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For description of the chemical designation of most of the products listed the reader is referred to paper 8 by McBain and Merrill and to paper 12 by Van Antwerpen in the References above; also to the "Handbook of Material and Trade Names" by O. T. Zimmerman and I. Lavine (Industrial Research Service, Dover, N. H., 1946).

Hygroscopic Equilibrium of Cottonseed

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Introduction

ATMOSPHERIC conditions in sections of the cotton-producing area during the harvest season are often such that the seed may contain more moisture than is safe for storage. Such seed may undergo spontaneous heating and rapid deterioration. It is common practice under these conditions to draw air through the seed piles to reduce the temperature and lower the moisture content of the seed. If, however, the air drawn through the seed has a high relative humidity, it is conceivable that the moisture content of the entire seed pile might be increased instead of decreased, thus aggravating rather than mitigating the deteriorative processes. Investigations on the storage and respiration of cottonseed have shown that moisture content is a dominant factor in the biological activity of this seed. Cottonseed of high moisture content respires more vigorously, develops free fatty acids more rapidly, and generally deteriorates to a greater extent upon storage than seed of low moisture content (1, 2). Cottonseed like most other seeds tends to assume a moisture content in equilibrium with that of the surrounding atmosphere. Consequently, the relative humidity, the nature of the hygroscopic equilibrium, and the rate at which it is attained are of considerable importance in determining the storage properties of the seed.

Thornton and Briggs (3) measured the effect of the relative humidity of air circulating through cottonseed on the rate of absorption of moisture. Their experiments were of relatively short duration (165-180 hours) and, except where air of low relative humidity was used, equilibrium was not attained. Thornton and Bishop (4) attempted to use static-air conditions to achieve equilibrium. Here, too, equilibrium was not attained at the end of their experiments (675 hours) and the only conclusion which could be drawn was that the rate of absorption of water in cottonseed increased with rise in temperature. Franco (5) determined the hygroscopic equilibrium of the I. A. 7387 variety of cottonseed grown in the state of São Paulo, Brazil. His experiments were conducted at 19°C. and lasted 20 days, the period which was required to attain equilibrium. Simpson and Miller (6) determined the equilibrium moisture content of Stoneville variety of cottonseed by storing samples at 25° C. in air of known relative humidities maintained by sulfuric acid solutions. Moisture determinations were made at the end of 8 and 12 weeks of storage. The results of the analysis after equilibration for 12 weeks were considered to be equilibrium values.

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In this investigation the hygroscopic equilibrium of cottonseed was determined at 26° C. in air adjusted to constant relative humidities within the range 31 to 93%. The distribution of moisture in cottonseed between the kernels and hulls was also determined within the above mentioned range of relative humidities.

Materials and Methods

Saturated solutions of pure salts and salt mixtures provide an excellent means of obtaining a wide range of relative atmospheric humidities in closed spaces. Saturated salt solutions are superior to sulfuric acid solutions for this type of investigation because the former are usually not corrosive and are easier to handle, and because they maintain a constant relative humidity of air in contact with materials of high or low moisture content as long as an excess of the solid salt persists. Spencer (7) lists a number of saturated salt solutions which maintain relative humidities ranging from 30% to 93%. The salts used in this investigation and the relative humidity values of their saturated solutions at 25° C. are listed in Table I.

TABLE I

Relative Humidity of Confined Air in Contact With Saturated Solutions of Various Salts and Salt Mixtures at 25° C.

Salts	Relative humidity
	%
$CaCl_2 \cdot 6H_2O$	31
K_2CO_3	43
NH_4NO_3	62
$NH_4Cl + KNO_3$ (mutually sat.)	71.2
$(NH_4)_2SO_4$	81.1
$NH_4H_2PO_4$	93

The cottonseed used in this investigation was D & PL-45 variety from the 1944 crop grown at the Delta Branch of the Mississippi Agricultural Experiment Station, Stoneville, Miss. The seed as received contained 9.55% moisture. Analysis on a moisture-free basis gave: nitrogen, 3.49%; free fatty acids, 0.80%; lipids, 24.32%; ash, 4.17%; and crude fiber, 23.35%. Germination was practically 100%.

Saturated salt solutions, together with an excess of solid salt, were placed in the bottoms of large desiccators, and 200 g. samples of cottonseed were suspended above these solutions by means of wire gauze mats. The desiccators were kept in a room maintained at 26° C. Every few days they were opened to remove duplicate samples of 5-10 g. each which were weighed immediately in closed moisture dishes. All moisture determinations were made by heating the products at 101° C. for 16 hours in a forced-draft oven.

At the end of 36 days a 10-15 g. sample of seed was removed from each humidifying chamber. This sample was quantitatively separated into meats and hulls which were transferred to previously tared moisture dishes. Inasmuch as the separation of meats and hulls[2] required about 20 minutes, these dishes were replaced in the humidifying chamber from which the seed was originally taken for an additional 24 hours to assure that equilibrium with the humidified air was attained. At the end of the 24-hour period the moisture contents of the meats and hulls were determined in the same manner as with intact seed. The moisture content of the intact seed calculated from the weighed sum of the moisture contents of the meats and hulls was in excellent agreement with the experimentally determined values on the intact seed. The meats from the dried seed were then extracted in a Butt extractor to determine the content of oil. Percentages of oil in the seed obtained by fuming followed by extraction indicated that all the oil was contained in the meats.

Results

The effect of relative humidity on the change of moisture content is shown graphically in Figure 1. It can be seen that, except for 93% relative humidity, equilibrium was attained within 8 days.

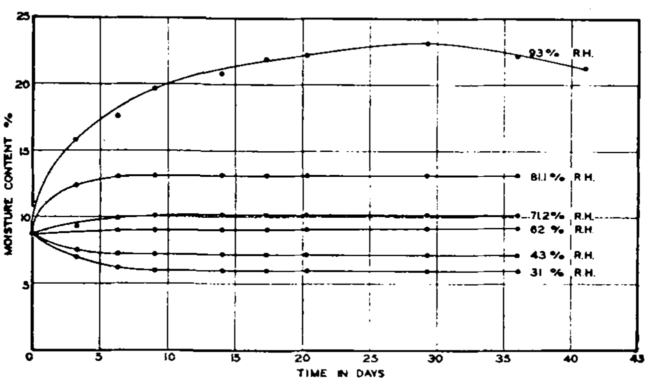

FIG. 1. Changes in moisture content of cottonseed exposed to various relative humidities for a period of 36 days.

The equilibrium moisture contents of intact cottonseed, meats, and hulls stored at various relative humidities are given in Table II and Figure 2.

When the moisture content of the meats is calculated on an oil-free basis it is observed that they attain the same equilibrium as hulls up to 70% relative humidity, but above that value the moisture content of the meats (oil-free basis) increases at a greater rate that does that of the hulls.

TABLE II

Equilibrium Moisture Contents of Whole Cottonseed, Meats, Hulls, and Oil-Free Meats at Various Relative Humidities

Relative humidity	Moisture content (wet basis)			
	Whole cottonseed	Meats	Hulls	Meats (oil-free)
%	%	%	%	%
31.0	6.03	5.13	7.67	8.32
43.0	7.23	5.92	9.60	9.33
62.0	9.25	7.73	11.85	12.11
71.2	10.27	8.89	12.62	13.72
81.1	13.21	11.73	15.31	18.29
93	22.19	21.40	22.35	32.80

Larmour *et al.* (8) investigated the hygroscopic equilibrium of soybeans, flaxseed, and sunflower seed as a function of relative humidity, and their data are compared in Table III and Figure 3 with those for cottonseed.

Since the oil is non-hygroscopic and does not change appreciably as the moisture content increases (except for free fatty acid formation) a more direct comparison of factors responsible for the hygroscopic behavior of seed can be made on an oil-free basis. The data of

[2] Hulls included linters.

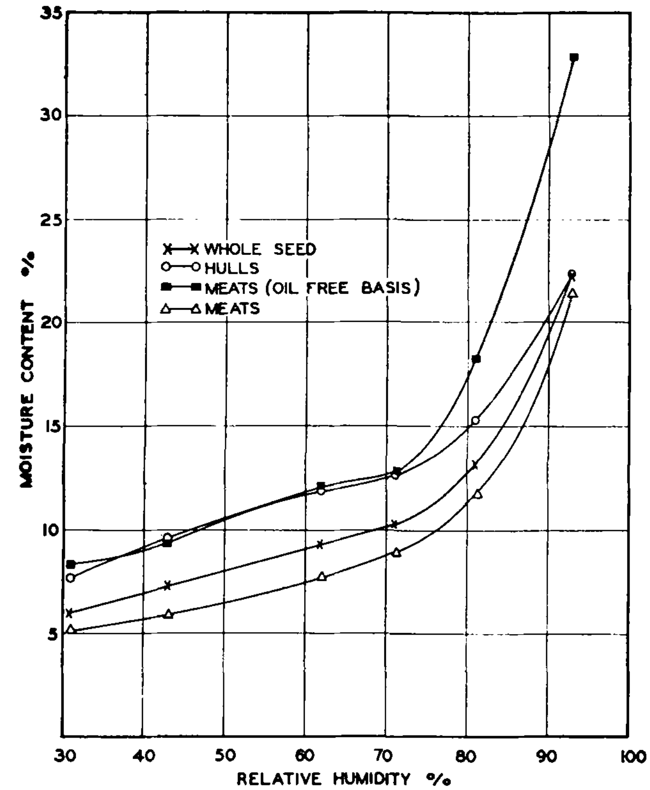

FIG. 2. Equilibrium moisture contents at different relative humidities of whole cottonseed, meats, hulls, and oil-free meats.

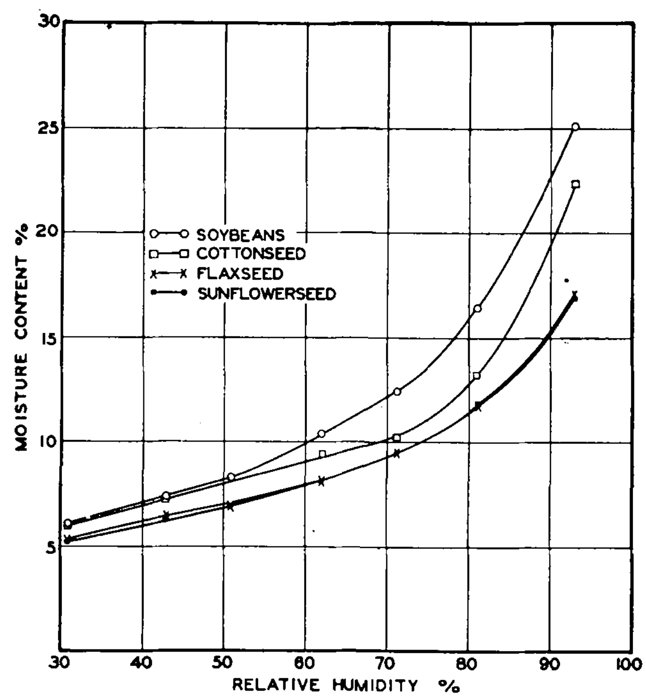

FIG. 3. Equilibrium moisture content at various relative humidities of soybeans, cottonseed, flaxseed, and sunflower seed. Data for soybeans, flaxseed, and sunflower seed from Larmour, Sallans, and Craig (8).

Table III, recalculated on an oil-free basis, are shown in Table IV and Figure 4. At low humidities the curve for the hygroscopic equilibrium of cottonseed (on an oil-free basis) is intermediate between the curves for flaxseed and sunflower seed. However, the moisture content of cottonseed increases more rapidly at humidities above 70% than does that of either flaxseed or sunflower seed. Furthermore, at 93% relative humidity cottonseed attains a higher equilibrium moisture content than do flaxseed and sunflower seed but one lower than that of soybeans.

TABLE III

Equilibrium Moisture Contents of Cottonseed, Sunflower Seed, Flaxseed, and Soybeans at Various Relative Humidities

Relative humidity	Moisture content (wet basis)			
	Cottonseed	Sunflower seed [a]	Flaxseed [a]	Soybeans [a]
%	%	%	%	%
31.0	6.0	5.2	5.3	6.1
43.0	7.2	6.3	6.4	7.4
51.0	......	6.9	6.9	8.3
62.0	9.3	8.1	8.2	10.4
71.2	10.3	9.5	9.5	12.4
81.1	13.2	11.7	11.6	16.4
93.0	22.2	16.9	17.1	25.1

[a] Data from Larmour, Sallans, and Craig (8).

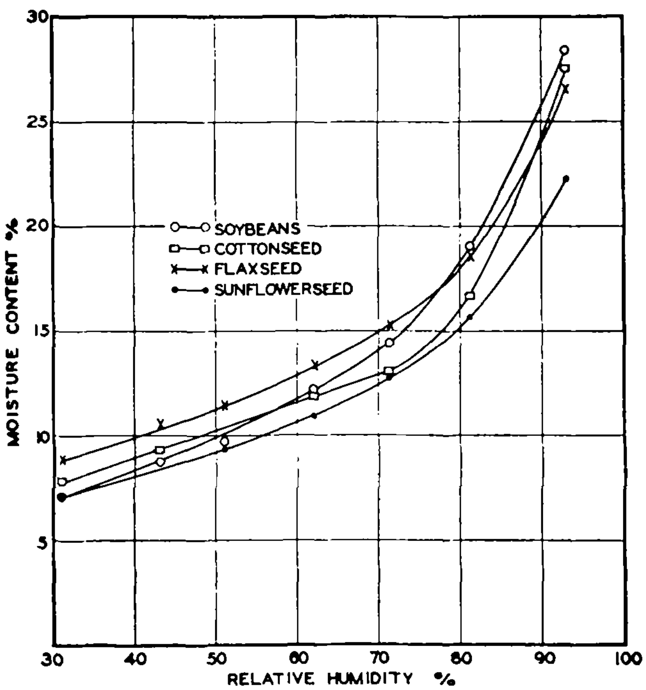

FIG. 4. Equilibrium moisture content at various relative humidities of soybeans, cottonseed, flaxseed, and sunflower seed on an oil-free basis. Data for soybeans, flaxseed, and sunflower seed from Larmour, Sallans, and Craig (8).

TABLE IV

Equilibrium Moisture Contents of Cottonseed, Sunflower Seed, Flaxseed and Soybeans at Various Relative Humidities Calculated on an Oil-Free Basis

Relative humidity	Moisture content (wet basis)			
	Cottonseed	Sunflower seed [a]	Flaxseed [a]	Soybeans [a]
%	%	%	%	%
31.0	7.8	7.1	8.9	7.1
43.0	9.3	8.6	10.6	8.7
51.0	......	9.3	11.4	9.7
62.0	12.0	10.9	13.3	12.2
71.2	13.1	12.7	15.2	14.4
81.1	16.7	15.6	18.6	19.0
93.0	27.5	22.0	26.3	28.5

[a] Data from Larmour, Sallans, and Craig (8).

Summary and Conclusion

An investigation has been made of the hygroscopic equilibrium of cottonseed over a range of 31% to 93% relative humidity. From 31% to 71% relative humidity the moisture content of cottonseed increased linearly from 6.03% to 10.27%. From 71% to 93% relative humidity the moisture content increased rapidly from 10.27% to 22.19%. When cottonseed was separated into meats and hulls, including linters, it was found that the hulls contained more moisture than the meats.

On the basis of these results it is apparent that, when stored cottonseed is aerated, consideration should be given to the effect of local atmospheric conditions. The relative humidity of the air used for aeration can affect the moisture content of the stored seed either favorably or adversely. Although it may temporarily reduce heating by conduction of the heat of respiration, it may increase the moisture content and thus stimulate further respiration and heating.

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Stability of Bound Gossypol to Digestion

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THE ether soluble, toxic gossypol of cottonseed is largely converted to the ether insoluble, "bound" gossypol in the cooking process. It is generally assumed to be bound to amino groups of the protein from analogy to the combination with aniline to form the very insoluble dianiline gossypol, which was found by Withers and Carruth (1) to be non-toxic to rats and rabbits. It colored the light colored feces of rats on a milk-starch diet a distinct orange color. Clark (2) found that "bound" gossypol in cottonseed meal could be converted to free gossypol after extraction with hot aniline.

Gallup (2) reported apparent digestibility coefficients of 61.2% to 71.5% for cottonseed meal protein compared to 73.6% to 81.2% for ether extracted raw cottonseed in rat diets. This suggests that the bound gossypol may have withheld some protein from digestion. Jones and Waterman (4) seeking to explain the relatively low digestibility of cottonseed meal protein, found that peptic and tryptic digestion of casein and

* The author is indebted to Dr. T. F. Zucker of Columbia University for the samples of feces and the "Proflo" flour and information that the feces weighed 28% of the diet.