HYSTERESIS EFFECTS IN MIXTURES OF WHEATS TAKEN FROM THE SAME SAMPLE BUT HAVING DIFFERENT MOISTURE CONTENTS[1]

J. R. HART[2]

ABSTRACT

Hysteresis effects are studied in mixtures of dyed and undyed kernels of wheat taken from the same sample and having different moisture contents. The hysteresis effect is the difference in moisture content between dyed and undyed kernels in a mixture held in a sealed container until no further changes in moisture contents occur. Mixtures having an original difference of 4% or greater showed a difference of 0.76% in moisture at equilibrium. The equilibrium difference decreases as the original difference in moisture content decreases below 4%, reaching a value of −0.28% at 0.00% original difference. Equilibrium differences decrease with increase in temperature. Application of pressure or using different proportions of high- and low-moisture wheat in the mixtures has little effect. When mixtures have equal weights of large and small kernels, greater equilibrium differences occur when the larger kernels have the higher original moisture content. Repeated wetting and drying before mixing has only slight effect.

The usual procedure in studying hysteresis effects in wheat and other grains has been to establish adsorption and desorption isotherms for the sample studied (1–4). A portion of the sample is brought to equilibrium with different atmospheres, each having a relative humidity that is successively higher (adsorption) or successively lower (desorption). The moisture content of the sample is determined at each relative humidity. Adsorption and desorption curves are prepared by plotting moisture contents against the corresponding equilibrium relative humidities. The hysteresis effect is considered to be the difference in moisture content between the adsorbing and desorbing portions of the sample at any given r.h.

The conditions under which hysteresis manifests itself in the normal storage and handling of wheat are more evident in the methods of study used by a few other investigators. In one, two portions of wheat of different varieties, having different moisture contents, are held in the same closed container until equilibrium is reached (5). In others, mixtures of two types of wheat, of different moisture contents, which differ in color or kernel size so that they can be separated by hand-picking (6) or by sieving (7), are held in a closed container until they reach equilibrium with each other. The differences in moisture con-

[1]Manuscript received February 20, 1964.

[2]Research Chemist, Field Crops and Animal Products Branch, Market Quality Research Division, AMS, USDA, Beltsville, Maryland.

tent at equilibrium constitute the hysteresis effect. All types of wheat, however, may not be affected by hysteresis to the same extent; nor is the effect of differences in kernel size known. A more accurate approach would be to use mixtures of different portions, of the same sample, having different moisture contents. If the kernels in one of the portions are colored with dye, they may be separated from the mixture by hand-picking; such mixtures were used in the present study.

The purpose of this investigation was to determine moisture hysteresis in mixtures of wheat kernels from the same sample, but having different moisture contents. Various factors which might affect hysteresis were also investigated.

Materials and Methods

Ten samples of hard red winter wheat, ranging in moisture content from 11.64 to 12.35%, were used. All moisture determinations were made by the official air-oven method of the U.S. Department of Agriculture (8).

Part of each sample was dyed. Approximately 100 g. of wheat in a beaker was covered with a solution containing 0.0675 g. gentian violet dye per liter of water. The beaker was immediately emptied onto a sieve and the liquid drained off. The wetted kernels were then spread in one layer on paper towels, and paper towels were pressed down on top of them to absorb surface liquid. After surface drying, they were held in the refrigerator in a Mason jar. The moisture content was never greater than 16%. A germination test indicated that the absorption of the dye did not affect the viability of the seeds. To obtain the desired differences in moisture content in the dyed and undyed parts of a sample, moisture was increased in a humidity cabinet or decreased by drying which would normally be encountered in stored grain. The r.h. in the cabinet was approximately 90% at 80°F. Samples were dried on top of an oven at about 104°F. The wetted or dried wheat was always held 4 or 5 days before being mixed. The part of the mixture having the higher original moisture content was in some cases the dyed kernels and in others the undyed.

Moisture contents of each portion were determined, and 50 g. each of the dyed and undyed parts of a sample were placed in a tightly sealed Mason jar and thoroughly mixed by shaking and inverting the jar. The jar was held in a constant-temperature room at 76°±1°F.

At intervals, tests were made to determine whether or not the moisture content had become constant in the dyed and undyed parts of the mixture. About 5 g. of the mixture was poured onto a sheet

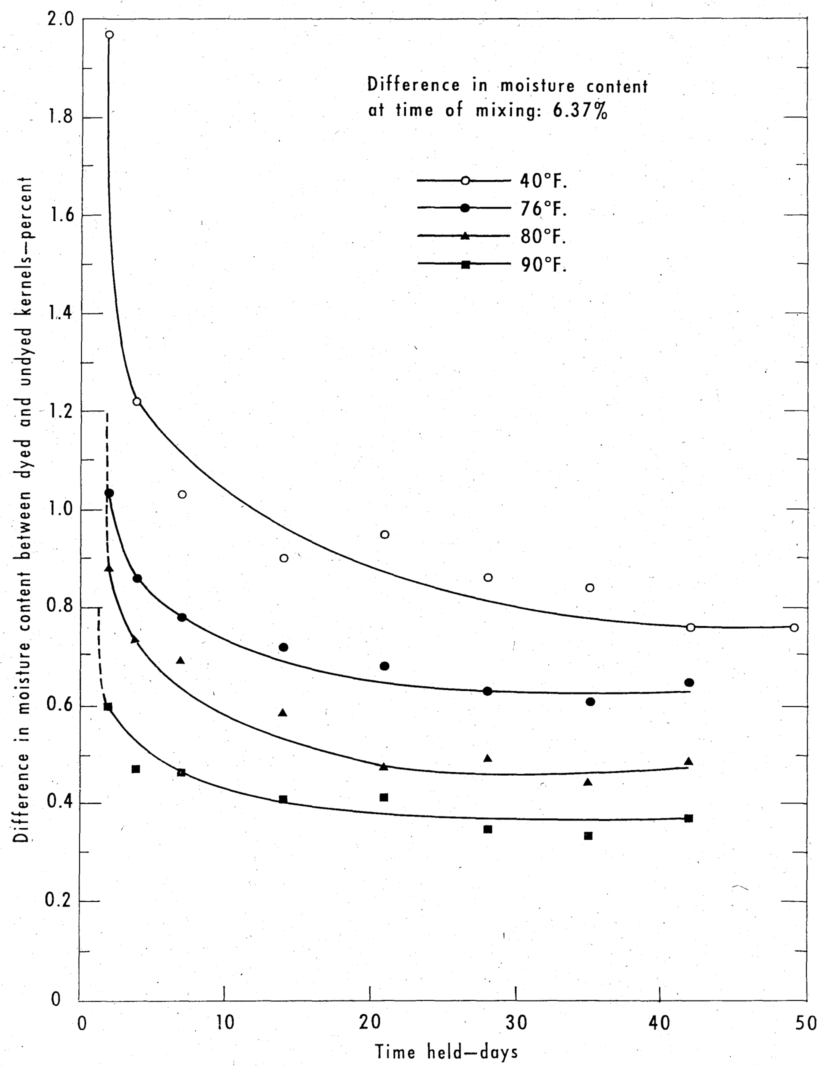

Fig. 2. Effect of temperature on the relationship between the time mixtures are held and the difference in moisture content of dyed and undyed kernels.

Varying the Proportions of High-Moisture and Low-Moisture Wheat in a Mixture. Mixtures of dyed (6.19% moisture) and undyed (12.12% moisture) wheat from the same sample, containing different proportions of the high- and low-moisture wheat, were prepared. Each mixture was held at 76°F. until constant weights were attained. Results are given in the table below. As the proportion of high-moisture

Composition by Weight of Mixtures		Difference in Moisture Content at Equilibrium
Dyed	*Undyed*	
%	%	%
87.5	12.5	0.64
75.0	25.0	0.79
62.5	37.5	0.87
50.0	50.0	0.87
37.5	62.5	0.89
25.0	75.0	0.89
12.5	87.5	0.91

kernels in the mixture increases, the difference in moisture content at equilibrium increases.

Kernel Size. Sieves with successively larger openings were used to separate samples of wheat into portions having different kernel sizes. In the portions having the largest kernels the range of size was 37.0 to 37.4 g. per 1,000 kernels. The smallest kernels ranged from 12.5 to 12.8 g. per 1,000 kernels. Mixtures were prepared which contained 20 g. each of the smallest and largest kernels taken from the same sample. Trash and broken kernels were hand-picked from the largest. In some cases they were not picked from the smallest kernels. Table I gives the difference in moisture content at equilibrium for each of the mixtures. When the large kernels in the mixture have the higher moisture content, the difference in moisture content at equilibrium between the dyed and undyed kernels is greater than when the large kernels have the lower initial moisture content.

TABLE I

RELATIONSHIP OF DIFFERENCE IN WHEAT KERNEL SIZE TO DIFFERENCE IN MOISTURE CONTENT AT EQUILIBRIUM

Composition and Original Moisture Contents of Mixture: Dyed Portion		Undyed Portion		Difference in Moisture Content at Equilibrium
	%		%	%
Large	14.78	Small	7.12	0.85
Large	13.85	Small	7.27	0.95
Large	14.78	Small and broken kernels; trash	6.89	0.87
Large	14.78	Broken kernels and trash only	7.00	0.71
Large	13.85	Small and broken kernels; trash	6.32	0.93
Small	6.57	Large	15.05	0.90
Small and broken kernels; trash	6.37	Large	11.79	1.02
Small	16.88	Large	8.16	0.48
Small and broken kernels; trash	14.08	Large	7.98	0.43
Large	8.31	Small	14.26	0.29

Varying the Original Difference in Moisture Content between Dyed and Undyed Kernels. Mixtures for these experiments were prepared by using, for the higher-moisture portion, wheat which had approached equilibrium while gaining moisture and, for the lower-moisture portion, wheat approaching equilibrium while losing moisture. Equal weights were mixed and held at 76°F. until equilibrium was reached. The initial difference in moisture content in the 75 mixtures tested ranged from 0 to 13.90%. Figure 3 shows how the differences in moisture content at equilibrium varied with the initial differences. When

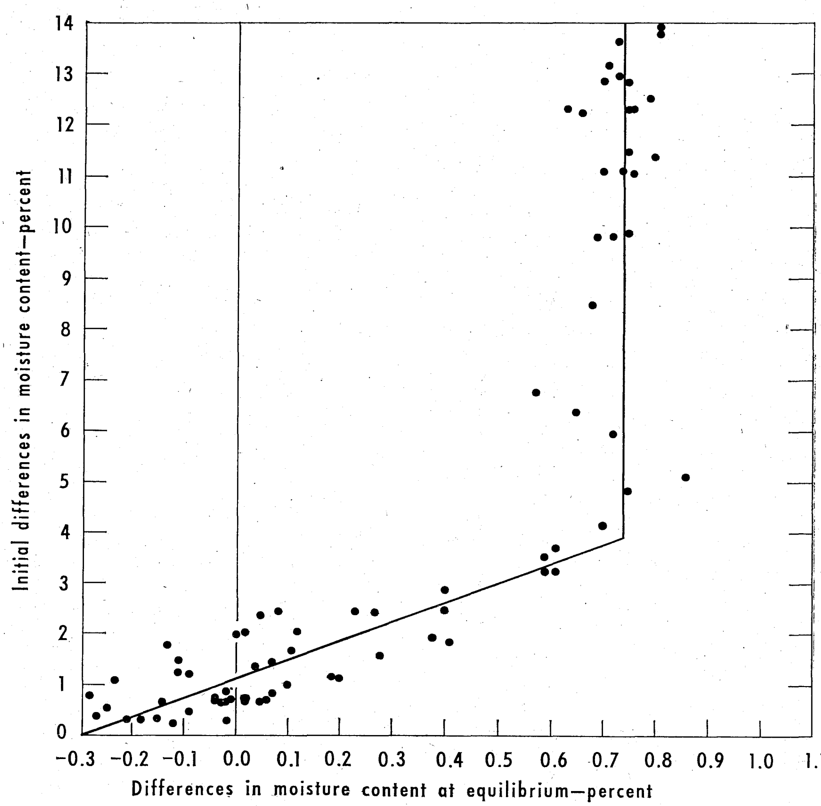

Fig. 3. Relation between initial difference in moisture content in a mixture and difference at equilibrium.

initial differences are in excess of approximately 4% moisture, the differences at equilibrium are constant. If the part of a mixture having the greater initial moisture content still has the higher moisture at equilibrium, this difference is considered to be positive. If it has the lower moisture content at equilibrium, the difference is negative.

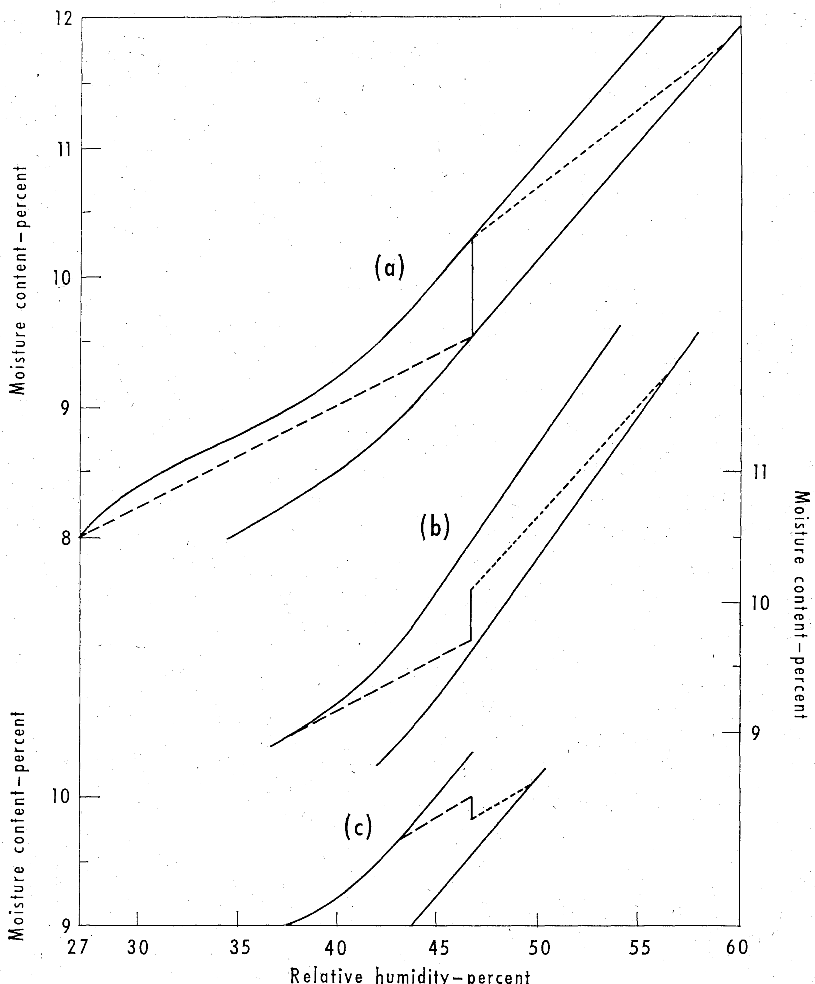

Fig. 4, a, b, and c. Short sections of the adsorption and desorption isotherms of Fig. 1, to illustrate how differences in moisture content at equilibrium decrease as initial differences decrease.

In Fig. 4 (a, b, and c), small sections of the adsorption and desorption isotherms of Fig. 1 are shown, and also the paths taken by the adsorbing (dotted-line) and desorbing (broken-line) portions as they change from one isotherm to the other after being placed in the mixture. Of course, the exact paths are not known, but the initial and terminal points can be determined from the experimental data. Such paths were observed by Rao (10) in his study of hysteresis in titania gel-water systems. The vertical lines joining the terminal ends of the paths represent the differences in moisture content at equilibrium. In (a) the transition is completed from one isotherm to the other and the maximum difference at equilibrium is shown. The paths reach the same r.h. before the transition is completed in (b) and the difference at equilibrium is less. In (c) the same r.h. is reached where the moisture content of the desorbing portion is above that of the adsorbing portion; so the difference at equilibrium is negative.

Repeated Raising and Lowering of Moisture Content. Reports in the literature indicate that, in the case of some cereals (11) and cellulose (12), the adsorption and desorption isotherms tend to conform more closely to each other with successive changes in moisture content. Hubbard *et al.* (4) found no effect when using ground wheat.

Ten samples were used to determine the effect by the procedure followed in this investigation. Moisture contents were alternately raised to about 16% in the humidity cabinet and lowered to about 6% with mild heat. Equal weights of dry and wet wheats were used to form mixtures representing each sample. They were held at 76° to equilibrium and the differences in moisture content were determined. Four series of mixtures were tested, each representing one, two, three, or four moisture-content changes. Table II shows the composition of each series in terms of the moisture changes, the range of differences, and the average difference in moisture content at equilibrium.

TABLE II

EFFECT OF REPEATED MOISTURE-CONTENT CHANGES IN WHEAT, PRIOR TO MIXING, ON THE EXTENT OF HYSTERESIS

	Composition of Mixtures in Each Series		Range of Differences in Moisture Content at Equilibrium	Average Difference at Equilibrium
	Dyed Portion	Undyed Portion	%	%
1.	Wetted	Dried	0.68 to 0.80	0.74
2.	Wetted-dried	Dried-wetted	0.56 to 0.65	0.60
3.	Wetted-dried-wetted	Dried-wetted-dried	0.50 to 0.78	0.61
4.	Wetted-dried-wetted-dried	Dried-wetted-dried-wetted	0.29 to 0.58	0.49

The results indicate that repeated wetting and drying does tend to decrease the hysteresis effect in mixed wheats to a slight extent.

Discussion

The hysteresis effects reported here are lower than those obtained by other workers who used isotherms obtained by bringing wheat to equilibrium with successively higher or lower relative humidities. Babbitt (1) found a difference of approximately 4% between adsorption and desorption isotherms at r.h. 44% and temperature 77°F. Hubbard *et al.* (4) found a difference of 1.28% under the same conditions. They also found that at temperatures from 77° to 95°F. there was practically no change in this difference, whereas Fig. 2 shows a continuous decrease in the difference as temperature increases. Some of the differences reported by workers using mixtures of two different types of wheat are not materially different from the results reported here. Fisher and Jones (6) reported differences ranging from 0.58 to 1.85% for different mixtures. Fairbrother (7) found differences of approximately 2%.

The fact that compression of the mixtures failed to decrease the hysteresis effect suggests that moisture interchange takes place through the intergranular air. This view was held by Fisher and Jones (6). Perhaps the lower effects obtained with mixtures can be explained by the overlapping at contact points of the "stationary film of air" surrounding each kernel which was described by Lewis (13). The transfer of evaporated water to and from surfaces and the transfer of heat would be more complete.

The initial rate at which a high-moisture wheat loses moisture is much greater than the initial rate at which a low-moisture wheat regains moisture when the two are placed in the same closed container (14). Thus, the changing r.h. would stay closer to equilibrium with the high-moisture wheat than with the low. A time would come when the high-moisture wheat would reach a moisture content corresponding to equilibrium with the then existing r.h. while the low-moisture wheat would still be gaining moisture. This would produce a reversal of the sorption process and a decrease in the difference in moisture content at equilibrium. At higher temperatures, where sorption rates are higher, the effect of the reversal would be greater. In following the moisture changes in mixtures of dyed and undyed wheat, it was found that the moisture content of the higher-moisture kernels reached a minimum value and then increased, usually by 0.1% or less, in succeeding determinations.

The sorption of water by wheat depends upon the area of wheat surface, the rate of diffusion to the interior, and the adsorbing capacities of the wheat tissues. The data shown in Table I could be explained if it is assumed that the surface layers of the kernels have low adsorbing power relative to the endosperm. Thus, small kernels having relatively large total surface area (15) and small amounts of endosperm would lose water more readily to large kernels having small total surface and large amounts of endosperm. The probability that the small kernels differ from the large kernels in their chemical and physical characteristics cannot be ruled out as a factor in the sorption behavior of these mixtures. Small kernels often result from frost damage, immaturity, and other types of crop damage which would affect their structure and chemical composition. When two varieties of wheat are used in mixtures, great variation is found in the hysteresis effect. Two investigators found differences in moisture content ranging from 1.72 to 2.40% (7) and from 0.58 to 1.85% (6), respectively.

In the case of mixtures having different proportions of dyed and undyed kernels (see second unnumbered table), when the total surface area of the high- or low-moisture portion is high, the amount of endosperm is also high. The result is that the effect of one factor is canceled out by the effect of the other; hence, there is little change in the hysteresis effect with change in proportions.

Literature Cited

1. BABBITT, J. D. Hysteresis in the adsorption of water vapour by wheat. Nature **156:** 265–266 (1945).
2. BREESE, M. H. Hysteresis in the hygroscopic equilibria of rough rice at 25°C. Cereal Chem. **32:** 481–487 (1955).
3. HOUSTON, D. F., and KESTER, E. B. Hygroscopic equilibria of whole grain edible forms of rice. Food Technol. **8:** 302–304 (1954).
4. HUBBARD, J. E., EARLE, F. R., and SENTI, F. R. Moisture relations in wheat and corn. Cereal Chem. **34:** 422–433 (1957).
5. PAP, L. Hygroscopicity of wheat. Cereal Chem. **8:** 200–206 (1931).
6. FISHER, E. A., and JONES, C. R. A note on moisture interchange in mixed wheats, with observations on the rate of absorption of moisture by wheat. Cereal Chem. **16:** 573–583 (1939).
7. FAIRBROTHER, T. H. The influence of environment on the moisture content of flour and wheat. Cereal Chem. **6:** 379–395 (1929).
8. UNITED STATES DEPARTMENT OF AGRICULTURE. Air-oven and water-oven methods specified in the official grain standards of the United States for determining the moisture content of grain. U.S. Dept. Agr., Service and Regulatory Announcement No. 147 (1941).
9. KETCHUM, M. S. The design of walls, bins and grain elevators (3rd ed.). McGraw-Hill: New York (1919).
10. RAO, K. S. Hysteresis in sorption. II. Scanning the hysteresis loop. Titania-gel water system. J. Phys. Chem. **45:** 506–512 (1941).
11. RAO, K. S. Hysteresis in sorption. VI. Disappearance of the hysteresis loop. The role of elasticity of organogels in hysteresis in sorption. Sorption of water on some cereals. J. Phys. Chem. **45:** 531–539 (1941).
12. SHEPPARD, S. E., and NEWSOME, P. T. The sorption of water vapor by cellulose and its derivatives. J. Phys. Chem. **33:** 1817–1835 (1929).
13. LEWIS, W. K. Evaporation of a liquid into a gas. Chem. and Met. Eng. **27:** 112–114 (1922).
14. ANKER, C. F., GEDDES, W. F., and BAILEY, C. H. A study of the net weight changes and moisture content of wheat flour at various relative humidities. Cereal Chem. **19:** 128–150 (1942).
15. HART, J. R., and FEINSTEIN, L. A method for the determination of the surface area of wheat kernels. Cereal Chem. **35:** 395–399 (1958).