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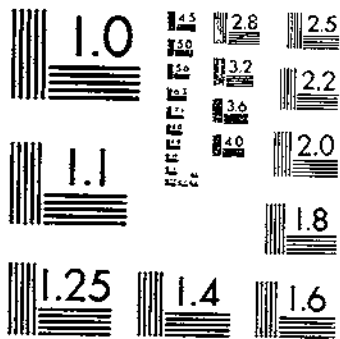
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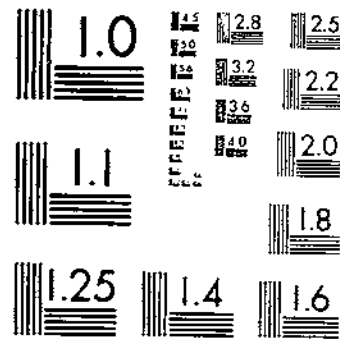
BRANDENBURG, N. R. HARMOND, J. E.

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MICROCOPY RESOLUTION TEST CHART  
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MICROCOPY RESOLUTION TEST CHART  
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**Equilibrium  
Moisture  
Content of  
FIBER FLAX**

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Technical Bulletin No. 1200

Agricultural Research Service  
UNITED STATES DEPARTMENT OF AGRICULTURE  
In cooperation with the Oregon Agricultural Experiment Station

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TB 100 (1957)

# Equilibrium Moisture Content of FIBER FLAX

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## SUMMARY

Since about 1938, the United States Department of Agriculture and the Oregon Agricultural Experiment Station have cooperated in an engineering research program conducted to improve equipment and methods used in processing fiber flax. Moisture content of flax straw and fiber has been found to be an important factor in efficiency of processing, but available literature concerning these products has contained little information on moisture.

The study reported in this publication was undertaken to determine the moisture content of flax straw and fiber when these products were conditioned in varying atmospheres. This research had two main objectives—supplying data needed by processors, and contributing basic information to a relatively unexplored field.

Equilibrium moisture content of retted flax straw was investigated initially in various relative humidities and at temperatures of 100° and 140° F. The investigations were later extended to include the flax substances described later in this bulletin and were conducted at 80° F. Materials were brought to moisture equilibrium in controlled atmospheres provided by a temperature-humidity cabinet with forced circulation. The substances were subjected to 9 levels of relative humidity ranging from 26 percent to 96 percent, and moisture content for each product was determined at each level.

When moisture content was plotted against relative humidity, S-shaped curves typical of hygroscopic materials were formed. All types of flax straw studied exhibited greater equilibrium moisture content (moisture regain) than did flax line fiber. Moisture regain curves for adsorption and desorption exposures enclosed distinct hysteresis areas for each substance. At 79 percent relative humidity, the regain for unretted flax straw was 14 percent by adsorption and 17 percent by desorption. Moisture regain values for three forms of flax tow fiber displayed an inverse relationship to the degree of processing, while forced-air drying appeared to affect the hygroscopic properties of retted flax straw.

## INTRODUCTION

Numerous substances have the ability to take up or release moisture under varying atmospheric conditions. These substances are referred to as hygroscopic. Wood, paper, leather, foodstuffs, and tex-

tiles are typical examples. Moisture retained by such materials at a specific atmospheric condition is called equilibrium moisture content because moisture is neither entering nor leaving the substance. A state of equilibrium then exists between the retained moisture and the water vapor in the surrounding air (10, p. 810).<sup>1</sup>

Other terms used to refer to equilibrium moisture content are moisture regain and hygroscopic moisture. Moisture regain is the most specific of the terms and it is commonly expressed as percent of water weight to the bone-dry weight of a material. This term, as used in this bulletin, refers to the regulated moisture content of the materials tested.

If not controlled, moisture may cause undesirable variations in weight, dimensions, strength, and other characteristics of hygroscopic materials during their processing, and later, during their use.

Air conditioning, as a means of regulating equilibrium moisture content, or regain, is considered essential in numerous manufacturing operations. Color printing, pharmaceuticals, candy manufacture, and textile processing are a few of the industries that employ controlled atmospheres to insure consistent production quality. In textile mills, for instance, it has been determined that the best condition for cotton spinning is a temperature of 60° to 80° F. with a relative humidity of 60 to 70 percent (7, p. 473). Different atmospheric conditions, however, may be optimum for other cotton operations, such as roving or weaving.

Hygroscopic moisture is important in other ways besides its influence on efficiency of processing and quality of the finished product. Numerous commodities are bought and sold on a weight basis, and their percentage of moisture content (regain) must be adjusted to make the transactions valid. Inasmuch as moisture content affects the physical properties of hygroscopic materials, tests of strength and of other properties should be conducted under a standardized specific moisture content of the substance.

Regain figures had previously been established for various materials, but this type of information for flax substances was extremely limited.

In an earlier study conducted in Australia in 1941, Greenhill and Pickering (5, p. 96) determined that in flax scutching (mechanical separation of fiber from stalk), fiber yields for dry straw increased about 50 percent when the moisture content of the straw was increased to the optimum.

In research conducted by the United States Department of Agriculture in 1954, Brandenburg<sup>2</sup> showed similar trends, with fiber yields increasing about 30 percent.

Another value of flax moisture regain, as pointed out by British investigators, is its use as an index of flax retting. Retting is peculiar to vegetable-fiber processing and consists of bacterial decomposition of pectins and other substances that tend to cement fiber to straw. A

<sup>1</sup> Italic numbers in parentheses refer to List of References, p. 14.

<sup>2</sup> BRANDENBURG, N. R. THE RELATION OF MOISTURE CONTENT AND FIBER YIELDS IN SCUTCHING. Agricultural Research Service, Agricultural Engineering Research Division Report, 1954. [Unpublished.]

British investigation reported by Samuels<sup>3</sup> indicates a close relationship between pectin content and moisture regain of flax straw.

Flax regain data are also important in drying, inasmuch as they indicate a limiting moisture content for specified conditions of air humidity and temperature.

### THE INVESTIGATIONS

In the initial investigation carried out by the United States Department of Agriculture, in 1948-49, in cooperation with the Oregon Agricultural Experiment Station, a study was made of moisture regain of retted flax straw in various relative humidities and at tem-

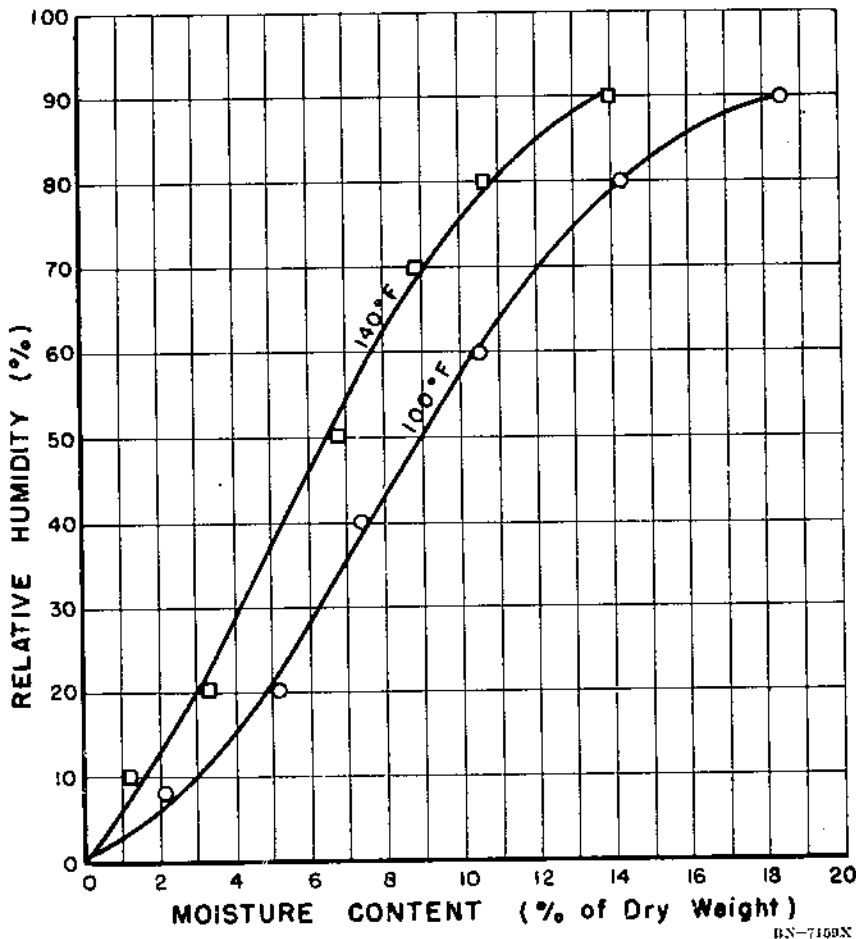


FIGURE 1.—Equilibrium moisture content (regain) of retted flax straw at varying relative humidities and temperatures.

<sup>3</sup> SAMUELS, D. E. J. A MATHEMATICAL TREATMENT OF BACTERIAL RETTING, AND THE SIGNIFICANCE OF THE MOISTURE REGAIN OF RETTED STRAW. Bd. of Trade, Dir. of Home Flax Prod., Flax Res. Com. B. O. T. S, FLX 550, 8 pp., illus. 1951. [Processed.]



peratures of 100° and 140° F.<sup>1</sup> The plotted results of this study, as shown in figure 1, illustrate S-shaped regain curves typical of hygroscopic materials.

The study reported in this publication was a continuation of the earlier research by the same two agencies. The study was extended to include the following flax substances: Unretted straw, field-dried retted straw, force-dried retted straw, line fiber, tow fiber, tow yarn, and linen tow fabric. Also tested were two common fiber materials—absorbent cotton and abaca (Manila hemp)—whose moisture characteristics are well known. All these substances were exposed to 9 levels of relative humidity at 80° F., and moisture content was determined at each level.

The mention in this publication of a commercial manufacturing establishment does not imply its endorsement by the United States Department of Agriculture over similar manufacturing establishments not named.

### Materials and Apparatus

Terms used in this publication in connection with the flax materials investigated are defined as follows:

*Retted straw.*—Straw that has been exposed to moisture and, as a result, has undergone progressive decomposition of pectin and other substances that tend to cement fiber to stalk.

*Field-dried, or air-dried, straw.*—Retted straw that has been air dried in the field after harvesting.

*Force-dried straw.*—Retted straw that has been dried in a forced-air drier rather than air dried in the field.

*Control straw.*—Retted straw of a given lot that has been field dried (air dried) for comparison with force-dried material of the same lot.

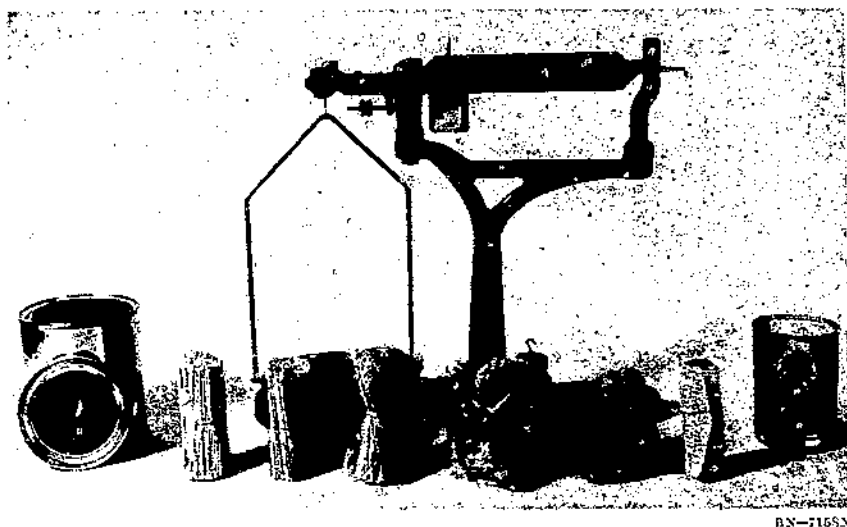
*Line fiber.*—Long, clean strands of fiber.

*Tow fiber.*—Short, tangled fibers with shives (small pieces of the stalk) adhering.

Samples of flax straw and fiber used in this study were composited from mill-run stocks of several processing plants in the Willamette Valley, Oreg. Unretted, force-dried, and control straws were long, medium-to-coarse flax of the Cascade variety, whereas retted straw was short and of fine-to-medium coarseness. Line and tow fiber samples were of average fineness and cleanness and were typical of commercial production. The flax tow yarn was spun from tow fiber and was 1-ply, 7-lea. The linen tow fabric was plain-weave, bleached, undyed material woven from this tow yarn. The cotton samples were prepared from sterilized surgical absorbent cotton. The abaca fiber (Manila hemp) was obtained by unravelling 3-strand regular-lay Manila rope.

All samples were approximately 2 inches in diameter and 3½ inches long. Throughout the study, they were contained in air-permeable

<sup>1</sup>Cooperative studies of United States Department of Agriculture and the Oregon Agricultural Experiment Station conducted in 1948 and 1949 by C. I. Branton, agricultural engineer. Mr. Branton is now located at the Agricultural Experiment Station, Palmer, Alaska.



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FIGURE 2.—Flax samples, from left to right: Unretted straw, retted straw, line fiber, tow fiber, tow yarn, and linen tow fabric. Also shown are airtight can, wire-mesh basket, and scales used to determine moisture content.

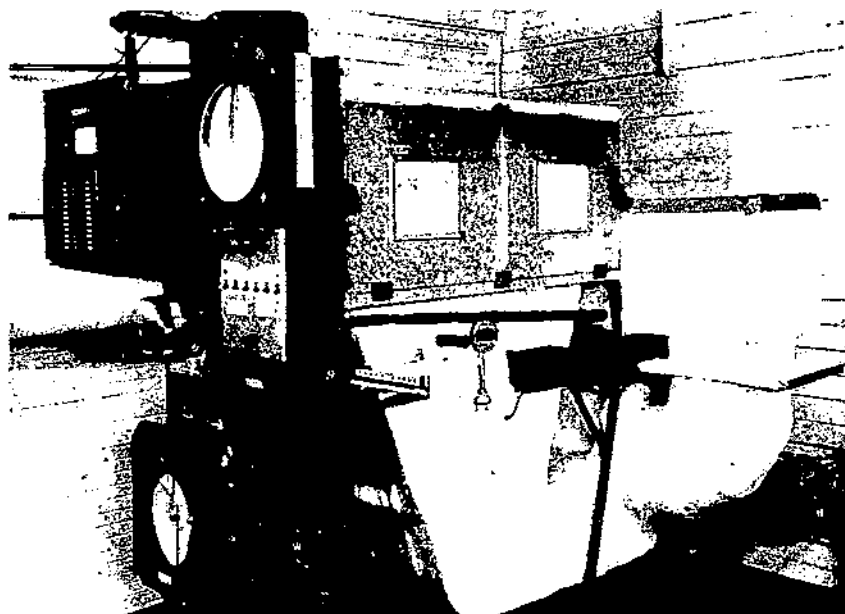
baskets of small-mesh screen. Flax samples, scales, and other equipment used are shown in figure 2.

Materials were conditioned to different moisture contents by exposure to controlled atmospheres in a temperature-humidity cabinet with air circulating through the samples. In this closed system, shown in figure 3, the source of heat consisted of thermally actuated electric-strip units. Moisture was supplied in the form of steam injected through a diaphragm valve that was operated pneumatically by a wet-bulb controller. For test runs of low relative humidity, conditioning air was forced through a 3-inch bed of silica gel inserted in the cabinet to adsorb moisture. To prevent air in the system from increasing in temperature beyond the desired point, room air surrounding the closed system was circulated through water-cooled coils.

Temperature readings were indicated by psychrometer, by dry-bulb wet-bulb recorder, and by potentiometer-pyrometer with copper-constantan thermocouples. An inclined draft gage was used to measure the airflow through the system. Moisture contents of samples were determined by using airtight cans, an electric oven, and a triple-beam balance.

### Test Procedures

A blending technique was used in preparing the flax samples, so that each was composed of parts of several commercial lots. Five replications were prepared in this way for each of the following classes of flax material: Unretted straw, field-dried straw, force-dried straw, force-dried control straw, line fiber, tow fiber, and tow yarn. Without blending, five replications were also prepared for linen (tow) fabric, abaca fiber (Manila hemp), and cotton fiber.



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FIGURE 3.—Temperature-humidity cabinet with air circulation to provide controlled conditioning atmosphere.

Average weights of the samples were: Flax straw (all classes), 20 grams; line fiber, 24 grams; tow fiber, 12 grams; abaca, 16 grams; cotton, 14 grams; tow yarn, 12 grams; and linen fabric, 6 grams.

Each sample was shaken vigorously and blown with air to remove loose material that might otherwise become detached during later handling. The sample was then placed in a wire basket where it remained throughout the test program.

During the conditioning procedure, all samples were brought to moisture equilibrium and weighed at progressively increasing and decreasing relative humidities. Dry weights were determined at the completion of the study. This technique was patterned after similar work by Wiegierink at the National Bureau of Standards (17, pp. 458-459).

All test exposures were run at a dry-bulb temperature of 80° F., but wet-bulb temperatures were varied to provide a different relative humidity for each exposure. Once the desired conditions were achieved in the closed conditioning system, the filled wire baskets were inserted in the cabinet in two layers. In each layer, the five baskets of any one material (line fiber, for instance) were interspersed among other baskets; each sample then occupied this same location in the cabinet in all the test runs. Dry- and wet-bulb temperatures were controlled and measured during the exposure period, and selected samples were removed, canned, and weighed periodically.

Conditioning was continued until repeated weights of the same sample were constant, at which time the baskets were quickly removed from the controlled atmosphere and sealed in airtight cans. Exposure

times varied from  $3\frac{1}{2}$  to  $8\frac{1}{2}$  hours; and after each exposure, sealed can weights were determined to the nearest 0.05 gram. Tests were run at exposures of 5 increasing relative humidities—46 percent, 57 percent, 70 percent, 81 percent, and 96 percent; and 4 decreasing relative humidities—76 percent, 53 percent, 32 percent, and 26 percent. After the 9 exposures, the samples were dried in an electric oven at  $212^{\circ}$  F. for 48 hours, then weighed to determine the bone-dry weight of each. The weight figures so obtained, together with can weight and sample weight recorded after each single conditioning exposure, were used to calculate moisture content, or regain, expressed on a percent-of-dry-weight basis.

### Test Results

Temperature control of the conditioned air is indicated in the following tabulation, which shows the complete range of dry-bulb and wet-bulb temperatures during heating-cooling cycles and steam-injection cycles of each of the nine exposures. All the dry-bulb temperature recordings were  $81^{\circ} \pm 2^{\circ}$  F.; and wet-bulb control appeared satisfactory except for low-humidity runs 1 and 9. In these two runs, with a silica-gel bed in the airstream, it was difficult to maintain low wet-bulb temperatures, probably because of a tendency for conditioned air to escape from the system and to be replaced by room air of greater relative humidity.

Conditioning exposure (No.)	Temperature range	
	Dry bulb	Wet bulb
1	$79 - 80\frac{1}{2}$	$57 - 62$
2	$79 - 81$	$65 - 66\frac{1}{2}$
3	$79 - 83$	$68 - 69\frac{1}{2}$
4	$80 - 82\frac{1}{2}$	$73 - 74\frac{1}{2}$
5	$79\frac{1}{2} - 82$	$76 - 78$
6	$79\frac{1}{2} - 82$	$78\frac{1}{2} - 80$
7	$79\frac{1}{2} - 82$	$73 - 76$
8	$80 - 82$	$68 - 69$
9	$79 - 82$	$56 - 60$

Conditioning data and moisture-content (regain) results from these investigations are presented in table 1. Most classes of material showed a relatively small range of moisture content within any 1 series of 5 replications. This range was about 1 percent for all flax substances except linen fabric, for which ranges of as much as 4 percent were observed. The greater variability in fabric moistures may have been related to bleaching, sizing, weaving, or other operations performed in the manufacturing of cloth; or to the fact that the fabric samples were relatively small.

### DISCUSSION

It is apparent from table 1 that 5 of the conditioning runs were from the adsorption and 4 from the desorption approach. In adsorption,

TABLE 1.—Conditioning data and moisture-content (regain) results from 9 test exposures of flax and other materials at a dry-bulb temperature of approximately 80° F. and at varying relative humidities

Conditioning exposure <sup>1</sup> (No.)	Conditioning data		Moisture content (regain) <sup>2</sup> of—									
	Relative humidity	Length of exposure	Unretted flax straw	Field-dried retted flax straw	Forced-dried retted flax straw	Field-dried retted flax straw (control)	Flax line fiber	Flax tow fiber	Flax tow yarn	Linen tow fabric	Absorbent cotton	Abaca (Manila hemp)
Adsorption: <sup>3</sup>	<i>Percent</i>	<i>Hours</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>
2.....	46	6½	7.7	8.0	7.3	7.7	6.7	8.3	7.0	6.7	4.8	9.5
3.....	57	6¼	8.8	9.0	8.4	9.0	8.0	9.6	8.6	7.7	5.9	10.4
4.....	70	8½	11.7	11.0	10.9	11.2	10.0	11.2	10.4	8.9	6.8	12.9
5.....	81	3½	15.0	13.8	13.2	14.0	11.6	13.8	12.7	10.4	8.3	15.7
6.....	96	6½	26.2	21.0	20.0	20.6	16.7	22.2	20.2	20.9	12.5	25.3
Desorption: <sup>4</sup>												
7.....	76	6½	16.1	15.2	14.8	15.4	12.7	13.6	12.4	10.9	8.3	16.7
8.....	53	6	10.4	10.9	10.4	10.8	9.2	10.2	9.0	8.1	6.1	11.9
1.....	32	6	6.6	7.0	6.1	6.8	5.7	7.4	6.2	6.1	4.4	8.6
9.....	26	5	5.9	5.8	5.6	5.9	5.0	6.1	5.1	4.4	3.3	6.8

<sup>1</sup> The conditioning exposures, or test runs, took place in the order of the numbers indicated. Conditioned air was circulated through the samples during exposures 2, 3, 4, 5, 6, 7, and 8 at 275 feet per minute, computed for the gross area of the conditioning chamber. Velocities were less in exposures 1 and 9 when silico gel was inserted in the system.

<sup>2</sup> Moisture content (regain) is expressed as percent-of-dry-weight, each figure being an average of 5 replications.

<sup>3</sup> In adsorption, the equilibrium moisture content is approached from a state of lesser moisture content.

<sup>4</sup> In desorption, the equilibrium moisture content is approached from a state of greater moisture content.

the approach is from a state of lesser moisture content; in desorption, from a state of greater moisture content. In both approaches, when moisture content (regain) is plotted against relative humidity, the adsorption-desorption curves obtained are typically sigmoid, or S-shaped. These curves at any selected constant temperatures are referred to as isotherms, and they usually fail to become superimposed on one another (8, p. 83). In textile research, the equilibrium region bounded by adsorption-desorption isotherms is called the hysteresis area. Strictly speaking, any point in this equilibrium region may be capable of representing the moisture regain of a sample.

The hysteresis phenomenon was investigated in this study, and all materials were found to exhibit such a characteristic to some degree. A well-defined hysteresis area, enclosed by adsorption-desorption curves, is shown for unretted flax straw in figure 4. As in all curves presented in this study, each point represents the average of five repli-

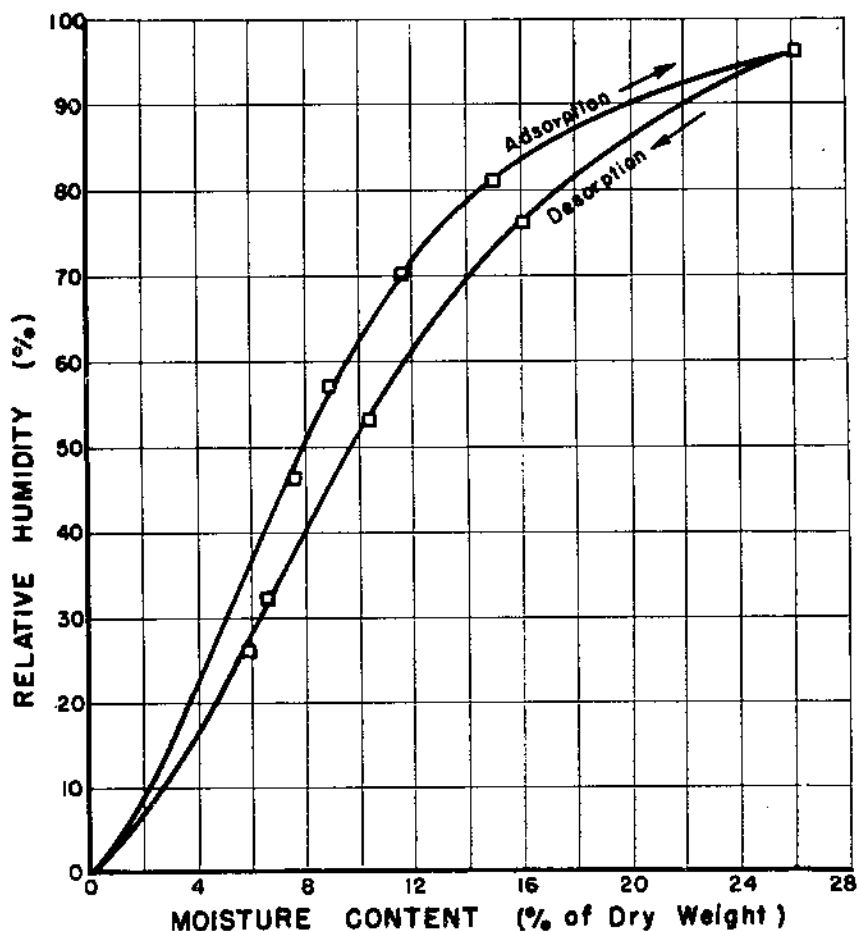


FIGURE 4.—Equilibrium moisture content (regain) of unretted flax straw at 80° F. and at varying relative humidities.

cations. The desorption curve is shown joining the adsorption curve at 96 percent relative humidity, whereas it would probably pass slightly below this point if higher humidities had been employed. The moisture regain values shown in this figure are similar in general to those determined by Zink for oat straw and alfalfa (17, p. 452).

Although the same typical hysteresis effect was evident for all materials tested in this study, as indicated by table 1, only desorption curves are presented in subsequent figures.

Equilibrium moisture content of three classes of flax straw are shown

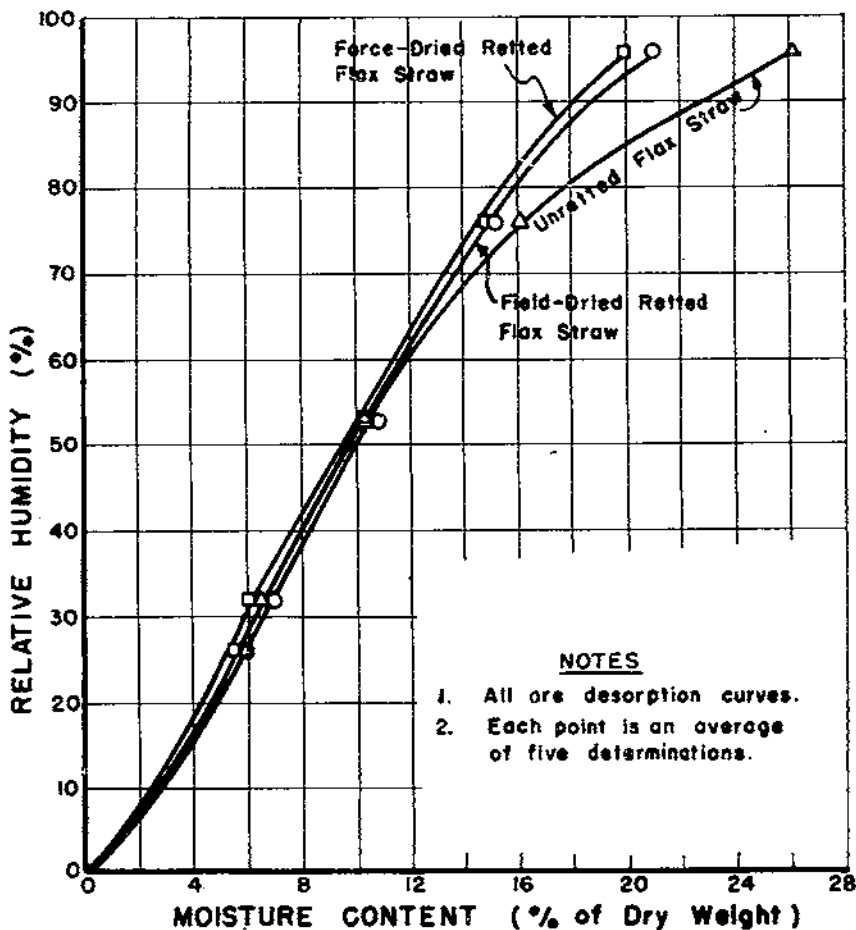


FIGURE 5.—Equilibrium moisture content (regain) of three classes of flax straw—unretted, field-dried retted, and force-dried retted—at 80° F. and at varying relative humidities.

in figure 5. Unretted, field-dried retted, and force-dried retted straw showed similar trends: but at higher relative humidities, moisture content of unretted straw was greater than that of retted straw. These findings are in general agreement with the British research mentioned

earlier (see footnote 3), which determined that moisture regain of flax decreased after retting. It seems probable that this decrease was caused by the removal of hygroscopic substances.

It is possible to make a direct comparison of regain values for force-dried retted straw and field-dried retted control samples of the same lot. If these were plotted, the regain values for the control samples would fall within the confines of the curves shown in figure 5. Although this suggests little difference between the regains for force-dried and control samples, it is interesting to note that at each exposure the control regain value would be the greater. This small but consistent difference indicates the possibility that forced-air drying affects the hygroscopic characteristics of retted flax straw.

In drying fruits and vegetables, it has been found that too rapid removal of water may cause a type of surface-cell distortion sometimes referred to as casehardening. This action could account for the lower regain values for force-dried straw. However, a later study of this straw, conducted in 1956 (2, p. 25), showed tensile strength and other spinning-quality factors of force-dried fiber to be equal or superior to those of field-dried fiber.

Flax fiber moisture-regain values that also revealed similarities in trend are shown in figure 6. Although the two classes of flax fiber were not from the same lot, they showed similar curves, with values for tow slightly greater than those for line. This relationship seems logical, inasmuch as tow fiber normally contains a considerable amount of residual straw particles, sometimes called shives; and regains for retted straw were consistently greater in this study than were those for line fiber.

In a Danish study (4, p. 53), these straw particles, or shives, were found to have a higher moisture regain than that of flax fiber. This was because they had a greater content of hygroscopic pectin.

The line and tow flax fiber regain values shown in figure 6 agree closely with the regains for line fiber reported in an Australian investigation (11, p. 9). In this comparison, tow values conformed more closely to the Australian data than did line values.

In all test runs, the moisture content of abaca (Manila hemp) was found to be greater than that of the other fibers shown in figure 6. A strong hygroscopic characteristic for abaca has been pointed out by Weindling (14, p. 41).

Cotton, by contrast, showed the lowest regain values recorded in this study. (See fig. 6.) The literature was searched to compare these values with the regains established for absorbent cotton in other experimental work. Data were found in several sources—International Critical Tables (9, p. 323); Heating, Ventilating, Air Conditioning Guide (1, p. 821); and an article by Hillen and coworkers in Chemical Engineers' Handbook (6, p. 777). The regain results set forth in these three citations were based on two earlier studies—one by Wilson and Fuwa in 1922 (10, p. 916) and another by Carrier Engineering Corporation Research Laboratories in 1929 (3, p. 180), both of which showed similar trends with considerably higher regains than those determined in the present study. At 90 percent relative humidity, for example, regains were about 25 percent in the earlier studies; at 96 percent relative humidity, regains were about 12.5 per-



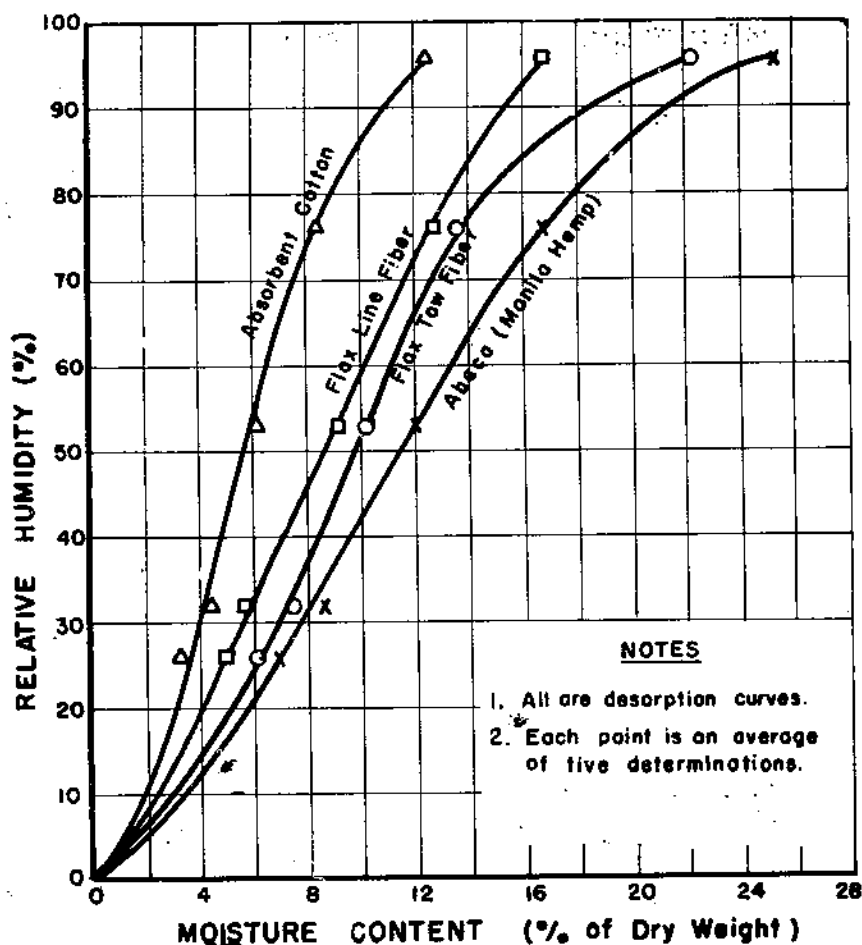


FIGURE 6.—Equilibrium moisture content (regain) of several fibers—absorbent cotton, flax line fiber, flax tow fiber, and abaca (Manila hemp)—at 80° F. and at varying relative humidities.

cent in the present study. The reason for such marked differences is not known.

An estimate of moisture regain for typical absorbent cotton now being produced was obtained from the research laboratory of a large manufacturer.<sup>5</sup> The regain figures determined commercially correspond closely with the cotton curve plotted in figure 6. At 90 percent relative humidity, the commercial regain is about 11.5 percent in contrast with 11 percent in the present study. In other comparisons, the cotton regain values shown in figure 6 are somewhat lower than those determined by Wiegierink at the National Bureau of Standards

<sup>5</sup> Personal communication from J. F. Ryan, Laboratory Director, The Ken'ell Company (Bauer and Black), Walpole, Mass., Jan. 16, 1958.

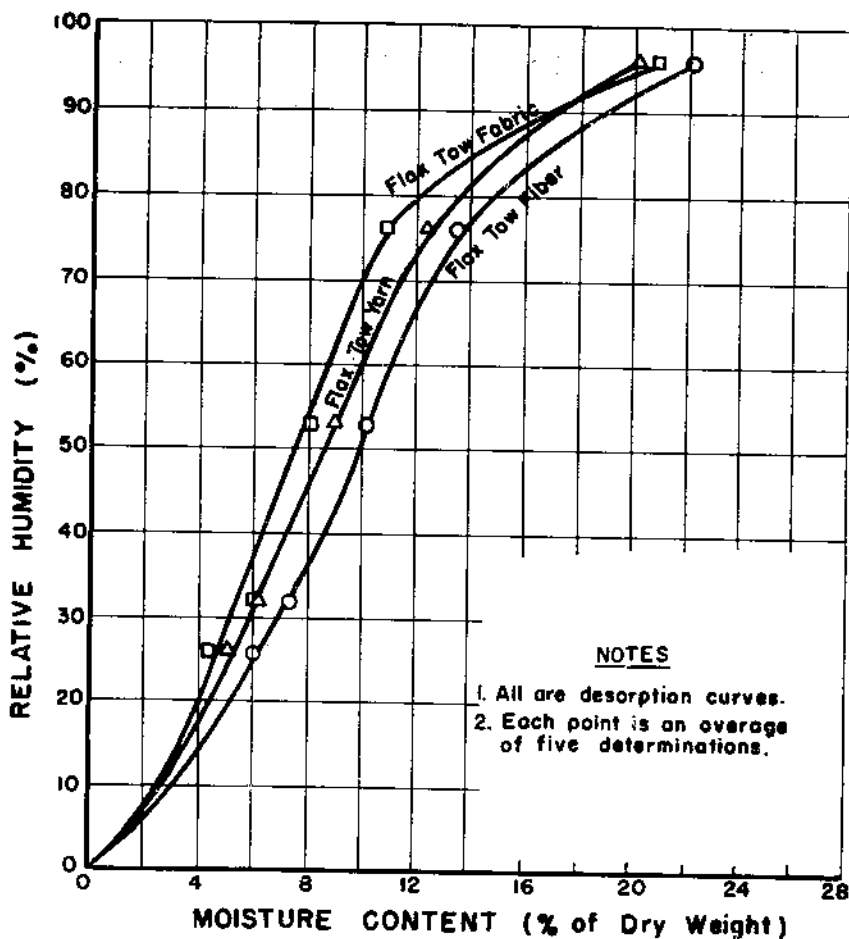


FIGURE 7.—Equilibrium moisture content (regain) of three classes of flax tow—fabric, yarn, and fiber—at 80° F. and at varying relative humidities.

for "purified" cotton (15, p. 360), but show a better correlation with results obtained by Urquhart and Williams for American cotton (13, p. 312).

The three classes of flax tow shown in figure 7 are presented together for comparison. The yarn and fabric were from the same lot, and the fiber was from similar stock. As might be expected, there is a pronounced likeness in the regain curves for the three materials. However, the moisture regain for tow appears inversely related to the degree of processing. Tow fiber, which represents the least processed state, shows greater regain in this graph than does tow yarn; the values for tow yarn, in turn, exceed those for tow fabric. It is possible that these trends are related to the presence of residual straw particles (shives) in most tow products, as explained in the discussion of figure 6. Shive content tends to decrease with processing, as the straw par-

ticles work free of the fiber; and the gradual removal of hygroscopic shives would effectively lower the moisture regain of the remaining material.

### CONCLUSIONS

Results of the investigations reported in this bulletin are summarized as follows:

1. All the substances tested exhibited definite hygroscopic characteristics and showed typical S-shaped curves when moisture regain (equilibrium moisture content) was plotted against relative humidity of a conditioning atmosphere.

2. A hysteresis phenomenon was demonstrated for each substance. This was seen in the fact that the moisture-regain curve obtained with increasing relative humidities (adsorption) showed less moisture than did the curve obtained with decreasing relative humidities (desorption). With unretted flax straw at 70 percent relative humidity, for example, the moisture regain was 14 percent by adsorption and 17 percent by desorption.

3. Unretted flax straw showed increasingly greater moisture regain than did retted flax straws at relative humidities above 60 percent in both adsorption and desorption exposures.

4. Evidence was found that forced-air drying affects the hygroscopic characteristics of retted flax straw. In both adsorption and desorption exposures, regain for force-dried material was slightly less than that for field-dried control samples of the same lot.

5. The three classes of flax straw—unretted, field-dried retted, and force-dried retted—showed greater moisture regain than did flax line fiber, in both adsorption and desorption exposures.

6. When two classes of flax fiber were compared, regain was found to be consistently greater for tow than for line. The difference was about 1 to 2 percent in both adsorption and desorption, except at high humidities where the regain for tow fiber was 5½ percent greater than for line fiber.

7. Moisture regain for three forms of flax tow seemed to be inversely related to the degree of processing. Tow fiber, representing the least processed state, showed greater regain than did tow yarn. The regain values for tow yarn, in turn, exceeded those for linen tow fabric.

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