

## INTERNAL POROSITY OF CORN EXTRUDATE AIR CELL WALL

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### Abstract

Transmission electron microscopy was used to observe structures within the air cell wall of a corn-based extrudate. Modified fixation and embedding techniques were employed to obtain optimum thin sections. Photomicrographs from these sections showed minute air cells measuring approximately 5  $\mu\text{m}$  or less. The size of air cells might impose a limiting factor on the infusion of particulate materials throughout the extrudate matrix.

### Introduction

The only conclusive method to differentiate physical change taking place within an extrudate as a result of the extrusion process is microscopy (Stanley, 1986a).

Gomez and Aguilera (1983) used polarized light microscopy to show that the morphology of extruded corn starch samples can be seen as a composite of gelatinized and dextrinized material. Using scanning electron microscopy (SEM) Gomez and Aguilera (1984) observed wall thinning in gelatinized corn starch extrudate and breakdown of the walls into flake-like structures as the extrudate became more gelatinized. Owusu-Ansah et al. (1983) used SEM to ascertain complete gelatinization in a corn starch extrudate. Harper (1986) compared the SEM microstructural differences between defatted soy protein and corn grit extrudates and found rougher cellular surfaces in the corn product compared with the soy extrudate. The general morphological characteristics of corn based extrudates were described by Stanley (1986b) who, using SEM, found these extrudates to have a porous structure consisting of air pockets which are surrounded by laminar sheets of gelatinized starch. To further elucidate extrudate morphology, in this paper we describe the procedure used to obtain microstructural evidence of air spaces or voids within air cell walls of a corn extrudate.

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### Materials and Methods

Samples used in this study were from a single batch of corn-based extrudate which had been freeze-dried.

Light Microscopy (LM). 4 mm<sup>2</sup> fragments of air cell walls were placed on a glass microscope slide and examined using a Zeiss Ultraphot Microscope equipped with Luminar optics and a 16 mm objective lens. Photographs were taken with reflected and transmitted light using Polaroid 55 P/N film.

Scanning Electron Microscopy. 4 mm<sup>2</sup> fragments of air cell walls were affixed to an SEM stub with silver paste, sputter coated with gold palladium in a Hummer X sputter coater, and examined in the SEM mode of a Hitachi 600-2 scanning transmission electron microscope (STEM) at 50 kV. Photographs were taken using Polaroid 55 P/N film.

Transmission Electron Microscopy (TEM). 1 mm<sup>2</sup> fragments similar to those used above were fixed in 2.5% glutaraldehyde in 0.1 M phosphate buffer (pH 7.2) overnight at 22°C (Table 1). The

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fixation, dehydration and embedding schedule is given in Table 1. The composition of the embedding medium is given in Table 2. The fragments were removed from the Epon, placed into embedding capsules containing fresh Epon and polymerized overnight at 60°C in a vacuum oven. After polymerization had taken place the blocks were trimmed and 100 nm sections were cut with a Sorvall MT2B ultramicrotome using a glass knife. The sections were mounted on 3 mm copper grids, stained with uranyl acetate and lead citrate, and examined using the TEM mode of a Hitachi 600-2 STEM (operated at 50 kV). The photographs were taken using Kodak 2415 35 mm Technical Pan film. The STEM objective moveable aperture was set at 50  $\mu\text{m}$  to obtain an image with greater contrast.

Table 1: Fixation, Dehydration and Embedding Schedule

1.	2.5% glutaraldehyde in 0.1 M phosphate buffer pH 7.2	overnight
2.	Buffer wash	1 hour
3.	Buffer wash	1 hour
4.	Buffer wash	1 hour
5.	Osmium tetroxide	1 hour
6.	Buffer wash as in steps 2, 3, 4	1 hour each
7.	50 % ethyl alcohol	30 minutes
8.	70 % ethyl alcohol	30 minutes
9.	80 % ethyl alcohol	30 minutes
10.	90 % ethyl alcohol	15 minutes
11.	95 % ethyl alcohol	15 minutes
12.	Absolute alcohol	15 minutes
13.	Absolute alcohol	15 minutes
14.	Absolute alcohol (over silica gel)	15 minutes
15.	Absolute alcohol / Epon mix (equal parts), without DMP-30	2 hours
16.	Epon mix with DMP-30	2 hours
17.	Polymerize at 60°C under vacuum	24 hours

Table 2: Composition of Embedding Medium (adapted from Dawes, 1979)

Solution A	Epon 812	3.1 ml
	DDSA	5.0 ml
Solution B	Epon 812	5.0 ml
	NMA	4.9 ml
<b>Final Mixture:</b>		
Solution A		7.0 ml
Solution B		3.0 ml
DMP-30		0.15 ml

## Results and Discussion

Examination with reflected light microscopy of the air cell wall portion of a highly porous corn-based extrudate showed an unremarkable rough surface (Fig. 1A); however, when the same sample was observed using transmitted light, air spaces (A) or voids could be seen throughout the sample (Fig. 1B). It appeared that the air spaces were separated by septa; however, none of the structures could be clearly distinguished at this relatively low magnification. At a higher magnification, using SEM, few pores were seen at the surface (Fig. 2). Although the possibility exists that the pores were covered by AuPd during sputtering, it is more likely that, in this sample, there was virtually no communication between the internal air spaces and the surface.

When sections of the air cell wall were examined by TEM the structures which were barely visible with LM could be readily seen (Fig. 3). Minute air spaces (A) or voids of varying sizes separated by septa (arrows) of varying thicknesses within the matrix of the cell wall are probably analogous to the observations (of minute vacuoles in the walls of the larger vacuoles in SEM micrographs of wheat and rye bread crumbs) made by Pomeranz and Meyer (1984).

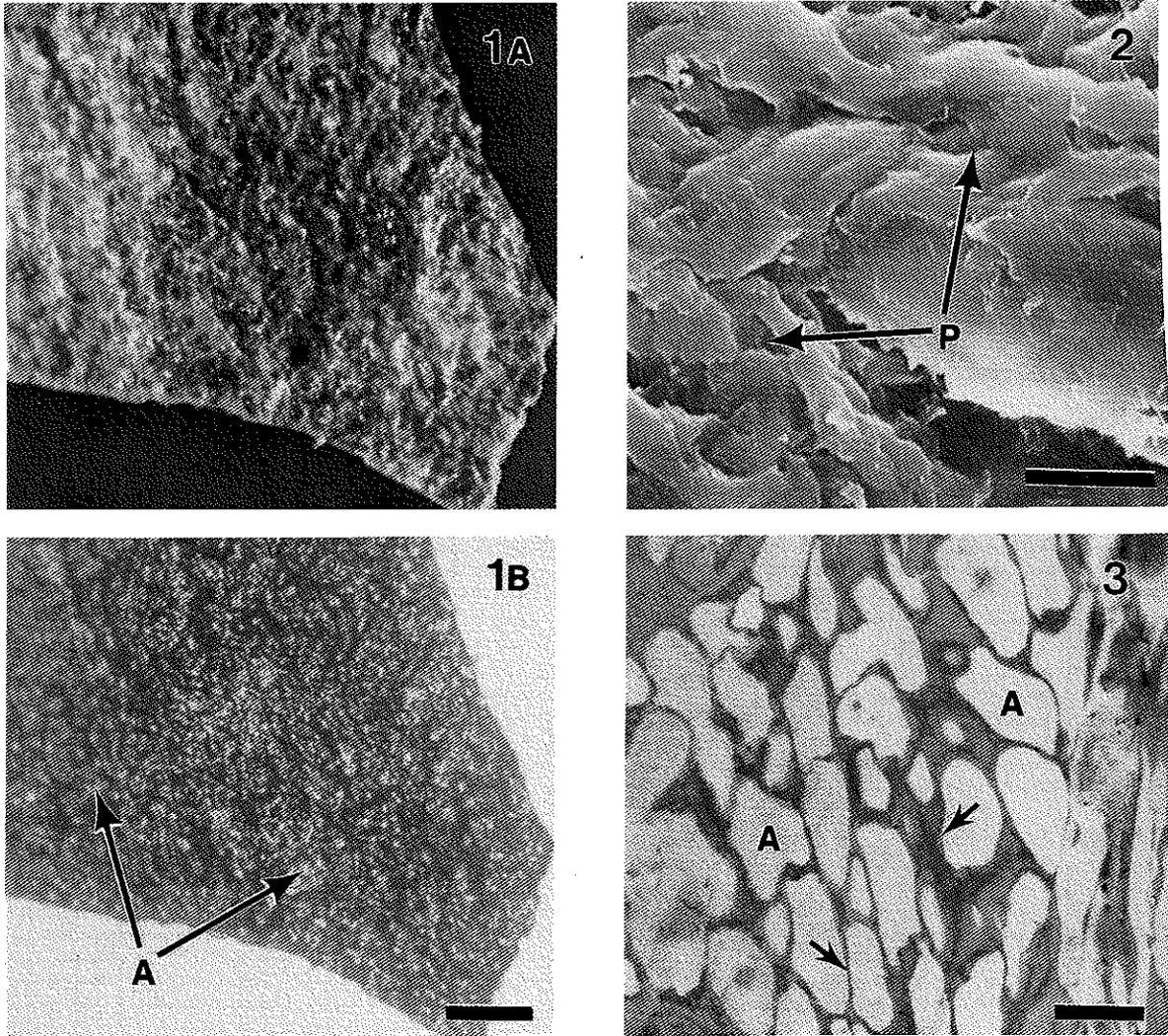
Expansive forces occurring during extrusion cause wall thinning (Gomez and Aguilera, 1984) and pore size as well as wall thickness were related to extrudate moisture content (Harper, 1986). These findings were based partially on SEM data. However, the same conditions affecting the morphology of large extrudate structures, whose dimensions are in the 50 to 100  $\mu\text{m}$  range, probably contribute to structures of 5  $\mu\text{m}$  or less within air cell walls.

Finally, elucidation by TEM of the internal microstructure of the corn extrudate air cell wall permits a more informed evaluation of the effects of changing extrusion parameters on infusion (air cell size may limit diffusion of infusate particulates), extrudate morphology and texture.

## References

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Internal Porosity of Corn Extrudate Air Cell Wall



**Figure 1.** Portion of an air cell wall. Reflected light micrograph (Figure 1A) showing unremarkable rough surface, and transmission light micrograph (Figure 1B) showing air spaces (A) or voids throughout the sample. Bar = 0.1 mm.

**Figure 2.** Scanning electron micrograph of portion of sample seen in Fig. 1. Some pores (P) can be seen within the rough surface. Bar = 50  $\mu\text{m}$ .

**Figure 3.** Transmission electron micrograph of a section of air cell wall. Minute air spaces (A) are separated by septa (arrows) of varying thicknesses. Bar = 1  $\mu\text{m}$ .

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