NOTE

Influence of Wheat-Drying Temperatures on the Birefringence and X-Ray Diffraction Patterns of Wet-Harvested Wheat Starch

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ABSTRACT

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A wheat sample (Gerek-79) harvested at 24% moisture content was dried in a semibatch tunnel dryer at different air temperatures (40, 60, 80, and 100°C). The relative crystallinity of the samples, calculated from the X-ray diffractograms, increased with increasing drying temperatures. The sample dried at 40°C contained A-type starch. An additional small peak appeared ($2\Theta = 20°$) at the drying temperature of 60°C. The intensity of this peak increased with increasing drying temperatures. The peak observed at $2\Theta = 20°$ (*d* spacing, 4.4 Å) is an indication of the formation of V-type structure. With drying temperatures of 80 and 100°C, 95% of the starch granules retained strong birefringence.

Cereal grains are usually harvested at relatively low moisture contents and can be stored for long periods of time under suitable conditions. Moisture is of great importance in their safe storage. In normal years, cereal grains usually dry in the field. However, large amounts of grain, produced in various countries, must be dried for safe storage. Considerable research has been undertaken to investigate possible ways of drying, with alternative methods ranging from ambient air to high temperatures (Pabis and Henderson 1961, Bowden et al 1983, Giner and Calvelo 1987, Biondi et al 1988, Şahbaz et al 1992). Heating the drying air increases its capacity to remove moisture and, thus, it permits more rapid drying than with unheated air.

On the other hand, it is generally accepted that higher temperatures cause damage to gluten proteins, resulting in lower baking quality. For example, when wheat at 14% moisture content was heated for 36 min, gluten was damaged by the temperatures in the 70–85°C range (Kent 1975). Also, the starch quality is significantly affected by the drying temperatures. Lasseran and Boigneville (1975) reported that a single-stage dryer had a marked effect on starch quality of corn varieties at temperatures above 90°C.

X-ray diffraction and birefringence are among the methods used for studying starch structure (Greenwood 1976, Nara et al 1978, MacArthur and D'Appolonia 1984, Hoseney 1986, Gomez et al 1992, Mistry 1992, Suzuki et al 1992). However, the influence of the drying temperatures of wheat on birefringence and X-ray diffraction patterns of starch have not been studied in detail. The objective of this study was to characterize starch by using X-ray diffractometer and bright-field and polarized-light microscopic examinations after applying various drying treatments.

MATERIAL AND METHODS

Wet-harvested Gerek-79 wheat was utilized for the sample. The initial moisture content of the wheat sample was determined according to AACC method 44-15A (AACC 1983) and found to be 24%. Wheat was dried in a semibatch tunnel dryer. Airflow rate was 2 m/sec and temperatures were 40, 60, 80, and 100°C. The heating system consisted of electrical resistances placed in the tunnel. Heating control was achieved by an on/off control system that adjusted the dry bulb temperature of the air. The temperature was measured with a temperature sensor (LM 335 integrated circuit).

Preparation of Flour Samples for Analysis

The wheat samples were dried to the final moisture contents of 8–4% and milled using a Brabender Quadrumat Junior laboratory mill (C. W. Brabender, Hackensack, NJ). These flour samples were held at room temperature under ambient atmosphere conditions until they were equilibrated to a water activity (a_W) value of 0.33, determined by an Aqua Lab model CX-2.

In preliminary studies, starch was isolated from flour according to the method proposed by Walden and McConnell (1955). The X-ray diffraction patterns of both flour and isolated starch were recorded. Although no difference was detected between the diffraction patterns, the flour sample was selected for the X-ray analysis to avoid the possibility of deformations occurring during the starch-isolation and freeze-drying steps.

X-Ray Diffractometry

The X-ray diffraction patterns of the flour samples were obtained on a Phillips X-ray diffractometer (PW 1140) equipped with a monochromator that selects the Kα radiation from a copper target generated under 40 kV and 18 mA. Patterns were recorded from a diffraction angle (2Θ) of 5–35° at a scan speed of 2°/min.

To measure the relative crystallinity of starch in the X-ray diffractogram, the baseline joining background scattering points was drawn (Fig. 1). A smooth curve was then drawn between the low- and high-angle points. The upper region, above this curve, represented X-ray scattering of the crystalline fraction, and the lower part represented that of the amorphous fraction. The whole diffractogram was carefully cut out and then cut again to isolate the two areas. The upper sections were weighed (Libror AEG-220), and the areas were calculated by the comparison of their weights with the weights of known areas prepared using the same paper.

Birefringence and Microscopic Studies

Birefringence was evaluated in water-glycerol (50:50) suspensions of flour. Flour samples were sieved to remove the larger endosperm pieces, leaving mostly particles smaller than 100 μm. Bright-field and polarized-light microscopic examinations were performed using a Zeiss Universal microscope equipped with a 100-W tungsten light source.

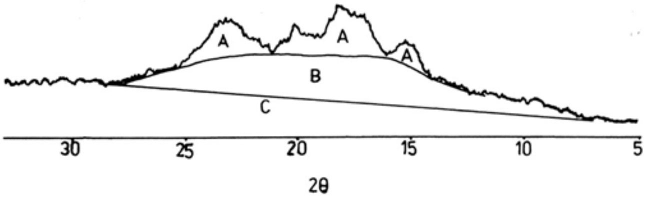

Fig. 1. Evaluation of X-ray diffractogram. **A**, crystalline area; **B**, amorphous area; **C**, baseline.

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RESULTS AND DISCUSSION

X-Ray Diffraction Patterns

The A-type X-ray diffraction pattern of the starch dried at 40°C was similar to that found in literature (Hoseney 1986, Zobel 1988 a,b). The spectrum showed three distinct, but broad, peaks between 2Θ angles of 14 and 16°, 16 and 19°, and 21.5 and 25° with *d* spacings of 5.8, 4.9, and 3.8 Å, respectively (Fig. 2). When the X-ray spectrums of the samples dried at 60, 80, and 100°C were evaluated, it was observed that an additional peak had appeared at about $2\Theta = 20°$ with *d* spacing of 4.4 Å. Although this was a minor peak at the 60°C drying temperature, its intensity had increased with the increasing drying temperatures (Fig. 2).

Zobel (1988 a,b) reported that naturally occurring fatty acids and phospholipids of wheat starch can complex with the amylose fraction and result in V structures. V-type X-ray diffraction patterns show three peaks at 12, 6.8, and 4.4 Å. The peak at 4.4 Å often appears as a first indication of V-complex formation. Favorable conditions for V formation include: moisture levels of 18–45%; temperatures of 90–130°C, and holding times of 1–16 hr. However, in this study, V formation started at lower temperatures (around 60°C).

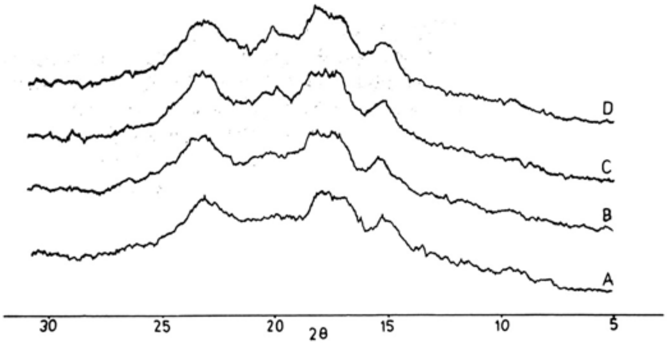

Fig. 2. X-ray diffractograms of samples dried at different temperatures. **A**, 40°C; **B**, 60°C; **C**, 80°C; **D**, 100°C.

TABLE I
Relative Crystallinity of Samples from X-Ray Diffraction Patterns

Drying Temperature (°C)	Area (cm^2)	Relative Crystallinity (%)
40	8.8	100
60	10.0	114
80	11.1	126
100	12.5	142

The distinct peaks demonstrate the crystalline nature of the starch granules, and the area above the curve can be taken as the measure of the crystallinity of starch. The crystallinity of the samples dried at different temperatures were calculated on the basis of the value of the crystallinity measured at 40°C. The measurements obtained from diffractograms of the samples dried at different temperatures and the relative crystallinities are given in Table I. The relative crystallinity of the samples (114, 126, and 142%), dried at different temperatures, increased with increasing temperatures.

Birefringence

As expected, all the starch granules of the wheat samples dried at 40°C displayed a clear maltese cross under polarized light (not shown). The majority of the starch granules (~95%) of the wheat samples dried at higher temperatures (60, 80, and 100°C) retained their birefringence (Figs. 3A–D). Of the remaining granules, ~5% partially lost their birefringence. Only a few granules (~1%) completely lost their birefringence properties, and they can be seen in among birefringent ones in Figure 3C and D. Micrographs typical of partially birefringent granules are shown in Figure 3E and F. Bright-field and polarized-light microscopy studies showed that partially birefringent granules had irregular shapes but retained birefringence in about half of each granule. Although the drying temperatures used in this study (80 and 100°C) exceeded the gelatinization temperature of wheat starch with excess water (Hoseney 1986, Zobel 1988b), we observed strong birefringence in most granules. Thus, in this study, the starch granules did not gelatinize during drying. Therefore, to explain our experimental results, we must consider the amount of water, a basic factor of swelling and gelatinization.

Our wheat sample contained 24% moisture at the start of drying. In a previous study related to drying of the same wheat samples (Şahbaz et al 1992), we determined that most of the initial water was removed during the early stages of drying. The dehydration rate increased with increasing temperatures. For example, 12% of water was removed at 40°C in the first 30 min. This percentage increased to 48% at 100°C for the same period. Therefore, the amount of water in the wheat sample during higher temperature

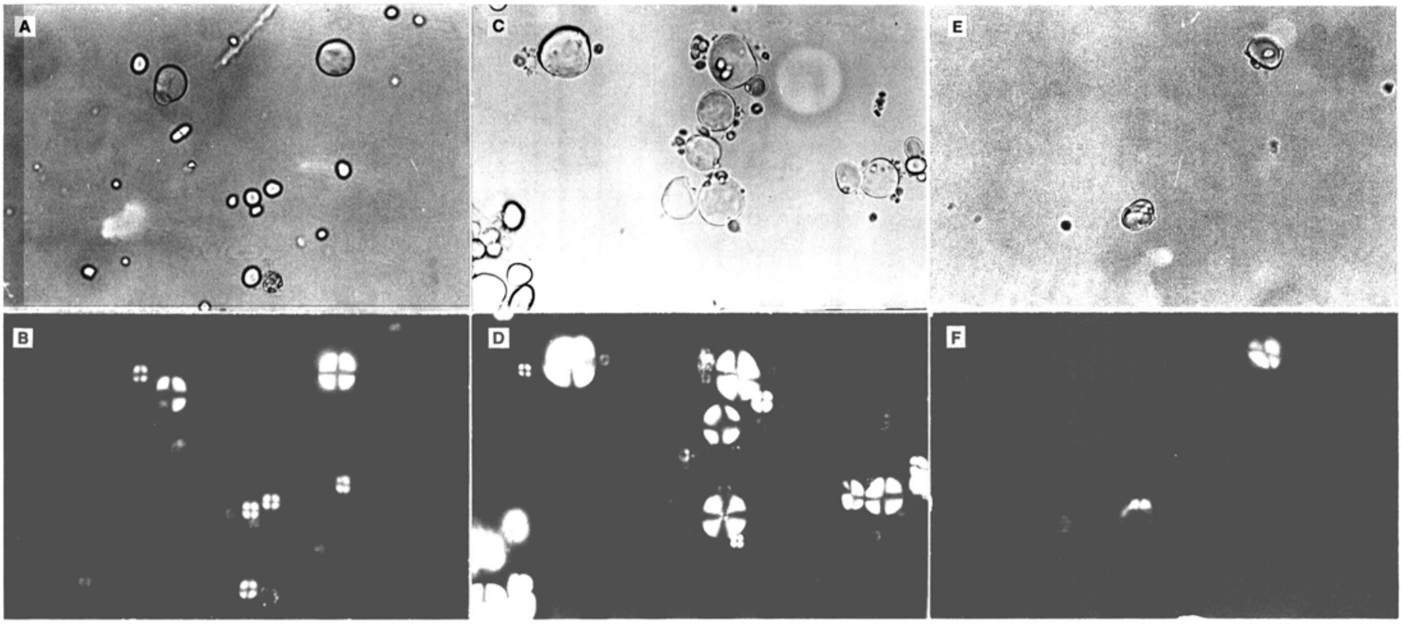

Fig. 3. Bright-field (top) and polarized-light (bottom) micrographs of samples dried at 60°C (**A** and **B**) and at 100°C (**C** and **D**). Partially birefringent starch granules (**E** and **F**).

drying may not be sufficient to cause swelling and gelatinization. The physical constraints of the endosperm cells may also limit the swelling of the starch granules. Thus, it can be concluded that the retention of birefringence during wheat drying is due to the lack of water and to the physical protection of starch by the kernel and endosperm cells. Further investigation, using techniques such as differential scanning calorimetry, is required to support these conclusions.

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