Effect of Moisture Content on Mechanical Properties of Shelled Corn[1]

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ABSTRACT

The effect of moisture content on mechanical properties of yellow dent corn was studied by applying uniaxial compression to individual kernels. Before testing, kernels were equilibrated at each of several relative humidity atmospheres to give a range in moisture contents of 6.53 to 28.00%. In addition, one sample was dried in a vacuum oven to a residual moisture level of 0.74%. The Instron testing machine was used, and loading was either by cylindrical indenter, parallel plates, or spherical indenter. Three moisture-dependent parameters were evaluated from load-deformation relations obtained at different loading levels: (a) the linear limit load, up to which the load-deformation relation was linear; (b) the apparent modulus of elasticity; and (c) the modulus of deformability. Each of these parameters decreased with increase in moisture content of the kernel. Under the testing conditions employed, the major contributor to the mechanical properties observed was the horny endosperm; at low moisture levels this was very stiff. Increasing moisture content reduced the friction coefficient of the system and caused deformations to increase and moduli to decrease as pressure was applied. Results from tests on the floury endosperm indicated considerably lower values for the three parameters evaluated but were not consistent.

One of the important steps in commercial dry-milling of corn is the tempering of the kernels, during which the moisture of the corn is usually raised to 18 to 22% or above. The process toughens the bran and germ and facilitates their release in the degerminating step which follows. Factors such as moisture level and temper time and temperature affect the degerminator efficiency (1), but optimal conditions have not yet been established.

No information is available on changes in mechanical and viscoelastic properties of single kernels of corn during tempering. The previous work on mechanical properties of the single kernel was exploratory only (2,3,4).

In the present work, the mechanical and viscoelastic behavior of the individual corn kernel was studied as its moisture content gradually increased, in order to establish quantitative rheological parameters which would reflect the changes that take place.

MATERIAL AND METHODS

Yellow dent hybrid corn (WF9MST × H71) (Oh43RF × B37RF) was used for this study. It was planted in May 1964 on Crosby Bookston soil and harvested when at a little over 20% moisture with an ear-corn picker. The corn was air-dried to 10.53% moisture. It contained (% m.f.b.) protein, 9.79; oil, 4.55; crude fiber, 3.36; and ash, 1.32.

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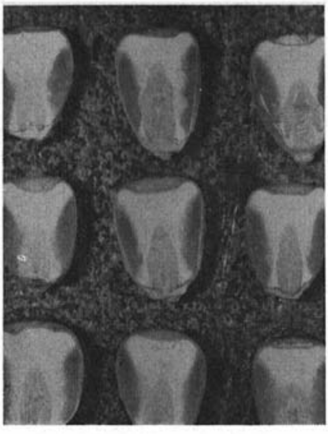
TABLE I. HYGROSCOPIC MOISTURE IN WHOLE CORN KERNEL, ENDOSPERM, AND GERM AT 72°F.

Relative Humidity %	Moisture Content: Whole Kernel % d.b.	Endosperm % d.b.	Germ % d.b.
11.1	6.53	6.97	6.50
32.9	9.75	10.47	8.47
54.6	12.24	12.66	8.71
75.5	16.80	17.70	15.13
92.5	25.90	24.30	33.50
97.0	28.00	27.40	45.60

Preparation of Samples

Hand-shelled center-cob kernels, free of visible cracks as tested by candling, were chosen for this study. The kernels were equilibrated over saturated salt solutions (5) at the following humidities: 11.1, 32.9, 54.6, 75.5, 92.5, and 97%. In addition, a sample was dried in a vacuum oven at 70°C. to constant weight (0.74% moisture). After equilibrium had been reached, as checked by absence of weight change in a sample of corn under each relative humidity, moisture contents of the whole kernel, germ, and endosperm were determined (air-oven at 103°C. for 72 hr., Table I). The germ-side surface of each kernel was lightly sanded and the kernels were glued, germ-side down, to a flat metal plate. A thin layer of Dupont Duco cement was used for kernels equilibrated at 54.6% r.h. or below, and Thiokol resin (Chemical Corp., Trenton, N. J.) was used for kernels at 75.5% r.h. and above, since the latter will dry at high-humidity atmospheres. After the glue had dried at the corresponding relative humidities, the upper surface of kernels was finely sanded parallel to the plate by means of a specially built mechanical sander. The exposed area consisted of horny endosperm, varying in depth, and floury endosperm at the dent (Fig. 1). The thickness



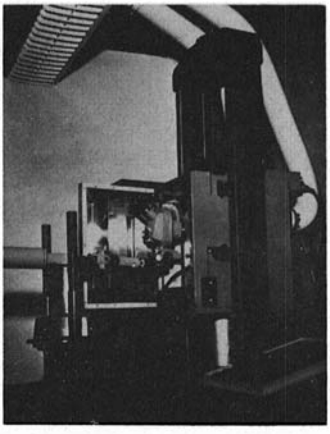


Fig. 1 (left). Plan view of slabs of the corn kernels mounted on testing plate.

Fig. 2 (right). Testing machine with environmental chamber and stereomicroscope.

of the kernels was controlled to 0.14 in. After the sanding, the specimens were returned to the corresponding humidity chambers until ready to be tested.

The mechanical properties of individual corn kernels were determined with an Instron table model testing machine equipped with the Instron environmental chamber (Fig. 2). The temperature throughout conditioning and testing was kept constant at 72°F. (± 1°). The relative humidity in the testing chamber was regulated by means of an Aminco Aire humidity-temperature control apparatus (American Instrument Co., Inc., Silver Springs, Md.) to the level used for conditioning the kernels.

THEORETICAL

When a corn kernel is compressed at a constant rate of deformation, a load-deformation curve is obtained which is linear over a considerable range. When a constant low load within the linear range is applied at a constant rate and immediately removed at the same rate, the deformation recorded upon loading is partly recovered (elastic) and partly residual (plastic and unrecovered elastic combined). Additional loading-unloading cycles show that the recovered deformation remains constant and the residual deformation gradually decreases to a constant value by the third cycle. At this point the kernel shows no additional plastic deformation and its behavior approaches that of an elastic body.

Under these conditions, the Boussinesq and Hertz solutions available for contact stresses in elastic bodies can be adapted to calculate an apparent modulus of elasticity for the corn kernel. The methods and their application to wheat grains were discussed elsewhere (6) but they will be reviewed here briefly.

Uniaxial Compression with a Cylindrical Rigid Indenter or a Spherical Indenter

Either a cylinder or a circular rigid indenter was pressed against the plane surface of the kernel on the horny endosperm. The apparent modulus of elasticity, E_a, of the compressed material when the cylindrical rigid indenter was used as the loading device was determined from the Boussinesq equation

$$E = \frac{P(1 - \mu^2)}{2\ a\ D} \tag{1}$$

where
P = load on the indenter (lb.)
μ = Poisson's ratio
a = radius of the indenter (in.)
D = displacement of the indenter (in.)

When the spherical indenter was used, the apparent modulus was determined from the Hertz equation given by

$$E = \frac{0.338k^{3/2}\ P(1 - \mu^2)}{D^{3/2}} \left(\frac{1}{R_1} + \frac{1}{R_1'} + \frac{1}{R_2} + \frac{1}{R_2'} \right)^{½} \tag{2}$$

where k is a constant, depending on the principal planes of curvature of the contacting bodies (7). Under testing conditions used, the radii of curvature of the corn kernel $R_1 = R_1' = \infty$ and the radii of curvature of the spherical indenter $R_2 = R_2' = 0.033$ in. Substituting these values and the value k = 1.3514, determined from the geometric properties of the contacting bodies (7), in equation 2,

$$E = \frac{4.16\ P(1-\mu^2)}{D^{3/2}} \tag{3}$$

Uniaxial Compression between Two Parallel Plates

The magnitude of unit contraction, ε, of the corn kernel under normal compressive stress σ is given by the equation

$$E = \frac{\sigma}{\varepsilon} = \frac{P/A}{D_e/H} \tag{4}$$

where A = contact area (sq. in.)
D_e = elastic deformation (in.)
H = initial thickness of the slab of the corn kernel (in.).

TESTING METHODS

Uniaxial compression was applied to mounted kernels with (a) the flat end of a cylindrical rigid indenter (0.016-in. diam.), (b) a parallel plate, and (c) a smooth spherical indenter (0.065-in. diam.).

The testing procedure in each method was similar and had two parts: A, compression of the kernel at a constant rate of deformation of 0.020 in. per min. up to loads where the load-deformation relation was clearly nonlinear; and B, loading-unloading cycling at the same constant rate of deformation to a constant load within the linear range of the curve, as determined by A above.

Fifty kernels at each of the seven moisture levels were used for each test. An Instron 200-lb. load cell was used for all testing. A 500-lb. load cell was employed for parallel-plate compression of dry corn. The load cells were calibrated before testing and their deflection was determined.

RESULTS

Load-deformation curves obtained for the horny endosperm by use of testing procedure A for 4 degrees of loading with the cylindrical indenter are shown in Fig. 3 (exact retracings). All four curves show curvature at the point of initial loading and then become linear up to a certain point. The fourth curve illustrates resultant deformation beyond the linear-limit load, the deformation increasing at a faster rate than the load. Similar curves were obtained with the spherical indenter and the parallel plate.

Assuming that ideally the loading curve will be linear even at the origin, a tangent to the linear portion of the curve was drawn; this made it possible to evaluate for each moisture level a linear limit load beyond which the load-deformation relation was no longer linear (Fig. 3). This parameter is summarized in Table II for the three testing methods at the different mois-

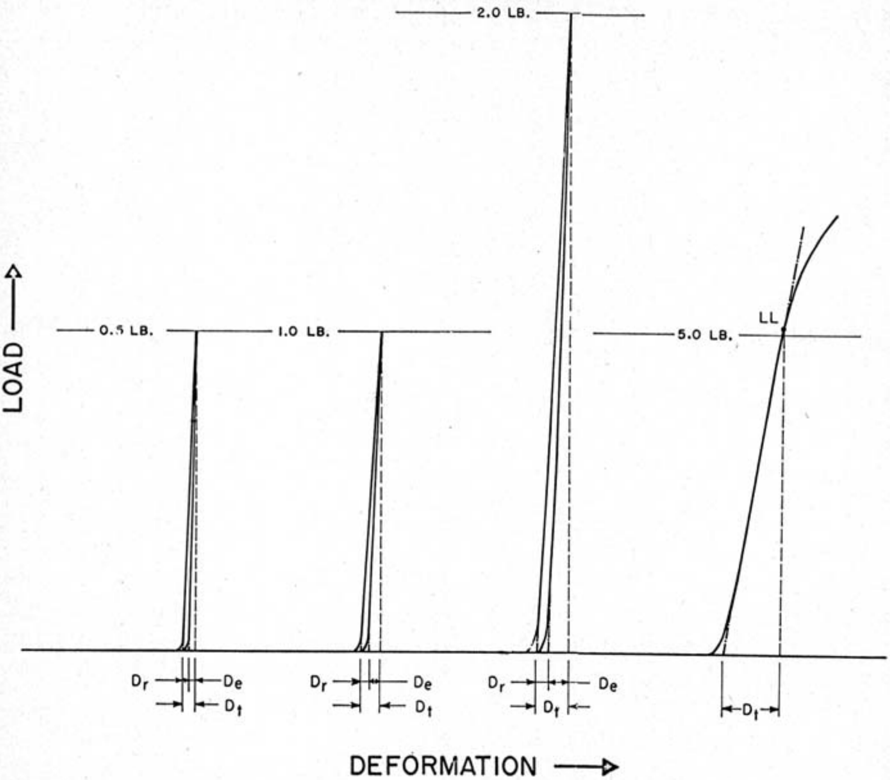


Fig. 3. Load-deformation curves obtained by uniaxial compression of the horny endosperm in slabs of corn kernels. Three left-hand curves illustrate deformation within the linear range; right-hand curve extends beyond the linear limit load. All curves made with the cylindrical indenter.

TABLE II. APPARENT MODULUS OF ELASTICITY, MODULUS OF DEFORMABILITY, AND LINEAR LIMIT LOAD FOR CORN KERNELS AT DIFFERENT MOISTURE LEVELS

Moisture Content % d.b.	Cylindrical Indenter			Parallel Plates			Spherical Indenter		
	E_a[a] (10^5 p.s.i.)	E_d[a] (10^5 p.s.i.)	LL Load (lb.)	E_a[b] (10^5 p.s.i.)	E_d[b] (10^5 p.s.i.)	LL Load (lb.)	E_a[c] (10^5 p.s.i.)	E_d[c] (10^5 p.s.i.)	LL Load (lb.)
00.74	1.48	1.29	13.80	2.37	1.24	..	15.95	9.80	38.2
03.35	n.d.	n.d.	11.40	n.d.	n.d.	400.0	n.d.	n.d.	28.7
06.53	1.36	0.90	7.20	2.11	1.10	307.0	12.10	7.02	18.5
09.75	1.64	1.16	5.99	2.63	0.88	..	12.00	5.98	13.0
12.24	0.96	0.47	4.05	1.50	0.57	223.0	11.10	4.15	10.0
16.80	1.25	0.71	3.48	1.07	0.56	154.0	7.45	2.94	8.8
25.90	0.89	0.58	1.97	0.80	0.20	66.6	3.07	0.55	3.9
28.00	0.56	0.24	1.90	0.49	0.12	47.0	1.16	0.14	3.9

[a] Values determined for 0.5-lb. load.
[b] Values determined for 50-lb. load.
[c] Values determined for 5-lb. load.

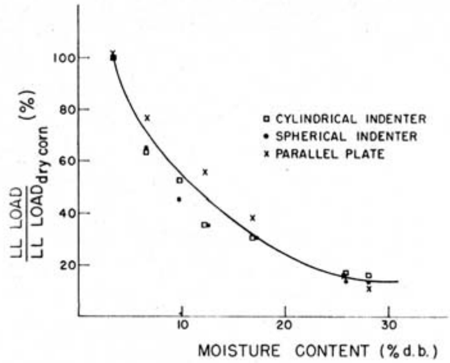


Fig. 4. Relative linear limit load as a function of moisture content of slabs of corn kernels.

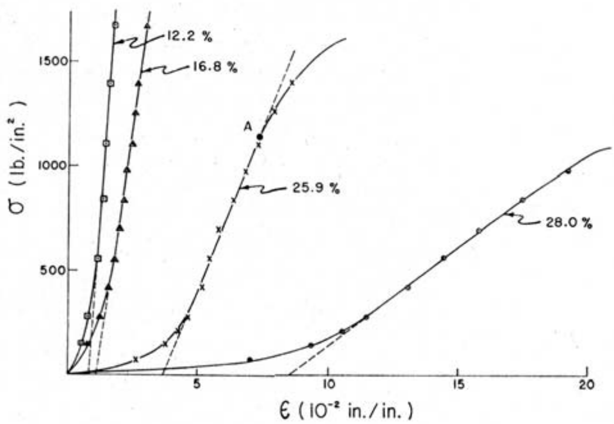


Fig. 5. Stress-strain relation for parallel plate testing of slabs of corn kernels at different moisture levels (% d.b.).

ture levels. A marked decrease in the linear limit load is observed with increase in moisture level of the corn. To compare the results in the three testing methods, the ratio of the linear limit load at each moisture level to the linear limit load of dry corn (3.35% d.b.) under each method was plotted against moisture content of the corn (Fig. 4)[3]. This graph shows that the relative decrease in linear limit load with moisture level over the range investigated is relatively independent of the loading device used.

The load-deformation curves obtained for the cylindrical and spherical indenters cannot be represented in a form of stress-strain curve, since the stresses and strains are not known. They can, however, be represented in this form for the parallel-plate tests, if the contact area is assumed to be constant throughout the loading process. These stress-strain curves for moisture content range of 12 to 28% (d.b.) are shown in Fig. 5. In these curves, the nonlinearity at the initial stress-strain relation was not elimi-

[3] The linear limit load of nearly bone-dry corn slabs under plate loading exceeded the load cell capacity. Therefore, all loads were referred to the linear limit loads of 3.35% m.c. corn. These values are given in Table II.

nated, showing a marked increase at the high moisture levels. A stress-strain relation, as illustrated by the 25.9% m.c. curve, shows the proportional limit, point A, beyond which the stress is no longer proportional to the strain. This point represents a strength value of the material, a value which can be used to calculate the resilience, or the internal work or strain energy per unit volume of the material required to stress the material to the proportional limit. Since these values could be computed only for the parallel-plate testing, they were not shown quantitatively. The value of slope of curve at each moisture level is equal to the apparent modulus of elasticity.

The loading-unloading curve to a constant load within the linear range (testing procedure B) showed that part of the total deformation, D_t, was recoverable and therefore might be considered elastic deformation, D_e, and part was residual, D_r (Fig. 3). The values of these two deformations were determined by extrapolating the tangent to the linear load-deformation curve to the point of zero load as described above. These values, obtained from the first loading-unloading cycle, are summarized in Table III. Cycling showed no significant change in the elastic deformation; the value of the residual deformation gradually decreased to a constant value by the third cycle.

TABLE III. EXPERIMENTAL RESULTS FOR UNIAXIAL COMPRESSION OF CORN KERNELS AT DIFFERENT MOISTURE LEVELS

Moisture content % (d.b.)	0.74	6.53	9.75	12.24	16.80	25.90	28.00
CYLINDRICAL INDENTER							
Elastic Deformation[a]							
Mean (10^{-3} in.)	0.21	0.23	0.19	0.32	0.25	0.35	0.56
Std. dev.		0.20	0.12	0.14	0.24	0.15	0.30
Coeff. var. %		43.1	31.9	21.1	47.0	20.9	26.6
Residual deformation[a]							
Mean (10^{-3} in.)	0.03	0.12	0.08	0.34	0.19	0.19	0.77
Std. dev.		0.09	0.07	0.16	0.16	0.06	0.38
Coeff. var. %		78.6	87.8	46.8	81.8	33.1	49.0
PARALLEL PLATES							
Elastic deformation[b]							
Mean (10^{-3} in.)	0.41	0.46	0.37	0.65	0.92	1.23	1.99
Std. dev.		0.17	0.22	0.14	0.26	0.25	0.40
Coeff. var. %		54.5	47.2	21.3	28.1	20.3	20.2
Residual deformation[b]							
Mean (10^{-3} in.)	0.37	0.42	0.74	1.08	0.83	3.74	6.40
Std. dev.		0.14	0.18	0.60	0.32	1.10	1.15
Coeff. var. %		33.6	22.2	55.7	39.1	32.6	18.0
SPHERICAL INDENTER							
Elastic Deformation[c]							
Mean (10^{-3} in.)	0.55	0.66	0.67	0.70	0.92	1.62	3.18
Std. dev.		0.07	0.05	0.09	0.08	0.27	0.48
Coeff. var. %		11.6	8.1	13.1	8.3	16.6	15.1
Residual Deformation[c]							
Mean (10^{-3} in.)	0.21	0.29	0.40	0.67	0.77	3.67	10.14
Std. dev.		0.14	0.11	0.21	0.14	0.76	2.28
Coeff. var. %		47.7	26.2	31.0	17.4	22.4	22.5

[a] Values determined for 0.5-lb. load.
[b] Values determined for 50-lb. load.
[c] Values determined for 5-lb. load.
Each value is a mean of 50 tests.

Calculation of the Modulus

Equations 1, 3, and 4 were derived for purely elastic, homogeneous, and isotropic materials, under specified conditions. Their application to an agricultural material requires some approximations which have been discussed for wheat grain (6) and can equally be applied to the corn kernel. Yet, even with the testing techniques used, where the load levels were low and applied for very short time, the corn kernel's behavior was not purely elastic. Therefore, the values calculated from eqs. 1, 3, and 4 were termed "apparent modulus of elasticity," E_a, and are summarized in Table II. Equations 1 and 3 require the value of Poisson's ratio, which has been found to be about 0.4 for corn of 10% m.c. (4) and will be assumed constant over the different moisture levels. The contact area, A, in eq. 4 was measured under a microscope by means of a calibrated grid-pattern disc. When the loading plate was coated with Prussian Blue, the print on the exposed area facilitated the measurement. A mean value of 0.072 in.^{2} (s.d. 0.0175) for 60 kernels was taken.

In addition, a "modulus of deformability," E_d, was calculated, as suggested by Shpolyanskaya (8) for wheat grain. This modulus takes into account the total deformation of the material in the first loading cycle.

The values of E_a and E_d decreased with increase in moisture content of the corn. There is some agreement between the values obtained by the cylindrical indenter and parallel plates, as seen from the plot in Fig. 6, but the

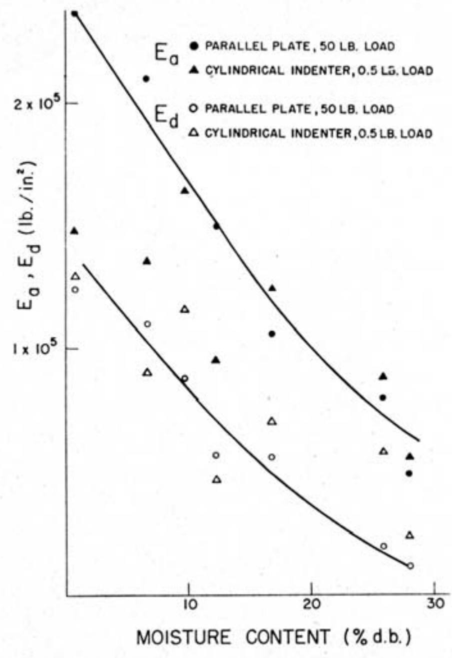


Fig. 6. Apparent elastic modulus, E_a, and modulus of deformability, E_d, as a function of moisture content for cylindrical indenter and parallel plate compression of slabs of corn kernels.

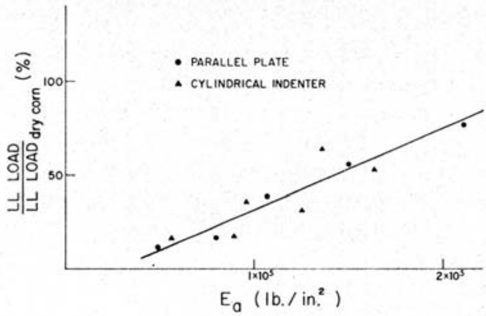


Fig. 7. Relative linear limit load vs. apparent modulus of elasticity at the corresponding moisture levels for cylindrical indenter and parallel plate compression.

TABLE IV. APPARENT MODULUS OF ELASTICITY, MODULUS OF DEFORMABILITY, AND LINEAR LIMIT LOAD FOR FLOURY ENDOSPERM AT DIFFERENT MOISTURE LEVELS AND CYLINDRICAL INDENTER LOADING

Moisture Content % d.b.	E_a[a] (10^5 p.s.i.)	E_d[a] (10^5 p.s.i.)	LL Load (lb.)
6.53	0.91	0.47	2.14
9.75	0.91	0.34	1.72
12.24	0.68	0.56	1.22
16.80	0.97	0.30	0.86
25.90	0.32	0.12	0.82

[a] Values determined for 0.5-lb. load.

values obtained by spherical indenter compression are 2 to 8 times as high. The same trend has been found for similar tests of wheat grain (6) and needs further clarification. The decrease in moduli as the moisture content increases is similar to that obtained for the change in relative linear limit load (Fig. 4). When the relative linear limit load was plotted against the apparent modulus of elasticity at the corresponding moisture levels for the cylindrical indenter and parallel plates, a linear relation was observed (Fig. 7). Thus, all the three parameters decrease with increase in moisture content of the corn.

Mechanical tests conducted on the floury portion by means of the cylindrical indenter showed that values of moduli and linear limit loads were much lower than those for the horny endosperm (Table IV). However, except for the linear limit load, the data do not show any consistent trend as was observed in the case of horny endosperm.

DISCUSSION

The results obtained in this study reflect mainly the mechanical properties of the endosperm, since the hull was removed prior to testing and the positioning of the kernel was such that only the endosperm was in direct contact with the loading devices. Table II shows that at any given moisture level the values of E_a and E_d are relatively in the same order of magnitude for the cylindrical indenter and the parallel plates. These values, particularly for the lower moisture levels, are considerably higher for the case of the spherical indenter. With the depth of indentation and the radius of the indenter known, the surface unit pressures under the spherical indenter were estimated and compared with those under the two other loading devices. As suspected, the values of unit pressures for the case of spherical indenter were much greater than those for the other two cases. Figures 3 and 5 show that because of the sigmoidal shape of the curves, the use of lower levels of unit surface pressure can result in smaller values of modulus (initial part of the curves), whereas the use of higher values of unit pressure, as has apparently been the case with the spherical indenter, can give higher values of modulus. Figure 5 shows that such a trend would be less pronounced for higher moisture levels. The reported modulus values for spherical indenter support this observation. In other words, if the force and deformation values used in eq. 2 were taken from a line tangent to the initial portion of the force-deformation curves, the values obtained by the spherical indenter would have been closer to those by the other types of loading devices. Later indentation experiments (9) with a spherical indenter of the same size, as well as tensile and dynamic tests of rectangular segments of horny endosperm subjected to very low levels of stress, have given modulus values in the range reported for cylindrical indenter and parallel plates in this paper.

The endosperm comprises about 80 to 84% of the weight of the corn kernel (10). In dent corn the horny endosperm lies chiefly at the sides and back of the kernel. Therefore, the results represent mainly properties of the horny portion. Since starch and protein are the major constituents of the corn endosperm, some of their characteristic properties as related to the present study should be pointed out. Comparison of a sorption isotherm of corn starch (11) with that of corn endosperm (5) shows that the water sorption of starch is, by about 2%, absolute, higher throughout the relative humidity range, the general shape of the isotherm being otherwise the same. Thus, the starch constituent in corn endosperm plays an important role in the water-sorption mechanism.

Microscopic study of dent corn endosperm has shown (12) that the individual starch granules in both the horny and the floury endosperm lay completely embedded in a matrix composed largely, if not wholly, of protein, and the majority of the molecules in the network are oriented. It is generally recognized that powerful intramolecular binding forces exist in crystalline or oriented high-polymeric substances, and this accounts for their toughness. This property of zein, the alcohol-soluble portion of corn protein, was once exploited in the manufacture of a synthetic fiber sold under the trade name Vicara. Zein fibers are crystalline and can be oriented fairly well by stretching (13).

Microscopic studies have also shown that the protein content is considerably greater in horny endosperm than in floury endosperm. The ratio of starch to protein is approximately 11:1 in floury and 6:1 in horny endosperm (12). Also, the shape and size of the starch granules vary with their location in the endosperm. Granules in the floury endosperm are large and loosely packed; those in the horny endosperm are relatively smaller and tightly packed.

From the above considerations, the mechanical properties discussed in this paper should be largely related to the whole proteinaceous matrix of the horny endosperm, as well as the shape, size, and compactness of the starch granules. At low moisture levels the corn kernel is very stiff, as its deformation in the elastic range is relatively small (Fig. 5, curve at left for example). When water molecules enter the chain units which are very close to each other, they have to rearrange their relative positions and the resistance to motion depends on details of local geometry. Apparently such geometrical rearrangement of the chain units affects the mechanical properties. Another factor which might affect these properties, as related to moisture content, is the friction coefficient. Behavior of the dry endosperm can be compared to an undiluted polymer with a relatively high friction coefficient. When the moisture level is increased, the friction coefficient becomes smaller, since some of the nearest neighbors of a polymer segment are solvent molecules and are much more mobile (14). Under such conditions the deformations under a given load gradually increase and the moduli decrease with increase in the number of water molecules present. Similar behavior has been observed experimentally in wood (13), in wheat gluten (15), in change of bulk modulus of corn kernel (4), and in corn and wheat (2). In none of these examples has a satisfactory theoretical explanation been given.

At this stage of the research the corn was not brought in direct contact with liquid water as in the tempering process, but, for better-defined conditions, was equilibrated at different relative humidity atmospheres. The two highest moisture levels (26 to 28% d.b.) are within the range used in the commercial dry-milling process, although differences exist as regards distribution of moisture within the kernel. Therefore the changes in the parameters shown here—namely, decreases in apparent modulus of elasticity, modulus of deformability, and linear limit load—should be representative to a degree of the changes taking place in the commercial process.

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