Effect of Free Fatty Acids Addition on Corn Grits Extrusion Cooking

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ABSTRACT

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The influence of added fatty acids on extrusion cooking of corn grits and extrudate properties was studied. Samples with three average corn grits particle sizes were processed in a twin-screw extruder; fatty acids content (0.2–0.8%, wb) varied, and experimental conditions were kept constant (moisture content 18.4% wb, barrel temperature 150°C, screw speed 165 rpm). The effect of adding fatty acids was studied by analyzing water solubility and absorption indices, expansion indices, and mechanical (puncture test) and structural (computerized image analysis) extrudate properties. When fatty acids were added, water solubility and absorption indices decreased, radial expansion index decreased, longitudinal expansion index increased, number of cells increased, and mechanical resistance of extrudates decreased. The influence of added fatty acids was attributed to the formation of fatty acids-amylose complexes.

Extrusion cooking is one of the most useful food-processing operations and is used to produce a wide variety of food products (snack foods, pet foods, etc.) with unique sensory attributes. Despite increased use of extrusion processing, extrusion is a complicated process that has yet to be mastered. Small variations in processing conditions affect process variables as well as product quality. Extrudate properties are particularly affected by physical (average particle size) or chemical (amylopectin-to-amylose ratio, lipid and protein contents) variations in raw materials. Although cereals such as wheat and corn meals are typically low (<2%) in lipid content (Camire et al 1992, Guzman et al 1992), small changes in lipid content may modify extrudate quality. Lipids can form complexes with amylose and, to a smaller extent, with proteins (Areas 1992, Ho and Izzo 1992). Amylose-lipid complex formation during extrusion has been widely studied (Mercier et al 1980, Colonna and Mercier 1983, Guzman et al 1992, Ho and Izzo 1992). Amylose-lipid complexes are V-polymorph-type complexes (Biliaderis et al 1985, 1986), and the temperature range for formation is generally 100–130°C, for example, 104°C for amylose-lauric acid complexes (Biliaderis et al 1986) and 107°C for amylose-stearic acid complexes (Bhatanagar and Hanna 1994a,b). Extrusion is a typical high-temperature short-time process, so the final structures of complexes are likely to be formed during the cooling period, outside the extruder (Staeger et al 1987). The rate of complex formation depends on the binding capacity of the lipids present. Fatty acids are good binding agents.

Most studies have reported on the effects of lipids on the mechanisms of complex formation. Modification of sectional expansion also has been studied. Only Faubion and Hoseney (1982), who have worked with raw and defatted wheat flours, have observed significant changes in extrudate structure as amounts of lipids varied using scanning electron microscopy. The purpose of our study was to analyze the effects of added fatty acids on the structural and textural properties of corn-based extrudates.

MATERIALS AND METHODS

Raw Materials

Corn grits (Champagne Maïs S10, Whesthove S.A., Saint Omer, France) with three average particle sizes were used: 100 µm (CG1), 200 µm (CG2), and 500 µm (CG3). Grinding began with corn grits >800 µm ground several times on cylinders and sieved for each fraction to carefully control size dispersion. Average particle size (D_m) and size dispersion coefficient (C_m) were determined by sieving ground samples of each batch. C_m was calculated using the definition of Iyer and Drzal (1988):

$$C_m = D_m(84\%) - D_m(16\%)/D_m \quad (1)$$

where $D_m(84\%)$ and $D_m(16\%)$ are the average particle sizes under which 84 and 16% of the total sample weight remains, respectively. When $C_m > 0.5$, size dispersion is wide; when $C_m < 0.5$, it is narrow; and when $C_m = 0.5$, it is normal.

Free lipids are defined as those that can be extracted with petroleum ether. Corn grits samples were ground, and Soxhlet extraction was performed for 8 hr with petroleum ether. For each batch, the average free lipids content was determined for three replicates. Fatty acids composition was obtained by gas chromatography on the extracted fraction of free lipids (Tables I and II).

Extrusion Cooking

Raw materials were extruded in a corotating twin-screw extruder (Clextral BC45) equipped with a 500-mm barrel. The screw configuration was kept constant (four screw elements with decreasing positive pitch and a reverse pitch element at the end). The extruder had a 250-mm feeding section and a 250-mm temperature control zone. The second section was heated by a 7-kW induction heater (Pyromat 300, Schlumberger, Paris, France) set at a wall temperature of 150°C. All extrusion experiments were performed at 18.4% moisture (wb). Water was introduced into the first section with a peristaltic pump (CFG Prominent).

TABLE I
Corn Grits Characteristics and Lipid Content

Corn Grits Sample	Average Grits Size (µm)	Size Distribution Coefficient (C_m)	Free Lipids (%, w/w)	Total Free Fatty Acids (%, w/w)
CG1	101 (10.7)[a]	0.79 (0.15)	0.592 (0.026)	0.19 (0.01)
CG2	208 (3.76)	0.38 (0.01)	0.66 (0.043)	0.2 (0.015)
CG3	490 (3.29)	0.37 (0.037)	0.59 (0.018)	0.16 (0.01)

[a] Standard deviations in parentheses.

TABLE II
Composition (%, w/w) of Free Fatty Acids (main components)

Corn Grits Sample	Palmitic Acid C16	Stearic Acid C18:0	Oleic Acid C18:1	Linoleic Acid C18:2
CG1	25.5	3.1	32.5	30.8
CG2	32.6	4.1	37.0	18.6
CG3	30.5	4.5	36.1	19.2

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Extrudates exited the extruder through a slit die. Melt temperature (T_m) and pressure (P_m) were controlled on-line at the front die plate (Dynisco transducer), located between the end of the screws and die. After leaving the die, cooked melt was directed onto a polyvinyl chloride (PVC) board inclined at a 30° angle. Extrudate samples (50 cm long) were cut after melt shrinkage and completion of water flash-off. Samples (30) were collected for each experiment, dried to 2.5–3% moisture (wb), and stored in opaque, sealed polyethylene bags until analyzed.

Extrusion was performed by automatic startup, allowing process conditions to stabilize for 50 min, and collecting the first samples. After any change, process conditions were allowed to stabilize for 20 min before collecting samples and attaining a steady state.

Experimental design. Barrel temperature was kept constant at 150°C. All experiments were performed with the same screw configuration, screw speed of 165 rpm, feed rate of 35 kg/hr, and moisture content of 18.4% (wb).

Extrusion runs consisted of extruding corn grits alone (CG1, CG2, and CG3), collecting extrusion response variables and extrudates at steady state, and changing the feed by adding a mix of free fatty acids to corn grits to obtain total free fatty acids contents of 0.2, 0.4, 0.6, and 0.8% (wb), respectively. The composition of the free fatty acids mix was similar to that of the native mix (Table II): for CG1, the mix of fatty acids added was 25.5% palmitic acid, 3.1% stearic acid, 32.5% oleic acid, and 30.8% linoleic acid. Corn grits and free fatty acids were premixed in a 50-L mixer (Lödige) for 45 min.

Expansion Indices

Longitudinal, sectional, and volumetric expansion indices (LEI, SEI, and VEI, respectively) were calculated from equations proposed by Alvarez-Martinez et al (1988) using caliper-square measurements:

$$\mathrm{LEI} = [(1 - M_d)\,\rho_d\,S_d]/[(1 - M_e)\,\rho\,S_e] \quad (2)$$

$$\mathrm{SEI} = (S_e/S_d)^2 \quad (3)$$

where ρ_d is melt density at the die (ρ_d = 1,400 kg m^{-3}), ρ is apparent density of extrudate (kg m^{-3}), S_d and S_e are cross sections (m^2) of die insert and extrudate, respectively, and M_d and M_e are moisture contents (%) of melt and extrudate, respectively. For each experiment, average expansion indices were determined from 10 replicates.

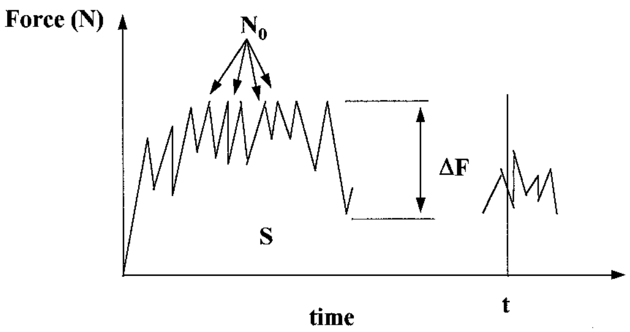

Fig. 1. Cylindrical puncturing test: force-time curve.

Structural Properties

Computerized image analysis was performed according to the method described by Smolarz et al (1989). A computer (VAX 750) linked to a vision system (Grinnel) equipped with a color monitor (Conrac). A camera (charge-coupled device) capable of recording images at 512 × 512 pixels was used. The method consisted of identifying the contours of cells and whole extrudate; eight replicates were collected randomly during extrusion runs. Transverse cuts of extrudates were made with a circular saw (14,500 rpm). Scanned images of cuts were stored as digital images (256 gray levels). Gray levels were grouped to obtain a binary image. Extraction of contours was performed by searching for contrast between dark and white areas of binary images. Structural parameters calculated based on this computerized image analysis included cell density (D_c, cell number per unit area) and mean cell size (S_c).

Mechanical Properties

Extrudates were tested by cylindrical puncturing in a rheometric apparatus (TA.XT2, Rheo, Palaiseau, France). For each experimental point, 10 replicates were used. The apparatus consisted of an electrical jack that moved the cylinder head (4.5 mm diameter) at a constant speed (0.6 mm/sec), a force gauge attached to the head, and a personal computer to operate the jack. The computer recorded and analyzed the force-time curve as the area under the curve (S) (Fig. 1), which represented the work done for a given displacement time (t) by the puncturing device. The force drop (ΔF) relative to each peak, representing local resistance of cell

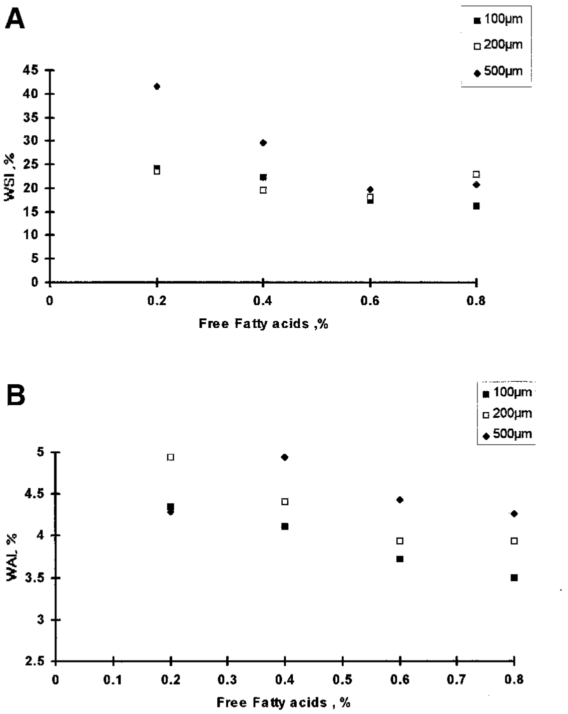

Fig. 2. Water solubility and absorption indices (**A,** WSI and **B,** WAI, respectively) as a function of free fatty acids content.

TABLE III
Statistical Analysis of Results (*P* values based on analysis of variance)[a]

Factor	WAI	WSI	LEI	SEI	D_c	S_c	F_p	F_{sr}
D_m	**0.087**	0.110	**0.0054**	**0.0083**	0.211	**0.0015**	**0.0047**	**0.0028**
%FA	**0.084**	**0.100**	**0.0050**	**0.0014**	0.481	0.6900	**0.0337**	**0.0158**

[a] *P* values are ratios of Fisher numbers. Bold values indicate a factor is significant at >90% (P < 0.1). WAI = water absorption index; WSI = water solubility index; LEI = longitudinal expansion index; SEI = sectional expansion index; D_c = cell density; S_c = mean cell size; F_p = average puncturing force; F_{sr} = average specific force of structural rupture; D_m = average particle size; %FA = percent fatty acids.

walls, and number of peaks (N_0) also were recorded. From these data, three puncture test parameters were calculated to represent mechanical behavior (Bouvier et al 1997): average puncturing force (F_p, N), which was equal to work (S) divided by displacement time (t); average specific force of structural ruptures (F_{sr}, N), which was the sum of force drops (ΔF) divided by number of peaks (N_0); and spatial frequency of structural ruptures (N_{sr}, mm), which was the total number of peaks per unit penetration depth.

Water Solubility and Absorption Indices

The water absorption index (WAI) is the weight of the gel obtained per gram of dry sample. The water solubility index (WSI) is the amount of solids recovered by evaporating the supernatant from the water absorption test. WAI and WSI were measured using the method developed by Anderson et al (1969).

Scanning Electron Microscopy

Dried transverse cuts of extrudates were coated with gold and viewed on a scanning electron microscope (JEOL JSM 840). Representative photos (10 each) were taken for each experimental point.

Statistical Analysis

Statistical analysis was performed with software (Statgraphics Plus, Uniware, Paris, France). The Yates algorithm was used to determine factors significant at >90% ($P < 0.1$; Table III).

RESULTS AND DISCUSSION

Characteristics of Corn Grits

All samples had narrow size dispersions (Table I). Soxhlet extraction revealed similar free lipids contents for CG1, CG2, and CG3 (Tables I and II). Although the free lipids content was approximately the same for the three grits sizes, the amount of free fatty acids decreased significantly when particle size increased, probably because of the effect of size on advancement of enzymatic hydrolysis of triglycerides: the larger the size, the lower the rate of enzymatic hydrolysis.

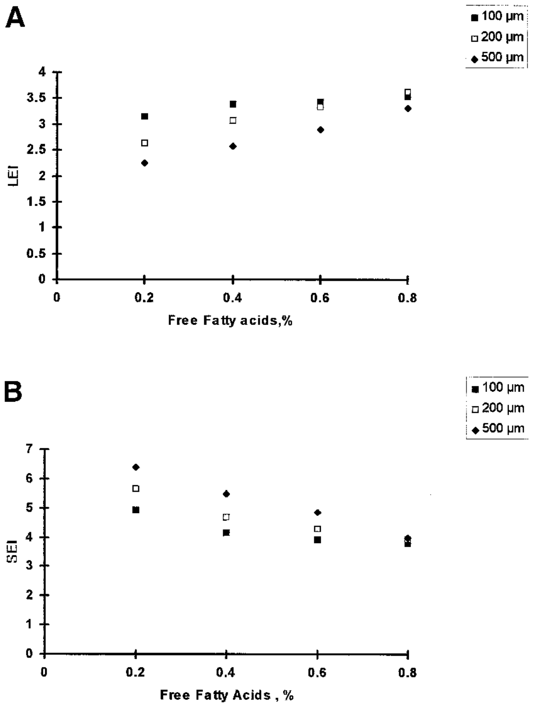

Fig. 3. Effect of free fatty acids on **A,** longitudinal (LEI) and **B,** sectional (SEI) expansion indices.

WSI and WAI

Statistical analysis (Table III) showed the significant effect of added fatty acids on WSI and WAI. As free fatty acids content increased from 0.2% (the reference) to 0.8%, WSI and WAI decreased (Fig. 2A and B). The results clearly showed that there was an inverse relationship of WSI to grits particle size, probably due to the formation of complexes between the macromolecules of amylose and free fatty acids. In fact, Bhatanagar and Hanna (1994a) reported that WSI of corn-based extrudates decreased when complexation rate increased, and the decrease in WSI was more important at lower complexation rates, although the decrease in WSI was relatively low at higher complexation rates. Because amylose-fatty acids complexes are insoluble (Colonna et al 1989), WSI decreased when free fatty acids content in the mix increased.

At lower free acids contents, WSI was higher when the particle size of corn grits was larger (particularly 500 µm) (Fig. 2A), probably due to the level of native free fatty acids, which was significantly lower for 500-µm particle samples: 0.16% (±0.01%) for CG1 compared with 0.2% (±0.015%) for CG3. Consequently, the concentration of complexes would be lower and WSI would be higher for 500-µm grits particle samples. Contrary to Bhatanagar and Hanna (1994a), who did not find any evolution of WAI with added free fatty acids level, we observed a significant reduction of WAI (Table III).

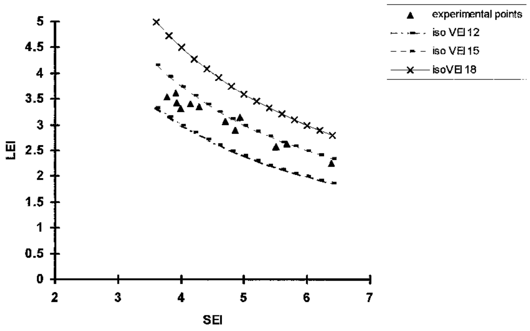

Fig. 4. Correlation between longitudinal (LEI) and sectional (SEI) expansion indices.

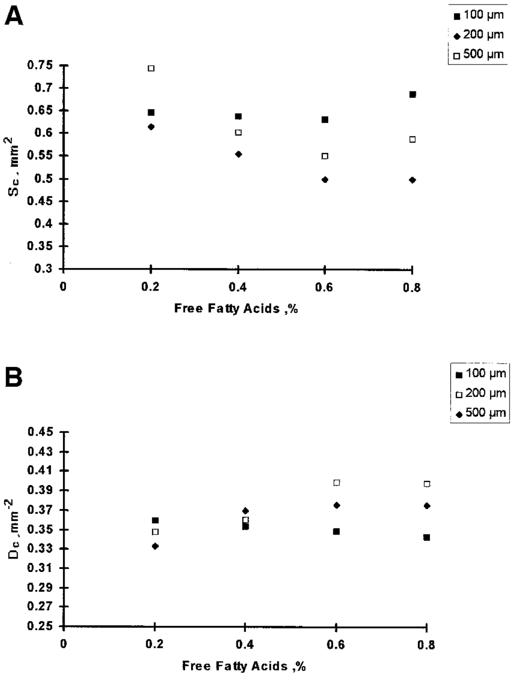

Fig. 5. Effect of free fatty acids on **A,** average cell size (S_c) and **B,** cell density (D_c) of corn grits extrudates.

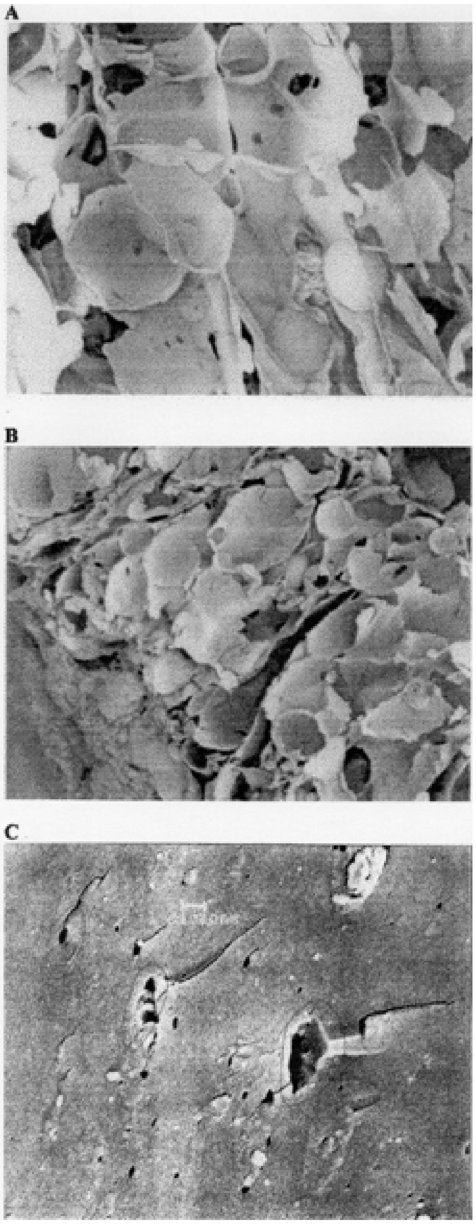

Fig. 6. Transverse cuts of corn grits extrudates. **A** and **B,** Average particle size 200 μm, without and with added fatty acids (0.8%), respectively (magnification at 25×). **C,** Cell walls (magnification at 1,000×; 1 cm = 1 μm).

Expansion of Extrudates

The physical characteristics of extrudates were affected by added free fatty acids. As free fatty acids were added to corn grits, a significant reduction in the radial expansion index (SEI) and an increase in the LEI were observed (Table III; Fig. 3A and B). Bhattacharya and Hanna (1988) and Launay (1994) observed the same tendencies. In a previous study, Desrumaux et al (1998) showed that the expansion phenomenon depends on viscoelastic melt properties. As shown by Colonna et al (1989), semicrystalline amylose-lipid complex formation generates rigid zones and produces a more rigid matrix that is less elastic. Because radial expansion is normally favored by the elastic properties of the melt, the loss of elasticity due to formation of amylose-lipid complexes may explain the decrease in SEI and, consequently, the increase in LEI when percent free fatty acids (%FA) increased. The evolution of both SEI and LEI can be expressed by linear relationships in which slopes depend on the average particle size of grits (Eq. 4):

SEI (100 μm) = 5.11 – 1.83(%FA); $r^2 = 0.86$
SEI (200 μm) = 6.06 – 2.85(%FA); $r^2 = 0.94$
SEI (500 μm) = 7.14 – 3.92(%FA); $r^2 = 0.99$
LEI (100 μm) = 3.71 + 0.62(%FA); $r^2 = 0.88$
LEI (200 μm) = 2.35 + 1.61(%FA); $r^2 = 0.94$
LEI (500 μm) = 1.88 + 1.76(%FA); $r^2 = 0.99$

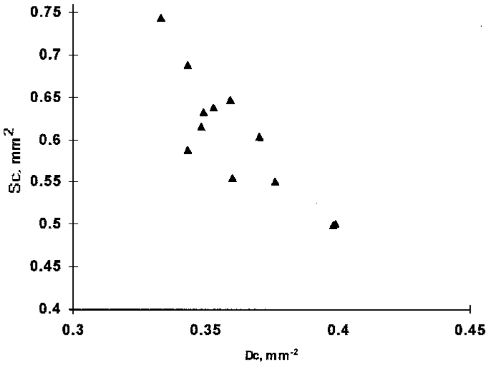

Fig. 7. Correlation between cell size (S_c) and cell density (D_c).

The absolute value of slopes increase when particle size increases. The result is consistent with previous observations of WSI. The evolution of SEI and LEI is more important when low levels of free fatty acids are added. Figure 3A and B also shows that LEI decreases and SEI increases as average grits size increases. However, the higher the average grits size, the less important the effect of particle size on SEI and LEI: at 0.8% free fatty acids content, SEI and LEI of CG1, CG2, and CG3 were very close. Statistical analysis of the results showed that the amount of fatty acids is more significant than particle size (Table III).

As observed previously (Desrumaux et al 1998), there was an inverse relationship between SEI and LEI: LEI = 11.3 $(\text{SEI})^{-0.85}$ with $r^2 = 0.96$ (Fig. 4). The experimental points ranged between 12 and 15 iso-VEI curves. Also, melt expansion was significantly anisotropic, because it expanded more radially (SEI).

Extrudate Structure

As free fatty acids content increased, average cell size decreased significantly (Table III; Fig. 5A). No significant effect was found for cell density. However, cell density increased for CG2 and CG3 (Fig. 5B). Faubion and Hoseney (1982) reported that defatted wheat flours produced extrudates with larger cell sizes. However, we observed the opposite trend for CG1 (100 μm average particle size). CG1 had the smallest average particle size and was obtained after several runs on cylinder grinders. Experimental measurements of the molecular profile of the starch by gel chromatography showed that starch in CG1 was more damaged and behaved like high amylomaize, which may explain the different behavior exhibited by CG1 extrudates.

Computerized image analysis and scanning electron microscope photographs showed the same trends: as the amount of fatty acids increased, there were more, but smaller, cells (Fig. 6A and B). In addition, cell walls were thinner and had more holes (Fig. 6C). Measurement of cell thickness in extrudates showed a significant decrease (by 100%) in CG3 cell wall the thickness as free fatty acids content decreased from 0.2 to 0.8%.

Formation of bubbles (Kokini et al 1992) began with nucleation in the die. Formation of amylose-fatty acids complexes in the extruder or at the exit of the die created heterogeneity in the dough and may have increased nucleation sites in the melt, which produced an increasing number of cells. Also, the increase in number of holes in cell walls (Fig. 6C) may have been due to the loss of melt elasticity, which led to a decrease of SEI and increase of LEI. As LEI increased, cell density increased, and cell size decreased (Desrumaux et al 1998). An inverse relationship also was observed between average cell size (S_c) and cell density (D_c): the higher the average cell size, the lower the cell density (Fig. 7).

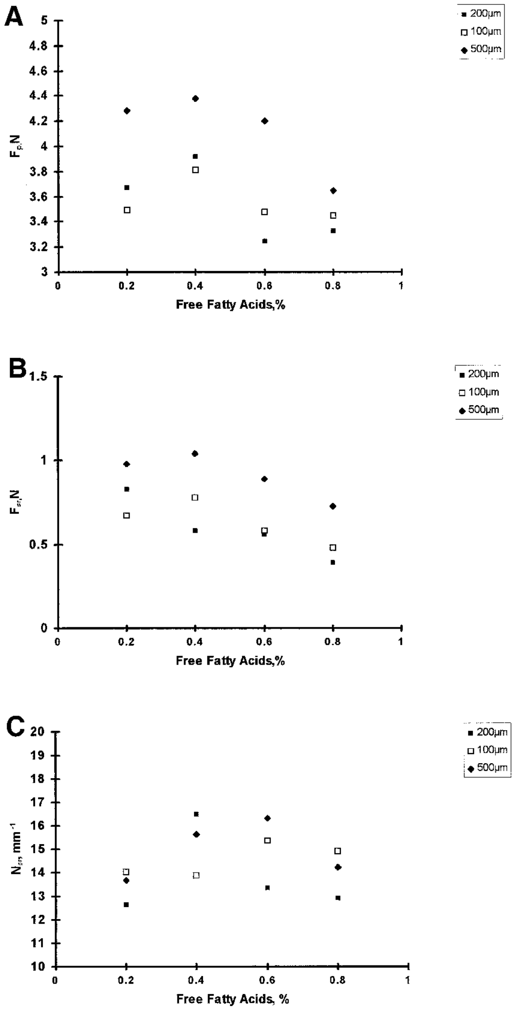

Fig. 8. Effect of added fatty acids on **A,** average puncturing force (F_p); **B,** average specific force of structural rupture (F_{sr}); and **C,** frequency of structural ruptures (N_{sr}).

Statistical analysis revealed the significant effect of added fatty acids on average puncturing force (F_p) and average specific force of structural rupture (F_{sr}) (Fig. 8A and B). The puncture test showed maximum mechanical resistance for extrudates with 0.4% free fatty acids. Mechanical resistance of extrudates was partially due to cell density—the higher the cell density, the higher the mechanical resistance—and partially due to the mechanical hardness of cell walls—the harder the cell walls, the higher the mechanical resistance. When fatty acids were added, the number of cells increased, whereas the thickness of the walls decreased and there were more holes in cell walls. These effects are conflicting: the increase in number of cells gives extrudates higher global resistance, whereas the decrease in thickness and increase in number of holes weakens the mechanical resistance of cells. These antagonistic effects may explain the maximum F_p and F_{sr} observed when 0.4% free fatty acids were added. When fatty acids contents are >0.4%, the global and local mechanical resistances (F_{sr} and F_p) of extrudates decreases, and the effect of decreasing wall thickness is greatest.

CONCLUSIONS

The addition of free fatty acids, even at low levels, significantly modified the quality of corn grits extrudates. As shown in Fig. 9, addition of fatty acids led to flatter products with reduced radial expansion (decrease of 100%). Computerized image analysis showed that extrudates had more and smaller cells. The mechanical resistance of extrudates increased with added free fatty acids of up to 0.4%, then decreased due to decreasing wall thickness and increasingly thin extrudates. Our study has shown that the amount of free fatty acids, even at low levels, does modify the structure and texture of extrudates. Industrial sources of grits vary in fatty acids contents and average particle sizes, which may explain fluctuations in extrudate quality under constant extrusion conditions. As a result, control of fatty acids content appears to be fundamental to quality-control of extruded products, even if native fatty acids form fewer complexes than added fatty acids, which are more accessible. From an industrial point of view, the effect of variation in fatty acids content can be counterbalanced by adjusting operating parameters (screw speed) (Desrumaux 1996).

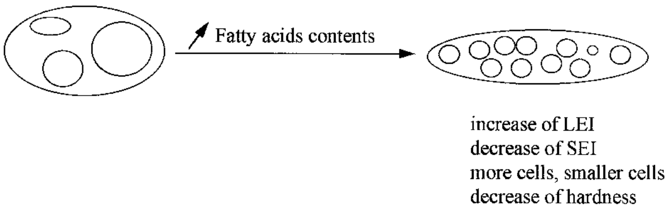

Fig. 9. Transverse cuts of corn grits extrudates.

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