source	F Ratio	Prob > F	Adj. R^2 (Model)	% RH	Least Sq mean[a]		95% Confidence limits
						70	0.47	B	0.46–0.48
						80	0.73	A	0.6–0.85
	Chewiness (N[b])	**SD [RH]**	11.64	< .0001	0.17	**30**	19.43	B	17.87-21
		RH	37.52	< .0001		**50**	21.18	B	19.67–22.7
						70	30.82	A	29.18–32.47
						80	35.62	C	33.65–37.59
	F/H	**SD [RH]**	161.04	< .0001	0.72	**30**	0.18	D	0.15–0.21
		RH	471.98	< .0001		**50**	0.25	C	0.22–0.27
						70	0.80	B	0.78–0.83
						80	1.00	A	0.94–1.06

[a]Different letters for the means in each RH group indicate significant difference between the means based on Tukey's HSD (α = 0.05).

[b]N-newton to indicate force.

3.2 Intact pecan-halve method

3.2.1 Fracturability and Hardness

The Fracturability of pecans was most significantly affected by RH as substantiated by goodness of fit (R^2 – 0.67). The fracturability could be defined as the first break point on TPA curve corresponding to a force value. Thus, lower force value corresponds to high fracturability as it signals early fracturability. For unpacked pecans, the increase in RH caused the fracturability value to increase indicating the pecans were losing brittleness (Table 1). The change in fracturability increased drastically as RH increased > 50% (Fig. 3).

For pecans stored in different package materials, the fracturability significantly decreased with change in RH, storage period and packaging material (Table 2). The loss of fracturability was minimum in pecans stored in metallic laminates and maximum in pecans stored in LDPE packages. The loss in fracturability was intermediate in PE and PP packages but the value was in proximity to that for samples in LDPE (Fig. 4A, Supplementary Table S3). The overall fracturability of pecans stored in LDPE, PP and PE at 58% RH was significantly lower than those

stored at 80% RH. The impact of environment RH was negligible for samples stored in metallic laminates. A detailed tabulation of changes in fracturability with RH conditions can be found in supplementary table S3. Unlike results measured using the core method, the TPA of packed and unpacked pecans did not reveal a definite pattern in terms of change in hardness across storage days and RH.

Table 2

Mixed model analysis and means for the effects of relative humidity (RH) and storage duration (SD, days) on textural attributes of pecan kernels in different types of packaging materials. The least mean square indicate the textural attribute for the pecan kernels stored in different packaging at 80% RH. For the analysis SD was nested within RH (SD[RH]).

Variable	Source	F Ratio	Prob > F	Adj. R^2 (Model)	Package[a]	Least sq mean[b]		95% Confidence limits
Fracturability (N[c])	**Package**	43.19	< 0.01	0.70	**AL**	30.70	C	14.18–47.21
	SD[RH]	83.32	< 0.01		**LDPE**	147.43	A	135.29-159.57
	RH	352.84	< 0.01		**PEN**	119.49	B	108.28-130.71
	Package*RH	39.44	< 0.01		**PP**	118.71	B	109.06-128.37
	SD*Package[RH]	8.01	< 0.01					
Cohesiveness	**Package**	5.98	< 0.01	0.17	**AL**	0.30	B	0.28–0.31
	SD[RH]	8.36	< 0.01		**LDPE**	0.33	A	0.32–0.34
	RH	23.501	< 0.01		**PEN**	0.31	B	0.3–0.32
	Package*RH	10.12	< 0.01		**PP**	0.31	B	0.3–0.32
	SD*Package[RH]	2.03	0.06					
Springiness	**Package**	0.68	0.567	0.11	**AL**	0.46	A	0.44–0.49
	SD[RH]	9.92	< 0.01		**LDPE**	0.45	A	0.44–0.47
	RH	9.15	< 0.01		**PP**	0.47	A	0.45–0.48
	Package*RH	1.30	0.27		**PEN**	0.46	A	0.45–0.47
	SD*Package[RH]	0.63	0.71					

[a] Packaging materials are LDPE = low density polypropylene, PE = polyethylene, PP = polypropylene, and ML = metal laminate.

[b]Different letters for the means in each packaging group indicate significant difference between the means based on Tukey's HSD (α = 0.05).

[c]N-newton to indicate force.

Variable	Source	F Ratio	Prob > F	Adj. R^2 (Model)	Package[a]	Least sq mean[b]		95% Confidence limits
Chewiness (N[b])	**Package**	1.38	0.25	0.14	**AL**	31.83	A	28.03–35.64
	SD[RH]	3.61	0.03		**LDPE**	34.51	A	31.98–37.05
	RH	11.91	< 0.01		**PEN**	31.99	A	29.52–34.47
	Package*RH	5.12	< 0.01		**PP**	34.79	A	32.63–36.95
	SD*Package[RH]	1.15	0.33					
F/H	**Package**	41.98	< 0.01	0.71	**AL**	0.13	C	0.07–0.2
	SD[RH]	82.78	< 0.01		**LDPE**	0.60	A	0.55–0.65
	RH	347.54	< 0.01		**PEN**	0.53	AB	0.48–0.57
	Package*RH	33.69	< 0.01		**PP**	0.49	B	0.45–0.53
	SD*Package[RH]	8.03	< 0.01					

[a] Packaging materials are LDPE = low density polypropylene, PE = polyethylene, PP = polypropylene, and ML = metal laminate.

[b]Different letters for the means in each packaging group indicate significant difference between the means based on Tukey's HSD (α = 0.05).

[c]N-newton to indicate force.

3.2.2 Rift ratio (F/H)

There were significant effects of RH and storage duration on F/H (Table 1). F/H was greatest at 80% RH and least at 30% RH, and the coefficient of determination of mixed model analysis ($R^2 = 0.72$) further indicated a relationship between RH and storage duration on F/H. There were significant effects of packaging on F/H (Table 2). The LDPE experienced maximum increase in *rift* ratio followed by PE, PP, and metallic laminate (ML). The difference in *rift* ratio of LDPE, PE, PP and ML stored at 58% RH were not significant. This ratio was further explored for use in predictive modeling to estimate loss of brittleness during storage. The 3PL model was employed to predict the change in *rift* ratio with storage time. The growth rate (change in response units per day) indicates an increase in RIFT ratio with time, the inflection point is defined as the time taken to lose half of the

fracturability value, and the asymptote refers to maximum *rift* ratio retained during storage. In addition, the time taken for pecan to lose all fracturability at constant RH condition can also be determined. During storage, it was observed that packaged and unpackaged pecans stored at 58% RH or below did not experience significant loss of fracturability. Thus, predictive model was made only for pecans stored at 75% or above.

Values of the logistic parameters for packaged and unpackaged pecans are tabulated in Tables 3 and 4. the growth rate and inflection point for unpackaged pecans significantly increased with increase in RH. From the *rift* point in Table 3, it was determined that unpackaged pecans stored at 75%, 80% and 90% lost half of the initial *rift* ratio at ~ 115 days, 3 days, and 0.15 days (~ 4 h), respectively. By multiplying the inflection point by 2, the time required at constant RH for complete loss of fracturability (*rift* ratio = 1.0) was calculated as 230 days for 75% RH, 6 days for 80% RH and 0.30 days (8 h) for 90% RH. The growth rate and inflection point for RH between 75–90% can be determined by following equations:

Table 3
The parameters for three-parameter logistic model of the rift (fracturability/hardness) ratio for nonpackaged pecan kernels stored at 75,80 and 90% relative hunididty (RH)

Parameter	RH	Least sq mean		95% Confidence limits	R^2
Growth rate (a)	75	1.23	C	0.34–2.12	0.90
	80	5.35	B	4.46–6.24	0.97
	90	11.89	A	11-12.78	0.63
Inflection point (b)	75	115.44	A	100.7-130.2	0.90
	80	3.17	B	1.60–4.74	0.97
	90	0.15	B	0.06–0.24	0.63
Asymptote (c)	75	0.99	A	0.88–1.09	0.90
	80	0.92	A	0.82–1.03	0.97
	90	0.95	A	0.85–1.05	0.63

[a]Different letters for each parameter group indicate significant difference between the means based on Tukey's HSD (α = 0.05).

Table 4
The parameters for 3PL model for packed pecans stored in different packaging materials at 80% RH. Pecan stored below 80% RH did not experience significant change in texture. The same is true for pecans stored in ML.

Parameter	Package[a]	Least Sq Mean	95% Confidence limits	R^2
Growth rate (a)	LDPE	0.06	0.06–0.06	0.88
	PE	0.02	0.02–0.02	0.98
	PP	0.03	0.03–0.03	0.98
Inflection point (b)	LDPE	26.25	26.23–26.28	0.88
	PE	78.05	75.01–81.1	0.98
	PP	56.99	54.17–59.81	0.98
Asymptote (c)	LDPE	1.00	1–1	0.88
	PE	1.01	1-1.03	0.98
	PP	1.01	0.99–1.02	0.98

[a]LDPE = low density polypropylene, PE = polyethylene, and PP = polypropylene.

$$Growthrate\,(unpackedpecans) = -51.24 + 0.70 * RH$$

3

$$Inflectionpoint\,(unpackedpecans) = 2.72^{(36.16-0.43*RH)}$$

4

Since no definite trend was observed for the asymptote, based on the value for nonpackaged pecans can be assumed to be 0.95 by taking average of asymptote

Table 4 contains the growth rate, inflection point and asymptote values for packaged pecans stored at 80% RH. The growth rate was highest in LDPE, followed by PP and PE, indicating a higher water absorption rate in the LDPE package. The pecans stored in LDPE, PP and PE at 80% RH lost half of initial the fracturability at ~ 26 days, 57 days, and 78 days, respectively. The number of days to reach complete loss of fracturability at constant RH was calculated as: LDPE – 52 days, PP – 114 days, and PE – 156 days. The *rift* ratio for packaged pecans stored in 58% RH remained unchanged during storage. Under extreme humidity conditions, the packages provided a decent barrier against moisture transfer, delaying loss of fracturability as compared to pecans with no package, where the fracturability loss occurred in matter of hours. The following equation predicts the *rift* ratio at a specific storage day for pecans packaged in abovementioned materials:

$$F/H = 0.35 + PC + 0.0026 * Storagedays$$

5

Where PC is a ‘package constant’ with values as follows: LDPE = 0.084, PE= -0.042, PP = -0.041. The water vapor transmission rate corresponding to LDPE, PE and PP are 1.30, 0.41, and 0.50 g.mL/24 hr.100 in^2, respectively

(38°C, 50 to 100% RH) (Tock 1983).

3.2.3 Cohesiveness

The cohesiveness of unpackaged and packaged pecan kernels was significantly affected by RH and storage duration, with adjusted R^2 of 0.12 and 0.17, respectively (Table 1). The cohesiveness of unpackaged pecans significantly increased with an increase in RH. The unpackaged pecans stored at 75% RH or above experienced a sharp increase in cohesiveness (Fig. 3). The pecans stored in metallic laminates had a minimum cohesiveness, however, and was not significantly different from PP and PE (Table 2). LDPE packaged pecans experienced the greatest change in cohesiveness among all the packages.

3.2.4 Springiness

The springiness of unpackaged pecans was significantly affected by RH and storage time (Table 1). The change in RH and packaging material had little to no impact on change in the springiness of packaged (< 58%) and unpackaged pecans (< 50%) (Table 2). The springiness increased significantly at RH levels higher than 75%. Despite exhibiting significant effect, the adjusted R^2 for unpackaged and packed pecans were 0.27 and 0.11, respectively, indicating limited ability of predictor variables to estimate textural attributes.

3.2.5 Chewiness

Unlike springiness, the chewiness of unpackaged and packaged pecans significantly increased with increase in RH. The unpackaged pecans stored at or below 50% was significantly less chewy as compared with pecans stored at 75% or higher. The packaging material had no significant impact on chewiness of pecans, indicating chewiness change was similar across packaged pecans (Tables 1 and 2, Supplementary table S3). As with cohesiveness and springiness, chewiness had low adjusted R^2 of 0.17 and 0.14 for unpackaged and packaged pecans, respectively.

Discussion

The fracturing of pecans is Ocòn et al. (1995)the first sensation that a consumer comes across when ingesting pecans. Given their irregular structure, one should expect variations in observations of instrumental texture analysis. The shape and size of pecan kernel is influenced by a number of factors such as sunlight exposure, cultivar, or damage by insects and rodents(Sparks 1993). To address this problem, Ocòn et al. (1995) suggested cutting cores out of pecans and cutting the ends of cores to form a cylinder with standardized dimensions. This sample preparation technique sacrifices important key textural attributes, specifically fracturability, due to removal of testa and absence of numerous fracture points. The role of pecan testa in fracturability of pecans will be explained in detail later in the manuscript. Another issue we experienced is that the pecan kernels kept at low RH (≦ 50%) started crumbling as the cork borer was inserted, making it difficult to maintain intact samples. As Ocòn et al. (1995) acknowledged by this sample preparation technique is also time consuming, making it an inconvenient protocol to follow in an extensive storage (Surjadinata et al. 2001; Anzaldúa-Morales et al. 1999a)study .

One issue with purely compressive tests is that each pecan has a unique overall size (within a range) and an undulating surface; thus, when a disk-shaped probe pushes through the sample it experiences differing forces based on both the material properties and the total cross-sectional area the probe is contacting. This influences

both the maximum measured force as well as the force experienced at the first fracture point if it exists. The reality of non-uniform samples has long hampered the ability to make precise measurements of texture attributes. One way researches have addressed this problem is to normalize any force-time data by the sample volume or weight. In this work we tested the hypothesis that normalizing the “fracturability”, that is the force at first break under compression, by the force experienced under full compression, often defined as the “hardness”, would help mitigate issues with sample variability. By taking the ratio of fracturability and hardness (*rift* ratio), the onset of fracturing could be compared across multiple pecan kernels, with varying mass and size, since the ratio would take into accounts maximum force experienced by the kernel during the test. The minimum value of the ratio could be 0, indicating a very brittle/crisp product (such as potato chips) while a maximum value of 1 would indicate no fracturability at all (such as chewy products) (Fig. 5). It was statistically determined that moisture was primarily responsible for changes in texture of pecan kernels stored under different temperature and RH conditions. To better understand the effect of moisture on textural attributes, the moisture migration from the environment to pecans (and vice versa) were tracked using % change in weight of pecan kernels. The multivariate analyses revealed that the *rift* ratio had a moderate positive correlation with change in weight of pecan kernels (Fig. 6). A sigmoid model could be used to predict F/H ratio with the change in % weight of pecans (Fig. 7). A sharp increase in F/H value can be detected as % weight of pecan kernels increases beyond 0.12%. The F/H ratio reaches 0.5 and 1.0 as change in weight reaches 0.25% and 0.50%, respectively. The logistic model parameter for F/H and % change in weight can be found in Supplementary Table S4.

The migration of moisture occurs due to a difference in water vapor pressure and water activity between product and surroundings. The food products with higher water activity leads to moisture loss from product to environment increases and vice versa (Afolabi 2014). At low moisture content, the plant cells become condensed and fragile, contributing to brittleness and easy fracturability (Capuano et al. 2018; Nikiforidis et al. 2013). Light micrographs revealed cells in the pecan testa are much more compacted than in cotyledon tissues (the white meat of the pecan kernel), contributing to brittleness of pecans (Fig. 7). In addition to that testa is present is the barrier between cotyledon and environment. Such an arrangement of cells make pecan testa more susceptible to moisture absorption (Rábago-Panduro et al. 2021). As moisture in the pecan increases, the compact cells start to swell and have greater cell wall flexibility, and increase in the intracellular distance, causing loss of fracturability. The increase in concentration of water molecules and presence of oleosomes (oil storage entities in pecans) contributes a cushioning effect against compressive forces and increases springiness (Nikiforidis 2019). As the moisture increased during storage, the pecans became more cohesive, indicating resistance towards breakdown. In addition, the overall kernel mass increased, which contributed to cohesiveness due to new hydrogen bond formation (Blahovec 2007). This would also indicate that the pecan with increased moisture levels required more work to chew, which was indeed indicated by an increase in chewiness.

For samples placed in any packaging, the fracturability loss was delayed by at least 8-fold (Tables 3 and 4). For packaged pecans exposed to 80% RH, kernels stored in LDPE experienced greater gain in moisture than those in PP, PE and laminate because of the greater water vapor transmission rate (Tock 1983). That is, the WVTR for LDPE was 1.30 g.mL/24 hr.100 in^2 (38°C, 50 to 100% RH), compared to values of 0.50 and 0.41 g.mL/24 hr.100 in^2 for PP and PE, respectively. Unlike LDPE, PE and PP, the metal laminate package was essentially impervious to moisture migration. As moisture could not enter, the textural attributes of pecans did not change significantly in the laminate packages, making them suitable packaging material for pecan kernels being handled in high RH environment.

Conclusion

Pecan texture is one of the important quality attributes of pecans, along with color and flavor, that is affected by storage, handling, and distribution conditions. This study investigated the two different texture methods, viz. the core method by Ocòn et al. (1995) and compression of intact pecans, for their versatility for studying the texture of pecans in differing packages and environmental conditions. The intact pecan method was found to be a reliable indicator of texture changes when analyzed using the *rift* ratio, that is by measuring the fracture force normalized by the maximum force experienced in compression. This helped reduce some of the variability of the data. Out of all the textural attributes, fracturability was found to be the most sensitive indicator in terms of reacting to environment moisture content. Pecans became less fracturable, and more cohesive, chewy and springy as moisture migrated from the environment into pecans. Fracturability was drastically reduced as the environment RH was > 50% for unpackaged pecans and > 58% for packaged pecans. It was found that the any kind of moisture barrier around pecans was able to deter texture change by at least 8 -fold. Pecans kept in LDPE experienced the greatest change in texture whereas pecans in metallic laminates did not change significantly during the storage. For the first time, a model and predictive equations were built to estimate changes in textural attributes of pecans along with meaningful model parameters such as growth rate and inflection point. Thus, our study explores the possibility of integration of stochastic models from other fields of STEM to food science research to build consequential models able to predict texture change in food.

Declarations

Conflict of Interest

The authors declare no competing interests.

Data Availability

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

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Author Contributions

Himanshu Prabhakar: Design of experiment, data collection, statistical analysis, writing. **Clive H. Bock**: statistical analysis, supervision, writing and editing. **William L. Kerr**: instrumental analysis, supervision, writing and editing. **Fanbin Kong**: Conceptualization and design of experimental, supervision, writing and editing.

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Figures

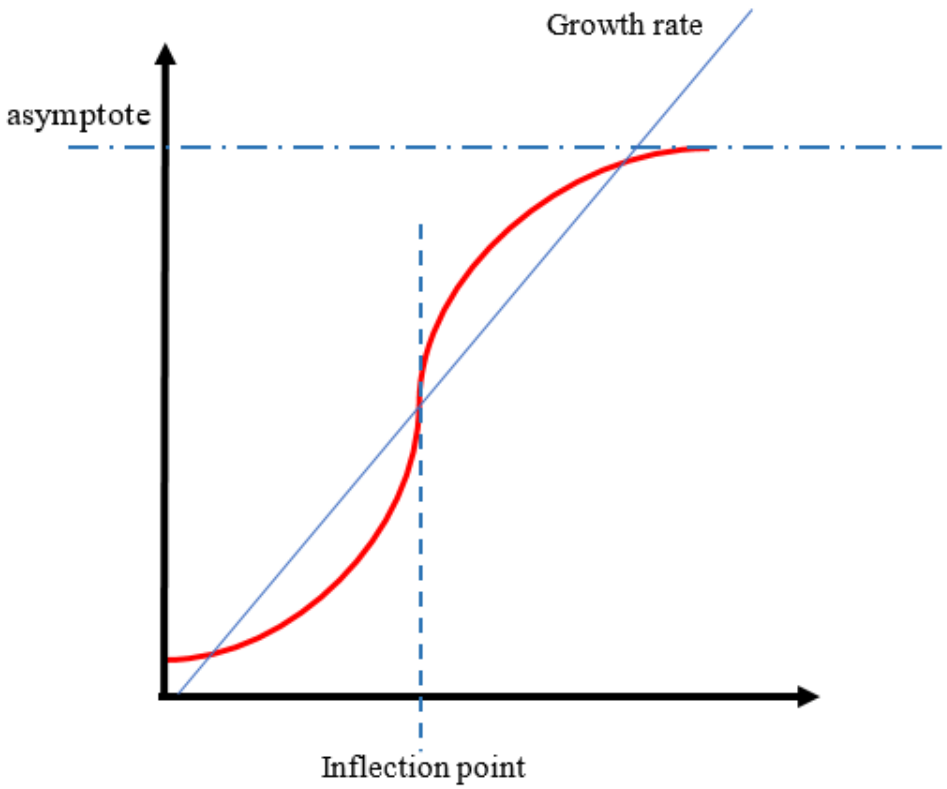

Figure 1

The three parameter logistic (3PL) model indicating the model parameters including the asymptote, inflection point and growth rate

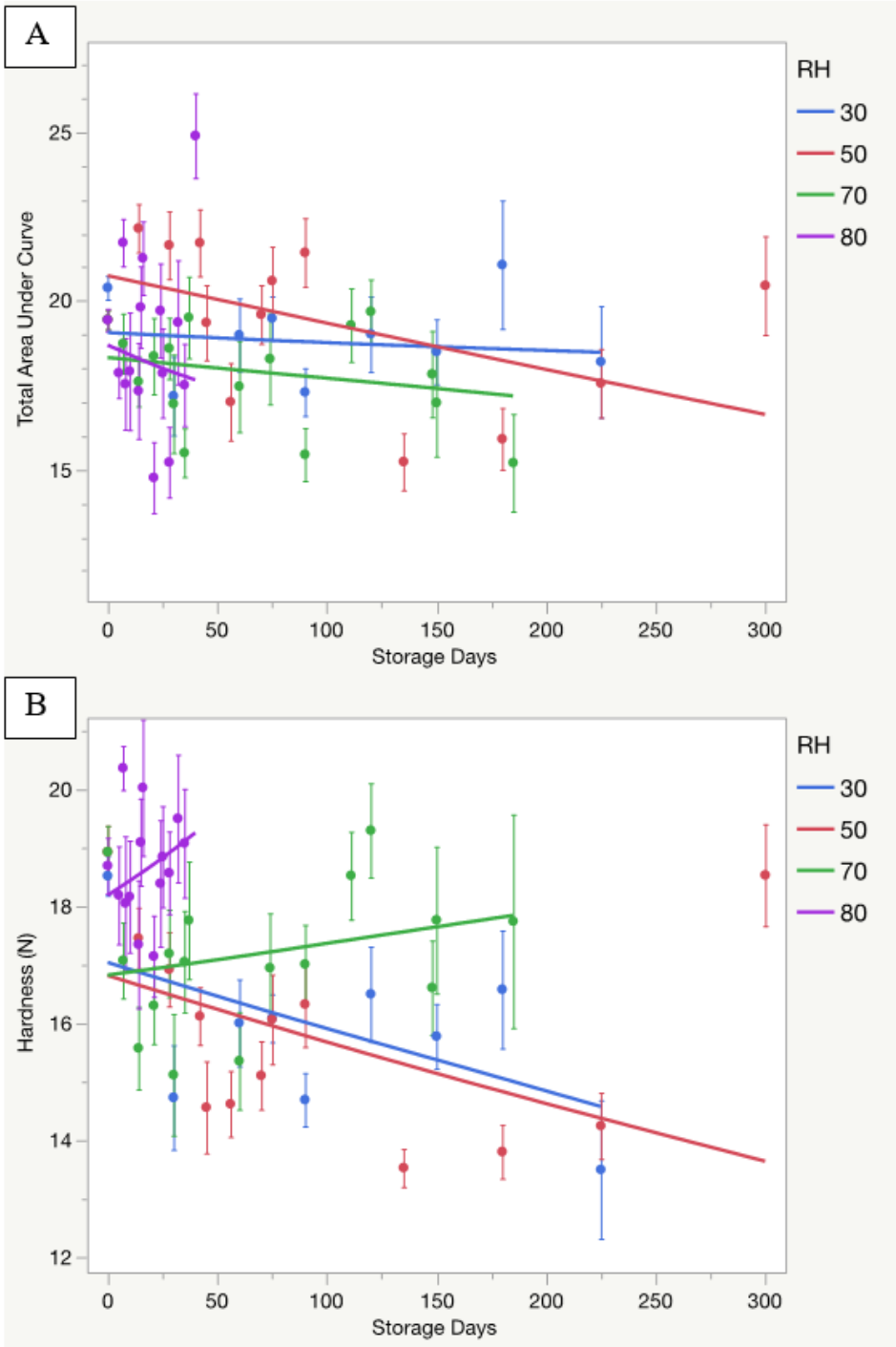

Figure 2

The change in area under curve (A), and hardness (B) of cores of unpackaged pecan kernels under different relative humidity conditions (RH) and storage durations.

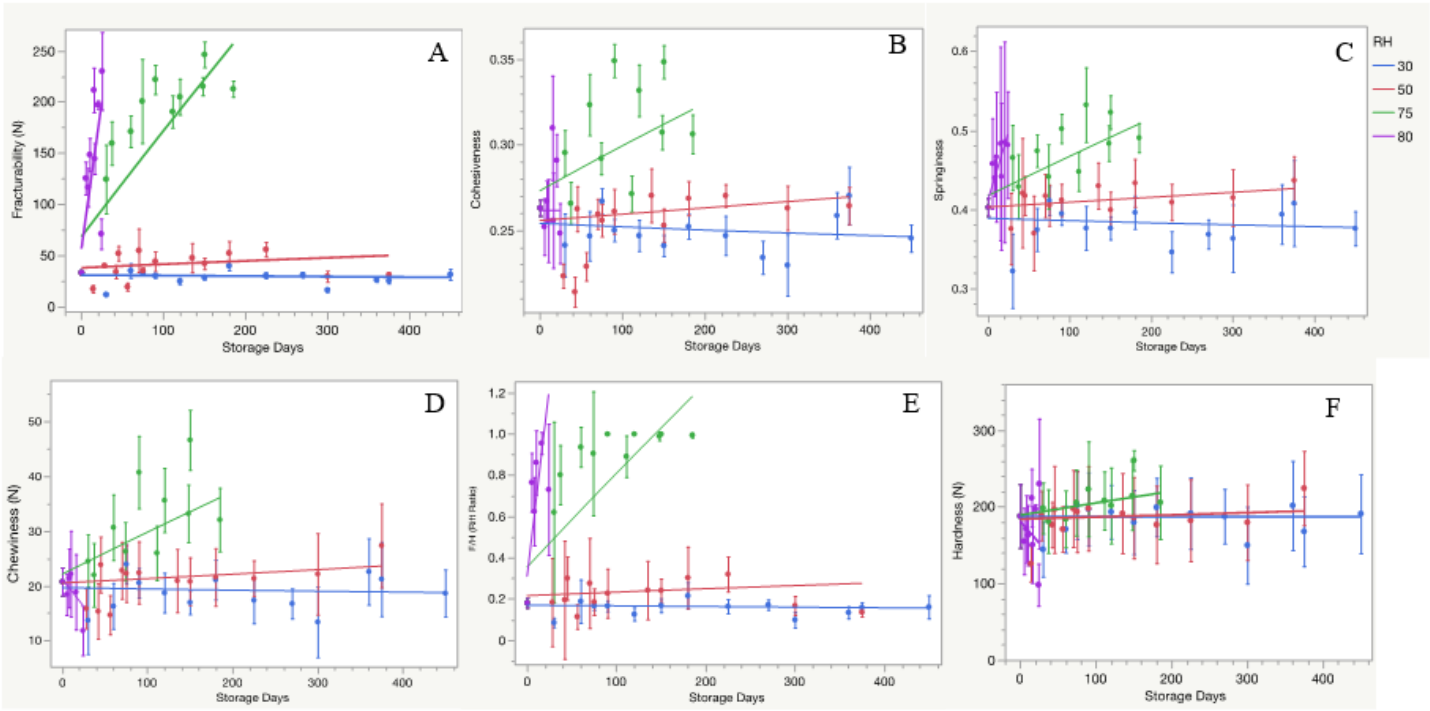

Figure 3

The change in textural attributes of nonpackaged pecan kernels under different relative humidities (RH) and storage duration. A. Fracturability, B. Cohesiveness, C. Springiness, D. Chewiness (N), E. F/H (rift) ratio and F. Hardness (N)

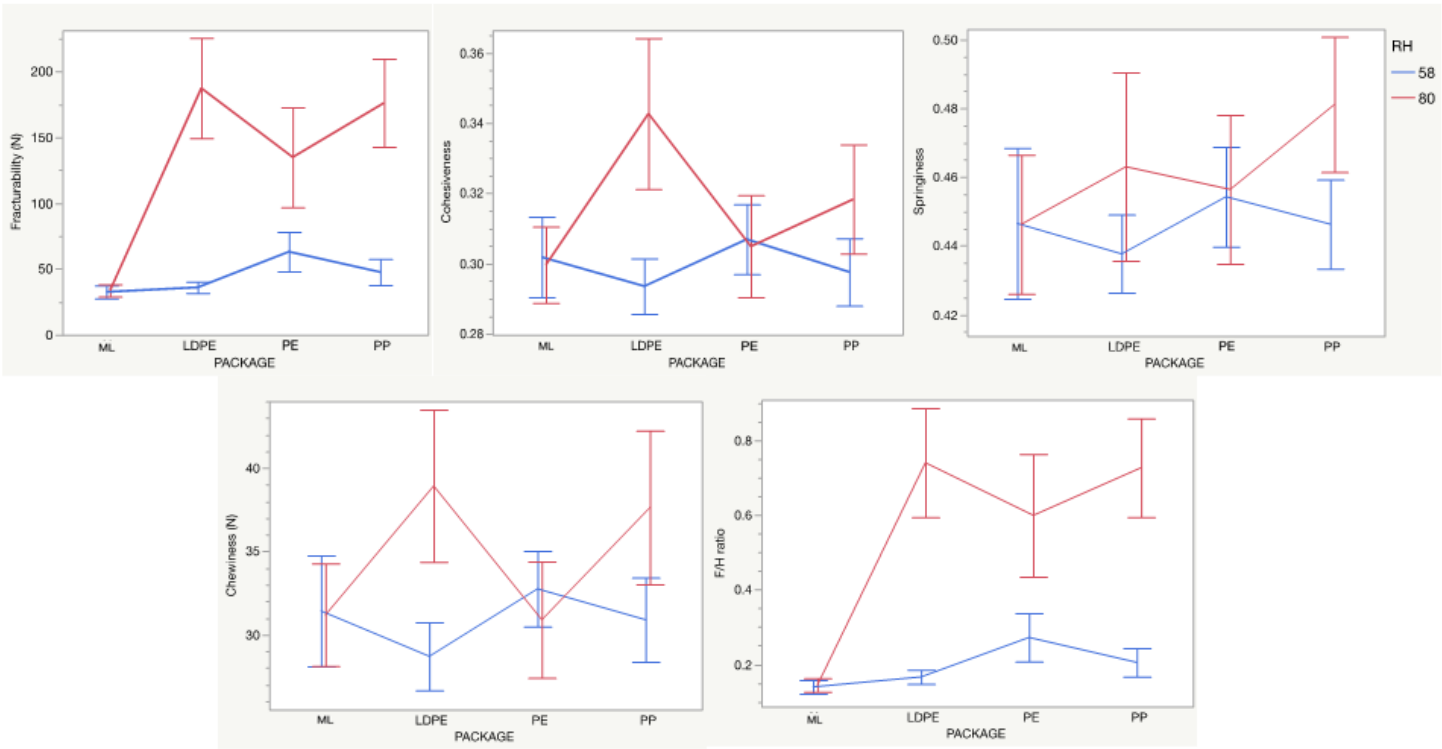

Figure 4

The interaction plots for change in textural attributes of pecan kernels packaged in different materials and under different relative humidities (RH) and storage durations. A. Fracturability, B. Cohesiveness, C. Springiness, D.Chewiness (N), and F. F/H (rift) ratio . Packaging materials are LDPE = low density polypropylene, PE = pol yethylene, PP = polypropylene, and ML = metal laminate.

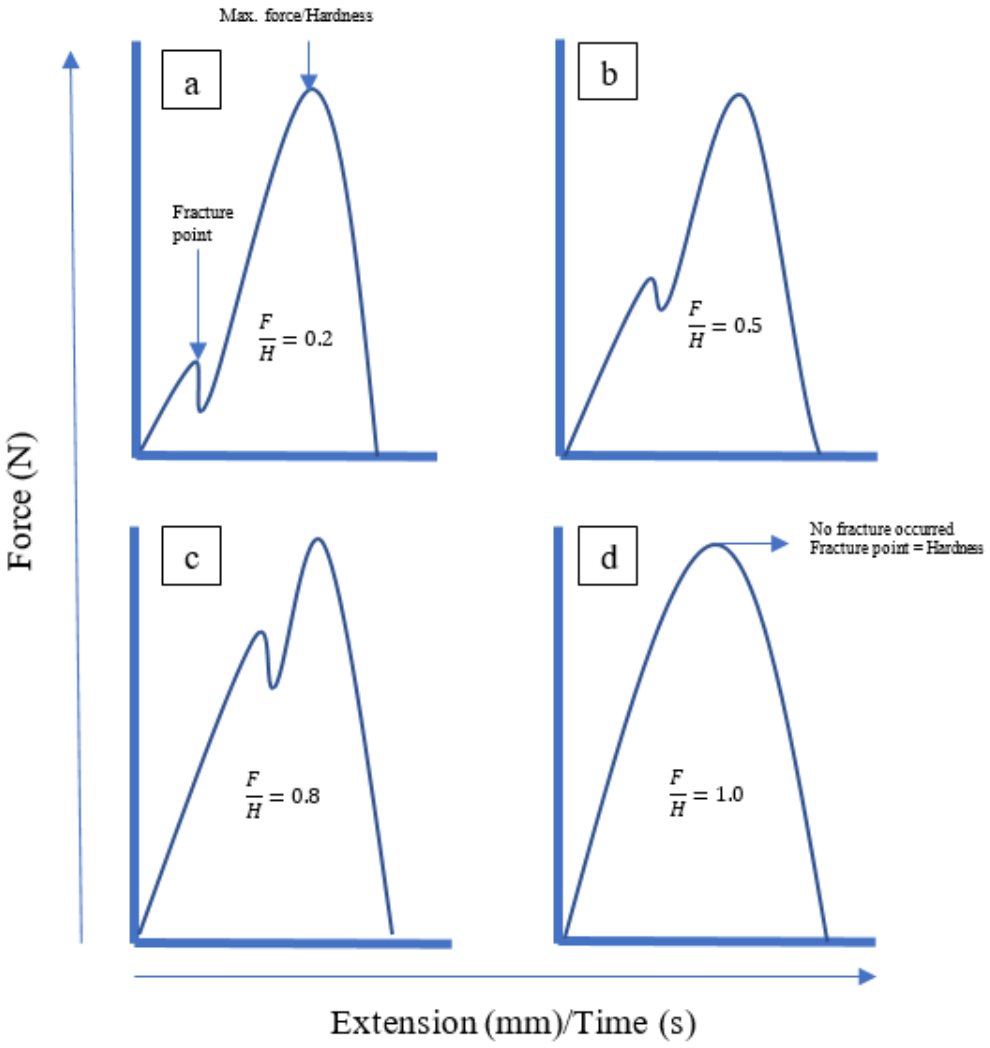

Figure 5

The change in the rift (F/H) ratio with constant hardness (N) during first compression. Graphs (a) to (d) represent with brittle/crisp texture to spongy/soft texture, respectively.

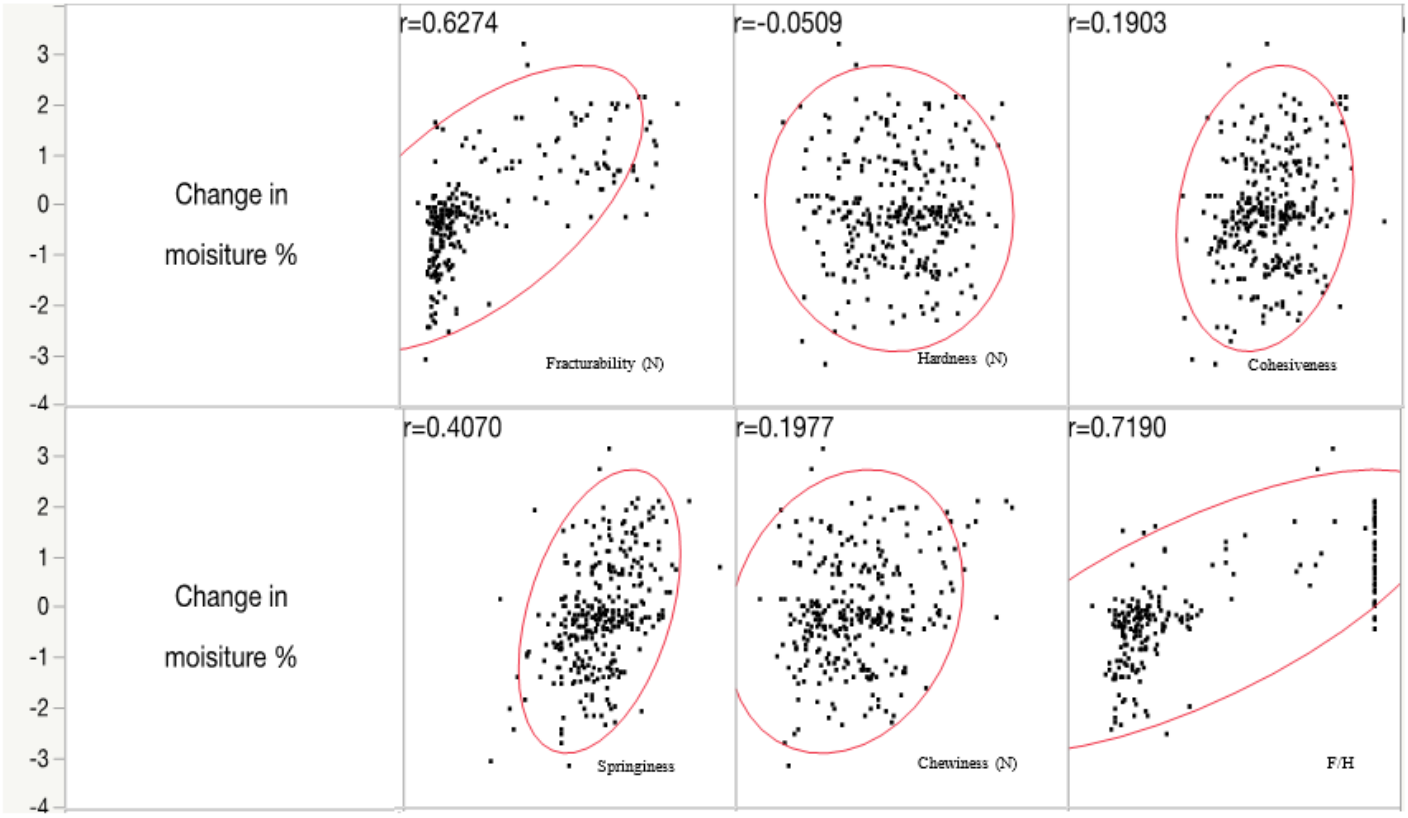

Figure 6

Correlation (r) between the percent change in weight and textural attributes of pecan kernels

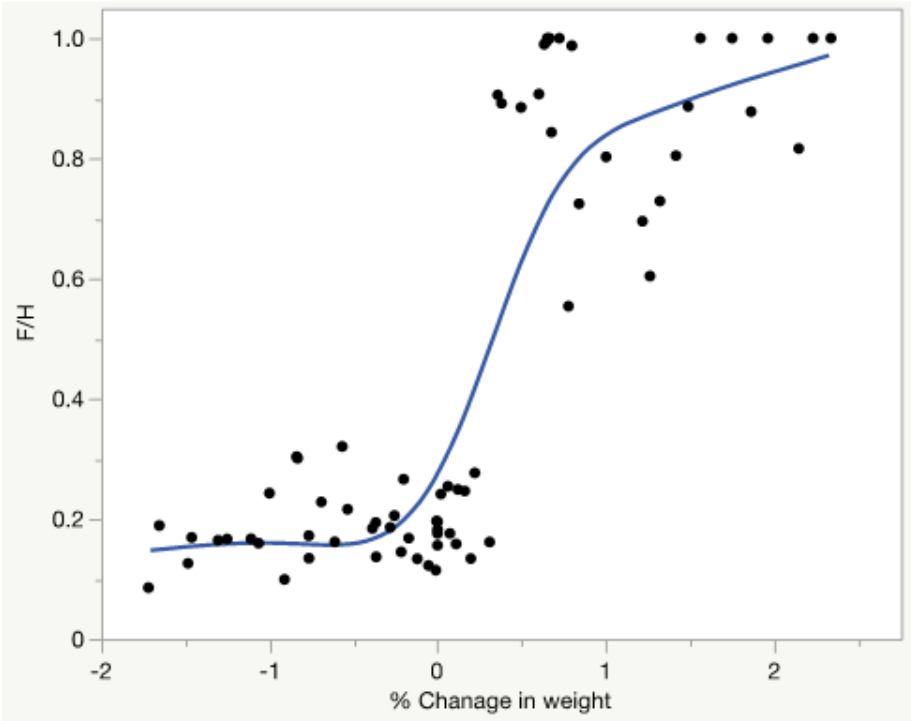

Figure 7

The sigmoid relationship between the rift (fracturability/hardness) ratio and the percent change in weight of unpacked pecan kernels stored under different RH (30 to 90%) primarily due to moisture (R^2 – 0.73)

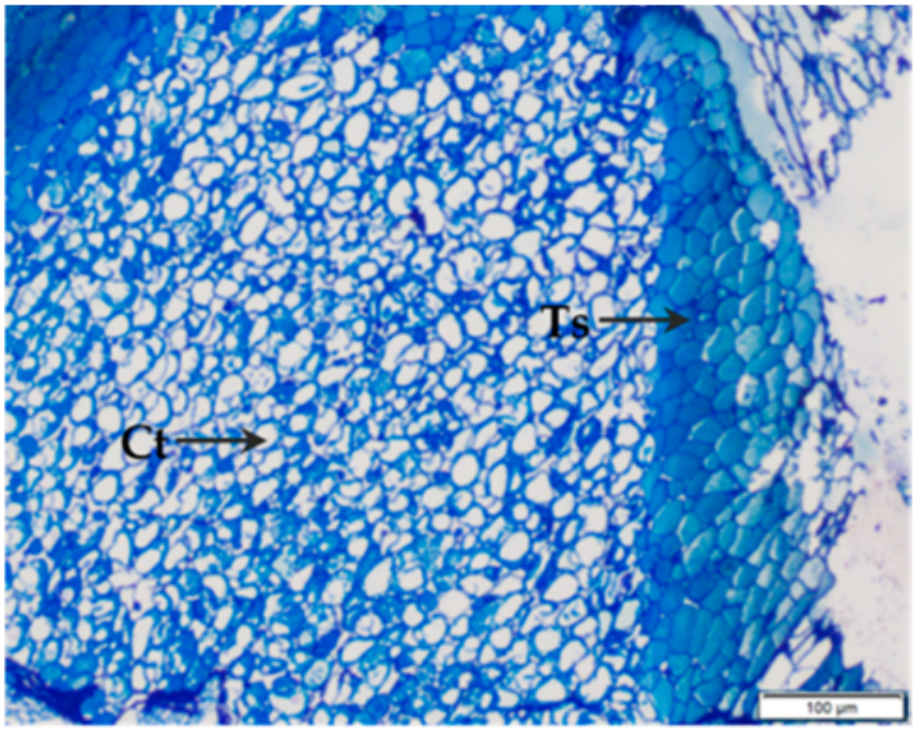

Figure 8

Light microscopy micrographs of the testa (Ts) and cotyledon tissue (Ct) of dry pecan nuts, adapted from Rábago-Panduro, Morales-de la Peña, Romero-Fabregat, Martín-Belloso, and Welti-Chanes (2021)

Supplementary Files

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- SupplementaryTableCB.docx