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Texture analysis of Supercritical Fluid Extrusion (SCFX) Products and Comparison with Traditional Puffed Extrusion

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ABSTRACT

The compressive stress-strain sigmoidal relationship of extruded products is commonly examined to quantify the qualities associated with human sensory perception. The hardness of extruded products can be compared by examining the stress for multiple products at a given strain value. Brittleness—also called crunchiness, or crumbliness—can be evaluated by examining the jaggedness of the normalized stress-strain curve for a given product. In this experiment, fourteen extruded, SCFX products made in the Rizvi Lab (Department of Food Science, Cornell University) were analyzed and compared to six commercially available extruded, puffed products. There were three primary objectives in this experiment. The first objective is to analyze the textural characteristics and compare SCFX products with commercial samples. The next objective is to test the applicability of this sigmoid stress-strain model to SCFX product. Finally, the overall aim is to ensure that SCFX product will meet consumer demands. According to the measurements collected in this experiment, the extruded, puffed products produced by the Rizvi Lab do exhibit textural characteristics within the same range as the commercial products tested. The sigmoidal stress-strain model was found to be appropriate for 8 of the 14 lab products and 3 of the six commercial products; suggesting that this model is useful, but not globally applicable. The data suggests that the Rizvi Lab's extruded, puffed products will meet consumer expectations for hardness and crunchiness.

Key words : Extrusion, SCFX, texture analysis

INTRODUCTION

Traditional extrusion technology is now widely used in manufacturing a diverse range of food products including pasta, snacks, breakfast cereals, confectionary, texturized meat substitutes, infant food formulations, precooked beverage powders, and extruded crispbread (Rizvi *et al.,* 1992). Supercritical fluid extrusion (SCFX) is a novel process combining extrusion and supercritical fluid technologies. SCFX utilizes supercritical carbon dioxide (SC−CO2) as a viscosity-lowering plasticizer and expansion/foaming agent (Rizvi *et al.,*1992). Traditional

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extrusion uses steam expansion as the driver for expanding or 'puffing' the extruded product; SCFX can operate below steam temperatures (Rizvi *et al.,*1992). An important advantage SCFX has over traditional extrusion is that it allows for simultaneous expansion, flavoring, and reduction of viscosity (Rizvi *et al.,*1992).

The textural characteristics of traditional extrusion products were analyzed and compared to SCFX products to determine what SCFX conditions generate product which texture profiles are consistent with commercial extruded products. Various researchers have put forth efforts to develop a quantitative model in order to compare the texture profiles of different extruded products (Hecke *et al*.,1998; Fan *et al*., 2013; Ding *et al*., 2005; Chanvrier *et al*., 2014). For our experiment, the texture of each product was characterized according to the compressive stress-strain relationship proposed by Nuebel and Peleg (Christoph *et al*., 1993).

Six commercial products made with traditional extrusion techniques were analyzed and compared to fourteen SCFX products. Quantitative measurements of the stress-strain relationships for each product were made with a texture analyzer (Instron Universal Testing machine model 1122) to assess the hardness and brittleness/crunchiness of the products. A sigmoidal mathematical model was employed to interpret the stress-strain curves (Christoph *et al*., 1993). The applicability of this sigmoidal model to the stress-strain relationships of a variety of spongy bakery products, popcorn and plastic foam was previously demonstrated (Peleg *et al*., 1998; Swyngedau *et al*., 1991; Anton *et al*., 1993). The force required to achieve a certain deformation demonstrates the hardness of the extruded products. The 'jaggedness' of the stress-strain curve is a manifestation of the material crunchiness or brittleness of the puffed extrudate (Rohde *et al*., 1993).

There were three primary objectives in this experiment. The first objective was to analyze the textural characteristics and compare SCFX products with commercial products. The next objective was to test the applicability of this sigmoid stress-strain model to SCFX product. Finally, the overall aim is to determine how to design SCFX products with specific texture profiles.

MATERIALS AND METHODS

Fourteen SCFX products were made by the Department of Food Science at Cornell University (Ithaca, NY) using a A pilot-scale Wenger TX-57 Magnum corotating, self-wiping, twin-screw extruder with a barrel diameter of 52 mm and length to diameter ratio (L/D) of 28.5:1 (Wenger Manufacturing, Sabetha, KS). The method used to create the SCFX products is described elsewhere (Rizvi *et al*., 1995). Six traditional-extrusions, commercial products were purchased from a local supermarket. The names and the compositions of the products are given in the Table 1. The commercial products were removed from their packages and each product (commercial and SCFX) was stored at ambient temperature (22^0c). Samples of each product were compressed in an Instron Universal Testing machine model 1122 at a speed of 10mm/min (Christoph *et al*., 1995). For each trial, the sample was arranged in the specimen holder as one layer. The device created a chart representing the stress-strain relationship as the sample was compressed. The data from the stress-strain chart for each sample were read and manually input into Microsoft Office Excel to generate the charts

shown in this paper. The stress-strain data were then analyzed using JMP software to determine if the stress-strain relationship for each product fit the empirical mathematical model that was developed for the description of the sigmoidal stress-strain relationships of cellular solids (Eq. 1) (Swyngedau *et al*., 1991). If the model does fit a given product, then the JMP software was used to determine the values of k_1, k_2, n_1, and n_2.

Equation 1, $\sigma = k_1\epsilon^{n_1} + k_1\epsilon^{n_2}$

Where σ is the engineering stress, ε is the engineering strain and k_1, k_2, n_1, and n_2 are constants depending on the specific texture of the products.

For each sample which fit the sigmoidal model expressed in (Eq. 1), the complete data set of the stress-strain relationship was used to calculate a normalized dimensionless stress $Y(\epsilon)$ using following transformation (Rohde et al., Barrett *et al.*)

Equation 2, $$Y = \frac{\sigma_{(\varepsilon)} - \sigma^*_{(\epsilon)}}{\sigma^*_{(\varepsilon)}}$$

Where σ* is the set of theoretical, smooth stress values generated from (Eq. 1) corresponding to the measured strain values. Values for σ* were produced after k_1, k_2, n_1, and n_2 were obtained from the model using the JMP software. The jaggedness of different samples may be compared by examining the Y-strain curve (Christoph *et al*., 1995). The validity of this Y value and its ability to express crunchiness or brittleness was one of the parameters being evaluated in this experiment.

Table 1. Source, name and description of the samples

Source of the Sample	Name	Description of the sample
SCFX Product (Department of Food Science, Cornell University, NY)	S1	F1 popples bad shape
	S2	F2 harder texture popples
	S3	F3 veggie puffs 150rpm 1% CO2
	S4	F1 Mexican Chocolate 150 rpm 1% CO2, 17.5% MC
	S5	F4 Grape Puffs 130 rpm 25% MC
	S6	F2 Savory Helium 1% CO2
	S7	F4 Grape Puff 1% CO2, 25% MC
	S8	Tomato Carrot 1005 IR
	S9	Broccoli 20% 1002
	S10	F1 MPC
	S11	Apple promace starch 70 %, 1% CO2, 100 rpm, 22.5% Mo
	S12	WPC5-8, WP 60%, Fiber 0%, 1800Ks
	S13	F4 Grape Puffed 130 rpm 17%MC
	S14	F4 Grape Puffs 25%MC 150 rpm 1% CO2
Commercial Product	S15	Gerber graduate yogurt melts
	S16	Cocoa puff whole grain
	S17	Gerber lil's crunchies garden tomato
	S18	Cheerios yogurt burst
	S19	Gerber graduates puffs peach whole grain
	S20	Kix crispy corn puff

For those products which did not fit the sigmoidal model (Eq. 1), the Y-strain curve could not be produced. Instead, only the hardness was quantified for these samples. The hardness of all samples was evaluated by comparing the stress value at 70% maximum strain, a higher stress value implied a higher degree of hardness (Katz *et al*., 1981)

RESULTS AND DISCUSSION

Of the products analyzed in this experiment, 8 of the 14 lab samples and 3 of the 6 commercial products were found to fit the model (Eq. 1). These products are listed in table 2. Figure 1 represents both the empirical stress-strain curves and the smooth curves calculated from the model (Eq. 1) for the lab products (Fig. 1a) and the commercial products (Fig. 1b). Figure 2 is a representation of the normalized and dimensionless stress $Y(\epsilon)$ plotted against strain for the lab products (Fig 2a) and the commercial products (Fig 2b). The $Y(\epsilon)$ -strain curves for lab products S5, S6, S7, S9, S13, and S14 were very similar so their $Y(\epsilon)$ values were averaged and collectively represented as SA in Figure 2a.

Table 2 shows the values of k_1, k_2, n_1, n_2, and stress values at 70% maximum strain for the products that did fit the empirical mathematical model given by equation 1.

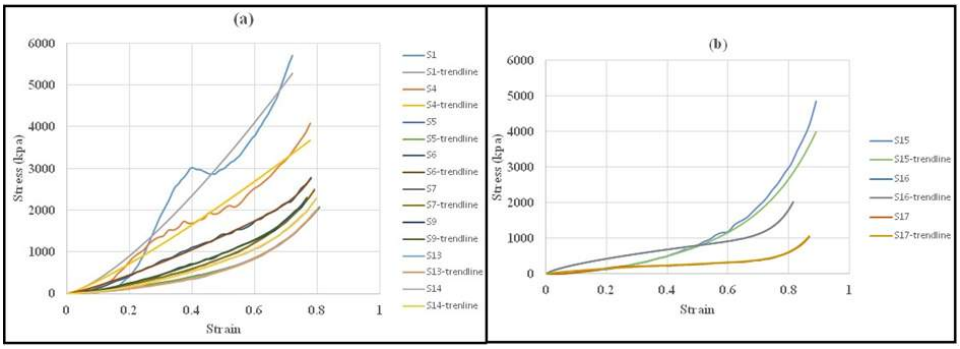

Figure 1. The typical stress-strain actual curves (σ) and trendline curves (σ^*) that fit the model (Equation 1) for, **(a)** SCFX samples S1, S4, S5, S6, S7, S9, S13, and S14, **(b)** Commercial samples S15, S16 and S17

Table 2. Shape characteristics of the stress-strain relationships for the samples that fit empirical mathematical model (equation 1).

Source of the Sample	Sample ID	k1	n1	k2	n2	Stress value for 70% Strain
SCFX (Department of Food Science, Cornell University, NY)	S1	8239.39	1.37	-25657546.07	3006.90	5325.6133
	S4	4970.48	1.20	13543.48	145.91	3181.5352
	S6	3273.79	1.23	25486.17	16.81	2178.6600
	S9	2703.44	1.48	10799.69	11.71	1770.5935
	S7	2305.52	1.48	4079.58	6.67	1715.2625
	S14	2410.80	1.65	5499.27	9.50	1535.4366
	S5	1234.27	1.29	3777.41	5.54	1327.9451
	S13	1467.39	1.58	3662.51	5.85	1286.4469
Commercially available	S15	2474.50	1.76	4133.50	6.40	1862.2695
	S16	1276.00	0.68	14227.00	13.52	1120.6038
	S17	451.50	0.67	4014.50	12.87	394.2337

The typical stress-strain curve of cellular solids has three linear regions connected by shoulders (Christoph *et al*., 1995) but the stress-strain curves for our products show only two regions and a slight shoulder. These two regions represent the deformation of the original structure and the subsequent collapse of the cell walls, respectively. Small or absent shoulders indicate that the product's cell walls have low mechanical resistance over a considerable strain.

The degree of jaggedness and the trend of the curve SA correlates to the curve for commercial products S16 and S17. The normalized curves for S1 and S4 are to some extent smooth with some small jaggedness; they follow the trend of the curve for S15. Thus, the runchiness or crubliness or brittleness of the laboratory products is similar to the tested commercial products, as demonstrated in their respective stress-strain curves. Products with higher moisture contents tend to be less brittle and more plastic, the result being that their stress-strain relationships become much smoother. The jaggedness of the stress-strain relationship is mainly a manifestation of the particles' mechanical disintegration. The disappearance of jaggedness is indicative of the transformation of the compression mechanism from primarily dominated by fracture to plastic deformation and collapse of intact cell wall (Christoph *et al*., 1995). The smooth nature of normalized curves of S1 and S4 suggests less mechanical disintegration of the particles (less fracturing); therefore, it is likely that these products had higher moisture contents and that the stress-strain relationships for their compressions were dominated by plastic deformation.

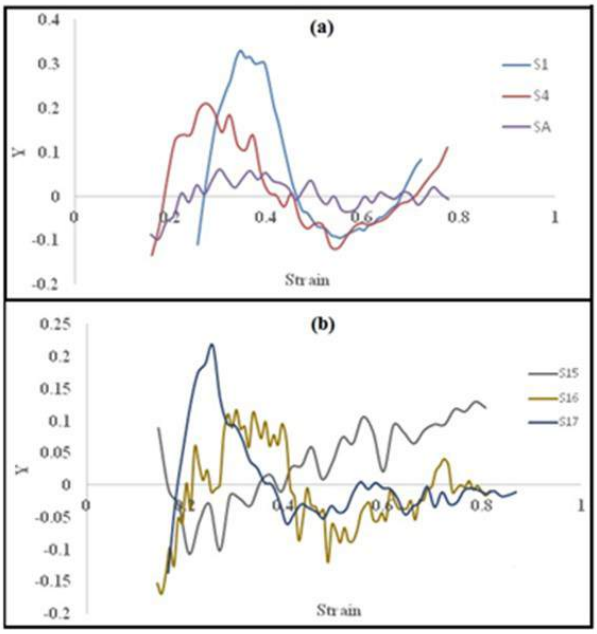

Figure 2: Normalized and dimensionless stress ($Y(\epsilon)$)-strain relationship for, **(a)** Samples from Rizvi Lab S1, S4, SA (the average of S5, S6, S7, S9, S13, and S14), **(b)** Commercial samples S15, S16 and S17.

The stress value ($\sigma_{(\epsilon)}$ at 70% maximum strain for each product was used to compare the hardness of the products. Table 2 shows the lab products and commercial products in descending order of hardness.

The hardness of all the lab products except S1, S4 and S6 is comparable to the hardness of commercial products S15 and S16. Products S1 and S4 have a high degree of hardness and very smooth normalized stress (Y(ϵ))-strain curves. There may be a correlation between product hardness and the smoothness of its normalized stress-strain curve.

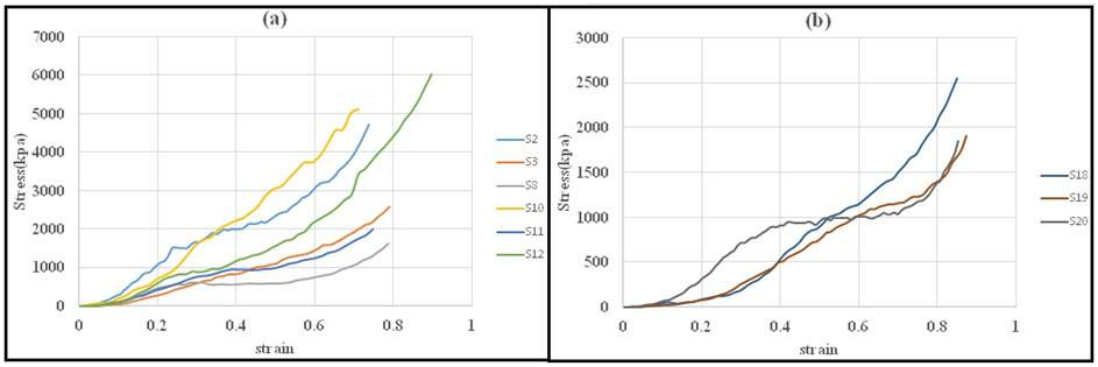

Figure 3. The typical stress-strain curves that do not fit the model (Equation 1) for, **(a)** SCFX samples S2, S3, S8, S10, S11 and S12, **(b)** Commercial samples S18, S19 and S20

Table 3. Shape characteristics of the stress-strain relationships for the samples that do not fit empirical mathematical model (equation 1). Strains 1 and 2 represent the ranges of the two linear portions of the sigmoidal curve respectively.

Source of the Sample	sample ID	Strain1	a1	r^2	Strain2	a2	r^2	Stress value for 70% Strain
SCFX sample (Department of Food Science, Cornell University, NY)	S10	0.13-0.26	5738.1	0.978	0.59-0.72	12162	0.9788	5014.3765
	S2	0.11-0.21	7692.2	0.9934	0.55-0.65	7105	0.9796	3942.3371
	S12	0.1-0.2	5023.2	0.9913	0.7-0.85	12130	0.9959	3164.2443
	S3	0.1-0.3	2857.6	0.9955	0.65-0.75	5783.1	0.9971	1922.7539
	S11	0.1-0.3	3367.3	0.9981	0.65-0.75	5766.6	0.9949	1687.5970
	S8	0.09-0.21	3766.6	0.9928	0.7- 0.8	6023.2	0.9844	1058.2063
Commercially available	S18	0.15-0.4	1630.7	0.9477	0.58-0.79	3823	0.9845	1576.9349
	S19	0.25-0.5	2495.8	0.9982	0.78-0.88	6173.4	0.9571	1161.9520
	S20	0.16-0.3	3889.7	0.994	0.76-0.85	6991.7	0.9735	1038.4571

The compressive stress-strain relationships of lab products S2, S3, S8, S10, S11, S12 and commercial products S18, S19 and S20 do not fit the model (Eq. 1) according to the JMP software analysis. Figure 3 shows the relationship between stress and strain for these products. The slopes of the linear parts of the stress-strain curves for these products were determined and are represented in the Table 3 as a_1 and a_2. The value r^2 is the coefficient of determination for the curves represented in Table 3. Slope a_1 represents the portion of compression dominated by elastic deformation while slope a_2 represents the portion dominated by cell wall collapse.

The comparative hardness of the products can be understood from the stress value at 70% maximum deformation. It is logical that the magnitude of slope a_1 for a given curve represents the elasticity of the product's super-

structure whereas the magnitude of slope a_2 for a given product represents the elasticity of its cell wall structure. A higher slope means more force is required to deform the product. All three regions of the stress-strain curve for all the samples except S3 and S10 are evident from Figure 3 (a, b). The highest stress, third region represents the densification of the collapsed cell walls.

The hardness at 70% deformation for products S3, S8 and S11 is in the same range as the hardness of commercial products S18, S19 and S20. The stress-strain curve for these products does not follow the sigmoidal model (Eq. 1) so the brittleness of these samples could not be characterized from the stress–strain curve. However, in the absence of the sigmoidal model, it may be possible to characterize the brittleness of these products based on the slopes of the a_1 and a_2 regions.

For all of the non-sigmoidal products (listed in table 3) except S2 the magnitude of slope a_2 is roughly twice that of slope a_1. This indicates the extent of the difference between the elasticity of the product's superstructure and its cellular structure.

CONCLUSION

The methodology employed in this experiment has allowed for the assessment of the sigmoidal model itself. The sigmoid model for compressive stress-strain relationships has been shown to be applicable to most—but not all—of the products examined in this experiment. Failure of the model for some products suggests a need for further research to develop a broader understanding of the compression mechanism. The model only seems to fit for products which exhibit deformation mechanisms and/or textural characteristics similar to those upon which the model is based.

The textural characteristics measured in this experiment (hardness, jaggedness) were chosen for their correlation with human perception of foods. The jaggedness and hardness of SCFX products and commercial products were found to be similar; thus, future commercial products with specific textural qualities can be produced using SCFX techniques.

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