

Correlation of Extrudate Infusibility with Bulk Properties using Image Analysis

ANN H. BARRETT and EDWARD W. ROSS

ABSTRACT

Corn meal extrudates were produced using process conditions designed to obtain various product structures. Extrudates were infused with a particle-containing, high melting point lipid suspension, a process used to produce calorically dense components for military rations. Image analysis was used to estimate the degree of particle penetration into the extruded matrices and determine cell size distributions. Infusion uniformity was correlated with structural attributes, such as density, expansion, and average cell size.

INTRODUCTION

VACUUM INFUSION of porous food matrices is currently used to produce a type of calorically dense ration component. This technique increases caloric value of food by filling the pore volume of the matrix with a calorically rich, usually lipid-based, substance (Briggs et al., 1986). Food matrices typically used in infusion are highly expanded, extruded, grain-based flatbread crackers, which generally have porosities of 90% or higher. The infusing liquid, or "infusate," consists of a high melting point fat blended with a high concentration of dehydrated foods, the actual composition of which are determined by formulation. Infusion is carried out at elevated temperatures so that the infusate is liquid; the infused product is subsequently cooled to room temperature and the infusate becomes solid.

Since the infusate will largely determine appearance, flavor, and caloric density of the finished product, maximum penetration of lipid and suspended particles into the matrix is desirable. Vacuum infusion is essentially a reverse filtration process during which a suspension containing a high concentration of particles is forced to permeate a porous structure. If suspended particles are too large to pass through the available fractures and intercellular channels in the extrudate, visible exclusion of the particles from some portions of the matrix will result. Since the suspending lipid is white when it is solid and food powders are frequently colored, separation of particles from the lipid will produce a nonuniform "patchwork" appearance in cut surfaces of the product. In these instances some cells are completely infused with the lipid and particles, and thus have the same appearance and color as the original formula; others, however, are virtually impermeable to particles, and exclusively contain the white suspension lipid (Fig. 1). Separation of food powders during infusion detracts from the quality, acceptance, and in extreme cases, the nutritive value of the infused product.

Efforts to optimize infused products have included milling to decrease suspended particle size (Barrett, 1986), use of surfactants (Barrett, 1987), and modeling the process with idealized components. Barrett et al. (1989) using standard filters and well characterized particles, found that penetration through a porous interface, prior to particle bridging and filtercake

formation, increased exponentially with the ratio of pore size to particle size; it was also found that the rate of particle bridging and clogging of pores was accelerated by increasing the concentration of particles in the suspension. Infusion of extruded matrices, however, is more complex than infusion through filters due to the difficulty in determining extrudate permeability, which can vary markedly from cell to cell within a given extruded sample. Quantitative assessment of particle penetration into the matrix is also not straight forward.

Many investigators have worked on relating process parameters to extrudate attributes, particularly expansion and density. Chinnaswamy and Hanna (1988a) reported a negative correlation between radial expansion of corn starch and moisture content for systems having at least 13% moisture, which they attributed to increased gelatinization at low moisture levels. They further suggested that reduced expansion at extremely low moisture contents, below 13%, was due to degradation of the starch into low molecular weight fragments. Fletcher et al. (1985) similarly reported that the radial expansion of extruded maize grits correlated negatively, and product density correlated positively, with moisture. Faubion and Hosney (1982), working with wheat starch, demonstrated a negative relationship between product diameter and moisture at levels above 17%.

A negative relationship between radial and longitudinal expansion has been documented by Launay and Lisch (1983), who attributed this correlation to the respective influences of melt elasticity and melt viscosity on the two indices. Alvarez-Martinez et al. (1988) incorporated radial and longitudinal expansion into a generalized model that showed a negative correlation between volumetric expansion and moisture content.

Starch type also influences expansion and density. Chinnaswamy and Hanna (1988b) demonstrated that the radial expansion of corn starch peaked at a 50:50 ratio between amylose and amylopectin. Bulk density, however, was negatively affected by amylose content throughout the experimental range.

Increased degradation of corn starch at low moisture, as indicated by increased extrudate solubility, gelatinization, and susceptibility to enzyme attack, has been documented by Gomez and Aguilera (1983, 1984). They (1984) also studied the issue of cell size, finding that extrudates were progressively broken into flaky, thin-walled structures as moisture was reduced. Faubion and Hosney (1982) described a wide distribution of cell sizes and noted ruptures in wheat starch products extruded at high moisture.

The objective of our study was to relate the functional properties--permeability and infusibility--of extrudates to other structural characteristics such as density, expansion, and cell size distribution; such correlation would assist development of infused products by indicating what types of extrudates are acceptable matrices. Experiments were therefore designed to measure penetration of a standard infusion formula into a series of widely varying extrudate structures.

MATERIALS & METHODS

EXTRUDATES were produced under conditions expected to yield a range of bulk properties, primarily through moisture variation. The products were infused using identical conditions. New analytical tech-

Author Barrett is with the Advanced Foods Branch, Technology Acquisition Division, Food Engineering Directorate, U.S. Army Natick RD&E Center, Natick, MA 01760. Author Ross is with the Dept. of Mathematical Sciences, Worcester Polytechnic Institute, Worcester, MA 01609.

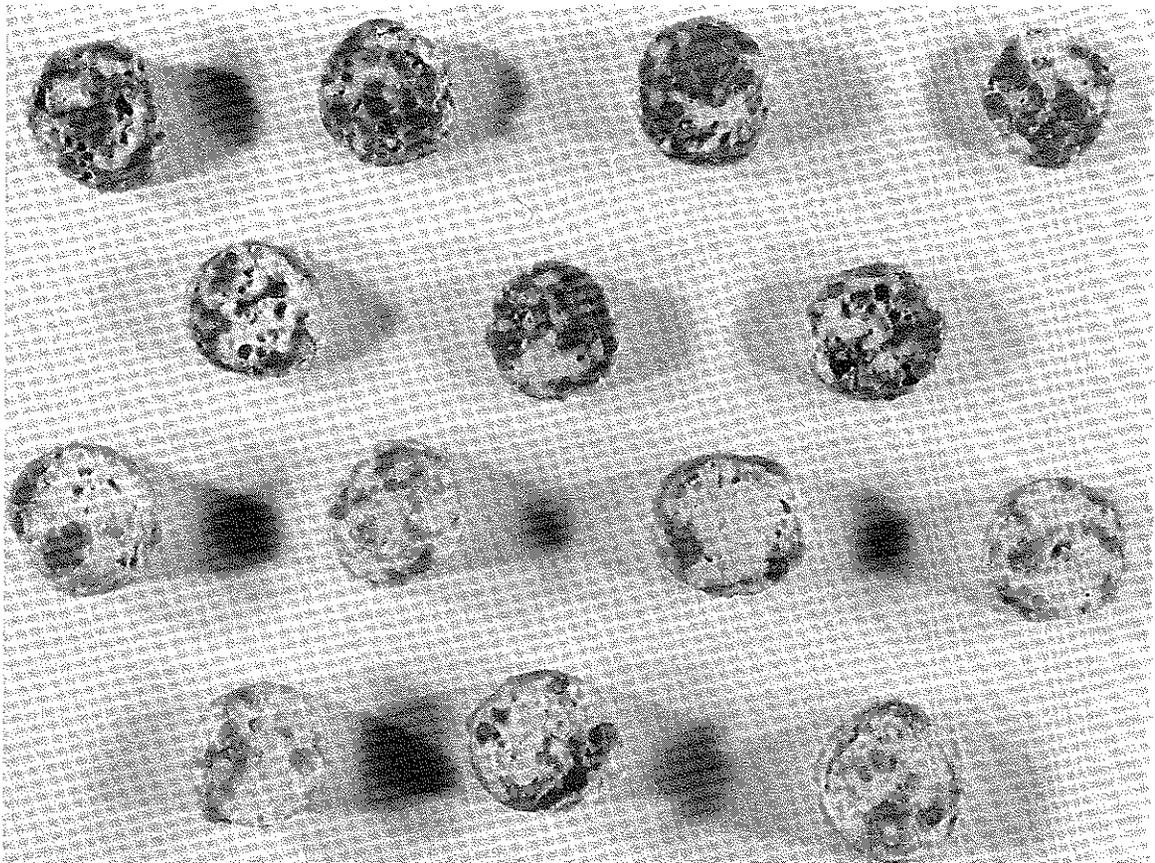


Fig. 1—Photograph of cross-sections of an infused extrudate, illustrating separation of particles. Dark regions contain particles; white regions were impermeable to particles and contain clarified fat.

niques were developed to evaluate extrudate structures and determine efficiency of infusion into extruded matrices. Image analysis was used to determine cell size distributions and to measure the uniformity (i.e. extent of visibly separated lipid) of infused products. Data from these determinations were correlated with results from other analyses, including product density, cross sectional area, linear weight, and solubility.

Extrusion parameters

Corn meal obtained from American Maize was processed on a Wenger X20 extruder through a 3 mm by 18 mm flatbread die. The moisture content of the dough was adjusted during extrusion to levels of 16, 18, 20, 22, 24, and 26% at solids feed rate of 1.8 kg/min. The extruder was operated at 350 rpm with no external heating or cooling. The die temperature varied inversely with moisture content from 155°C to 200°C.

All extrudates were dried at 115°C to a moisture content between 5% and 7%.

Infusion parameters

Each extruded product was vacuum infused with a formula consisting of 70% Durkee Kaomel, 15% Dazaan cocoa powder, 14% Domino confectionery sugar, and 1% lecithin. The formula was made by melting the Kaomel and mixing all ingredients in a high shear blender. Infusion was carried out in a small vacuum chamber. The chamber, with the extrudate within, was evacuated to 1.5 kPa pressure and a valve leading to the infusion liquid reservoir opened, allowing the liquid formula to fill the chamber and permeate the extrudate. The valve was closed and the system restored to atmospheric pressure and held for 2 min. The chamber was then drained and the products cooled to 22°C to solidify the lipid.

A cocoa-based formula was selected because separation of particles in this system produced a distinct pattern of suspension-filled (dark) cells and exclusively Kaomel-filled (white) cells that was suited to image analysis.

Image analysis techniques

An Olympus Cue 2 image analyzer was used to characterize the extrudates and infused extrudates. The image analyzer operated by assigning a grey level value (i.e. position between black and white) to each pixel in the image. Threshold levels were selected to construct a binary (black and white) image in which pixels having grey levels between the thresholds were black, and pixels having grey levels below these values were white. Black objects within the binary image were then counted, measured, and subjected to statistical and morphological analysis.

Measurements

Extrudate expansion and density. The bulk density of each product was calculated by measuring both linear weight (g/cm), determined by weighing measured lengths of the extrudate, and cross sectional area (cm²) determined by image analysis. Cross sectional area was measured by tracing the circumference of the extrudate image (cut cross section) on the video monitor using the computer mouse. The instrument, which was calibrated to convert number of pixels to square centimeters, measured the area of the image. Linear weight was then divided by radial area to determine density. For each sample, three sections were averaged for radial area and four lengths were averaged for linear weight. This method of measuring density was particularly useful for unevenly shaped products since cross-sectional area was measured directly and not calculated from a geometric formula based on a perfectly round, or in the case of flatbread crackers, a perfectly rectangular or oval shape.

Extrudate cell size distribution. The cell size distributions of extrudates were measured by cutting cross-sections of the products and blackening the cut, cell wall surfaces with ink (Fig. 2a), which delineated cells and allowed clear projection of the structure onto the video monitor. The problem of depth, or picking up features lying deeper than the blackened cut surface, was eliminated by adjusting grey level threshold values so that only truly black pixels were incorporated into the image. The result was a black and white image of the cell wall

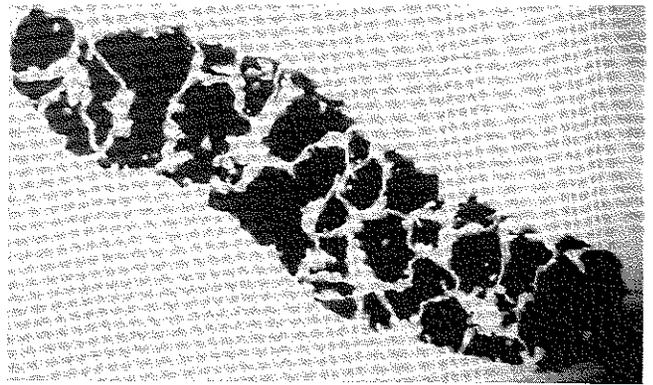
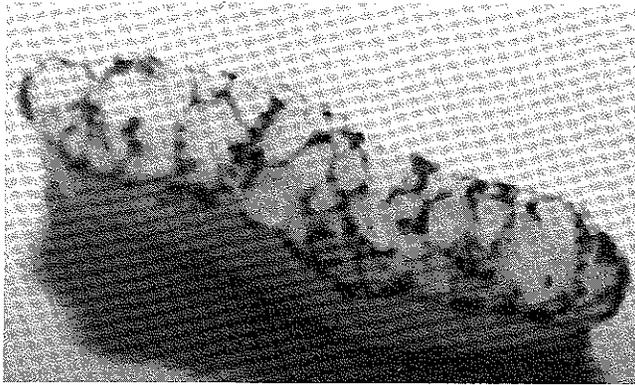


Fig. 2—(a) Photograph of inked extrudate section. (b) Video image of inked extrudate section, after thresholding, inversion, and enhancement.

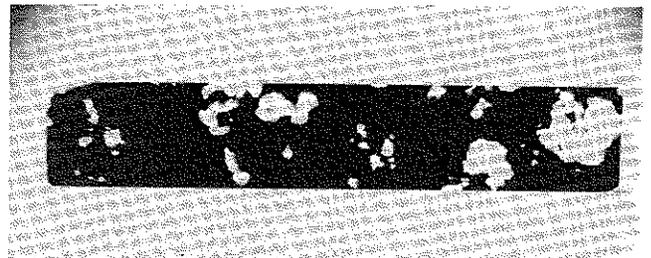
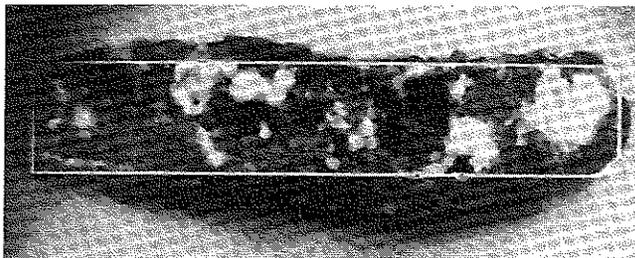


Fig. 3—(a) Video image of an infused extrudate cross section, with inscribed rectangle. (b) Binary video image of infused extrudate after thresholding.

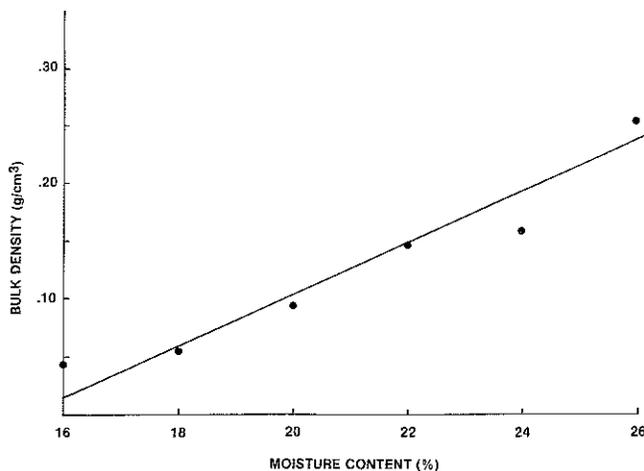


Fig. 4—Effect of extrusion moisture on extrudate bulk density.

“skeleton” at that particular plane. The computer mouse was used to trace over any slightly broken sections in the structure to ensure that cell walls were continuous, so that the program could measure discrete, separated cells. The image was then inverted—making black pixels white and white pixels black (Fig. 2b)—because the analyzer only measures black objects. Upon analysis, the number of cells in each section and the area of each cell (larger than the assigned cutoff of 0.5 mm², selected to eliminate noise) were measured. Three sections of each extrudate were analyzed and the cell area data combined so that between 60 and 130 cells of each product were measured by this technique. Statistical analysis, including calculation of average and median cell areas and evaluation of area distributions, was performed using a MINITAB (Minitab, Inc., State College, PA) program. The tests were repeated on selected samples cut axially to determine if orientation significantly affected cell size.

Extrudate solubility. The water-soluble fraction of the uninfused extrudates was measured by pulverizing each product in a coffee grinder and soaking 10g of the powder in 100 mL water overnight at 22°C. The slurries were then centrifuged at 5700 rpm for 15 minutes and aliquots of the supernatant dried to determine percent solubility.

Infusion uniformity. The infused products were sectioned into 1 cm slices. No further preparation for image analysis was required. The image of each sample was projected on the video monitor (Fig. 3a), and the largest rectangle that could be inscribed within the sample was constructed using the mouse; a rectangular image was required by the program for this function. Grey level threshold values were then selected so that a black and white image that closely corresponded to the sample could be formed (Fig. 3b). These initially selected threshold levels were subsequently used for every sample in the run. The program calculated percentages of the image containing black and white pixels. The fraction of the image that was black, which corresponded to the parts of the extrudate that were infused with particles, was recorded as an index of sample uniformity. The fraction that was white, which corresponded to sections of the extrudate that contained separated fat and no particles, was recorded as a measurement of visible particle separation. Six sections of each infused extrudate were analyzed.

RESULTS

VARYING MOISTURE CONTENT in the extrusion dough produced samples having extremely different densities and degrees of expansion, results similar to the findings of Chinnaswamy and Hanna (1988a) and Fletcher et al. (1985). Extrudate density followed an almost linear relationship with moisture [Bulk Density (g/cm³) = 0.02 + 0.021 × (Moisture(%) - 16); r² = 0.91] within the range of this experiment (Fig. 4). Cross-sectional area and linear weight, however, showed a more complex relation to moisture content (Fig. 5). Cross sectional area had the greatest rate of change at high moisture levels and was maximum at 20% moisture; linear weight had

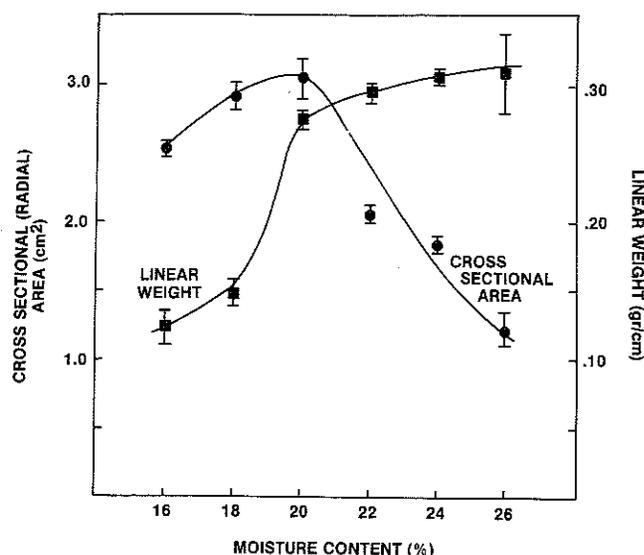


Fig. 5—Effect of extrusion moisture on extrudate cross sectional area (●) and linear weight (■).

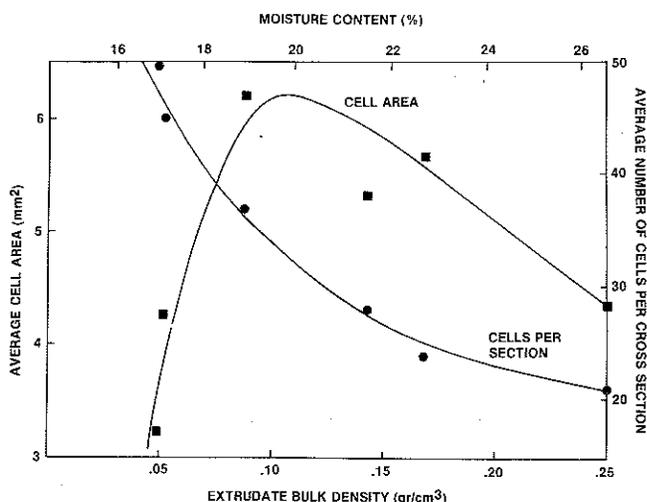


Fig. 6—Effect of extrudate bulk density and extrusion moisture on extrudate average cell area (■) and average number of cells per section (●). (% moisture scale calculated from density-moisture function, Fig. 4).

Table 1—Average and median cell areas of corn meal extrudates

	Moisture content (%)					
	16	18	20	22	24	26
Average cell area (mm²)	3.23	4.25	6.24	5.33	5.67	4.35
Median cell area (mm²)	2.27	2.73	5.04	3.89	3.47	3.53

the greatest rate of change at intermediate moistures (18–20%) and was fairly constant above 20% moisture.

Average cell size (i.e. area) roughly followed radial area and was maximum at about 0.10 g/cc bulk density, or roughly 20% moisture content (Fig. 6). Cell size for selected samples (24%, 20%, and 16% moisture contents) measured axially was only slightly larger than cell size measured radially. The respective values for radial and axial determinations of average cell area were: 24% moisture—5.67 vs. 5.74 mm²; 20% moisture—6.24 vs 6.72 mm²; and 16% moisture—3.23 vs. 3.78 mm². Cells measured axially also displayed slight orientation, in that many were aligned with the direction of extrusion (i.e., a high proportion of cells had their longest dimension within 15 degrees of the axis).

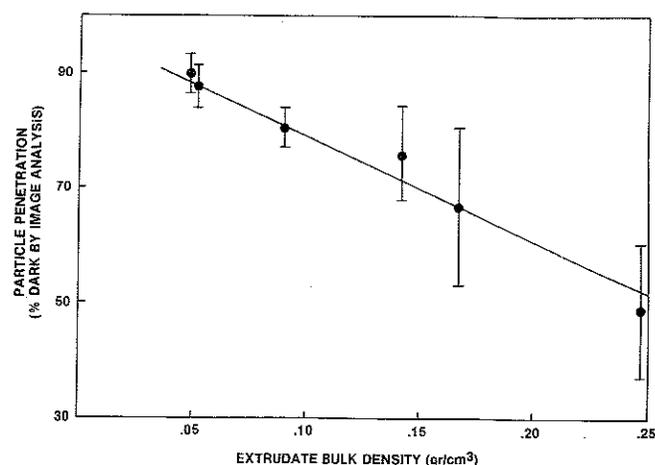


Fig. 7—Effect of extrudate bulk density on infusion uniformity (% dark).

Table 2—Water solubility of pulverized corn meal extrudates

	Moisture content (%)					
	16	18	20	22	24	26
% Soluble fraction	61	58	59	52	37	30
Standard deviation	3.5	3.2	2.9	1.8	2.0	2.3

Cell area distributions were in all cases skewed toward small sizes. Two of these distributions, for the 24% and 22% moisture content samples, were distinctly lognormal [log(cell area) normally distributed]. The others did not conform to lognormal distributions because of too few extreme values. Comparison of average and median values is shown in Table 1.

The number of cells per cross-section was also highly dependent on moisture and density, with low density products having the highest count (Fig. 6). Low density extrudates formed under low moisture processing conditions were generally much more finely subdivided, and had a greater number of small cells than did high density extrudates produced with a higher moisture content.

Infusion uniformity correlated strongly with extrudate bulk density (Fig. 7). Although there was substantial within-sample variation, which would be anticipated due to the inherent structural variation of extrudates, the average value for particle penetration—percent dark as determined by image analysis—correlated with bulk density and followed the linear relationship [% Dark = 98.5 - 187 × Extrudate Density (g/cm³); r² = 0.98]. Including all data points (instead of averages) in the regression gave [% Dark = 98.2 - 184 × Extrudate Density (g/cm³); r² = 0.73]. As is indicated by Fig. 7, most scatter occurred in dense, high moisture samples.

Solubility of the extrudates ranged from 30% to 60% (Table 2). Low moisture samples had twice as much soluble material as did high moisture samples, indicating greater degradation of starch into low molecular weight fragments.

DISCUSSION

THE SUITABILITY of extrudates as infusible matrices, assessed by infusion uniformity, varied widely. Infusibility with particles can be considered analogous to permeability: i.e., the larger in size and more numerous the openings that allow infusion, the more permeable the product. During high-temperature, short-time extrusion, the starch "melt" undergoes rapid expansion as it exits the extruder die due to abrupt reduction in pressure. The resultant structure is cellular but generally not closed, and the openings, consisting of intercellular channels or fractures in the cell walls, are pathways that allow infusion into the extrudate (Briggs et al. 1986). Such openings, pro-

duced during expansion, are failures in the structure and result from overstretching of the cell wall film while the extrudate is still plastic. The number, size, and distribution of these pores that connect cells therefore must be affected by properties of the starch melt; characteristics such as dough extensibility and elasticity can be expected to influence the cell wall thickness and in turn the permeability of the extrudate.

It is reasonable that a physical property such as density would influence permeability and infusion uniformity: the more mass in a unit volume, the higher the potential for increased resistance to penetration. Two extrudates having roughly similar cell size distributions but different densities would have different cell wall thicknesses. For example, an increase from 30 microns to 60 microns in cell wall thickness would not cause a noticeable difference in macroscopic measurements of cell size by image analysis; this two-fold increase in wall thickness would, however (all other factors being equal) reduce the likelihood of rupture during stretching. Thick cell walls are inherently less fragile and less likely to rupture than are thin cell walls.

A similar argument could be made for comparison of two samples of identical bulk density but different cell structures. Extrudates that have small cells are more finely subdivided and contain more cells per unit volume than do extrudates with large cells. If density is constant, finely subdivided extrudates have relatively thinner cell walls and are more likely to develop pores. An interesting correlation existed between density and average cell size in these experiments in that the lowest density extrudates had the smallest average cell size. Cell area, however, went through a maximum and declined beyond a density of about 0.10 g/cm^3 , possibly due to shrinkage of high moisture extrudates after puffing.

Cell size distribution is partly a result of moisture content in that cell walls in high moisture products are relatively more fluid and set more slowly than do cell walls in low moisture products, possibly allowing cells to coalesce. The structure of very high moisture products, however, is soft and subject to partial collapse before cooling. At the other extreme, at very low moisture, quick setting cell walls may be brittle and highly prone to fracture during expansion.

Another factor in permeability may be the chemical structure of starch at the time of expansion. Low moisture extrusion doughs are reported to undergo greater degradation or dextrin-

ization of starch than do high moisture doughs (Gomez and Aguilera 1983,1984). Our results, which show a two-fold increase in solubility due to reducing extrusion moisture from 26 to 16 percent, corroborate those findings. It is possible that cell-wall films containing a high proportion of low molecular weight starch molecules are physically not as extensible as less dextrinized cell-wall films.

CONCLUSIONS

STRUCTURAL ANALYSIS of extruded products provided an understanding of factors that render an extrudate permeable and infusible. Data on density, cell size, and solubility supported trends observed in the relative infusion efficiency of samples. New image analysis techniques provided useful data by which to characterize extrudate structures.

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