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SORPTION OF WATER VAPOR BY PAPERMAKING MATERIALS

Part 4. Irreversible Loss of Hygroscopicity Due to Drying



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SORPTION OF WATER VAPOR BY PAPERMAKING MATERIALS

Part 4. -- Irreversible Loss of Hygroscopicity Due to Drying¹

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The investigations reported here comprise a part of fundamental studies at the Forest Products Laboratory of fiber and stuff properties. They deal with hygroscopicity as a fiber property, its relation to other fiber properties, its modification by chemical and physical processing, and its bearing on the phenomenon of hydration, as the term is used in papermaking.

A comprehensive review of the extensive literature of the hygroscopicity of papermaking materials, exclusive of the finished product, is beyond the scope of this paper. Such information relative to the more significant results of past work is readily available in a recent publication by Stamm (15).² It is desirable to call particular attention, however, to the researches of Mason and Richards, Urquhart and Williams, Campbell, Pidgeon, and Maass, Sheppard and Newsome, and Oguri, all of which have contributed greatly to this field of knowledge. It is of historical interest to observe that Müller (6) in 1882 was probably the first to observe the definite relation between the amount of moisture in the atmosphere and that sorbed by a cellulosic material.

Sorption Hysteresis

The hysteresis effect was observed as early as 1906 when Mason and Richards (5) found that a different equilibrium moisture content resulted at a specified relative humidity depending on whether equilibrium was approached from a higher or lower relative humidity. They attributed this result to failure to attain equilibrium rather than to an inherent characteristic of cellulose. Hysteresis is now, however, generally accepted as an inherent property of cellulose.

Researches dealing with hysteresis have proved valuable as a basis for theories of the mechanism of sorption. These studies should continue to be fruitful for it is by no means certain that the fundamentals

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²Numbers in parentheses refer to literature citations at the end of this discussion.

of the phenomenon are fully understood. The practical importance of hysteresis, as manifested by its effect on the physical properties of papers and boards has, comparatively recently, been given careful consideration in a number of investigations (3, 4, 7, 10, 16, 17). Recently at the Forest Products Laboratory a study was made of hysteresis (9) to determine its characteristics during the course of consecutively repeated sorption cycles, exclusive of the original desorption.

A highly beaten unbleached spruce sulphite pulp was subjected to seven complete cycles, each beginning with adsorption from dryness, and carried stepwise up to and including 95 percent relative humidity and ending with desorption to dryness. Hysteresis persisted, undiminished, throughout the seven cycles. This was aptly illustrated by the ratio of adsorption to corresponding desorption equilibrium moisture content values over the humidity range of 12 to 88 percent. The ratio ranged from 0.83 to 0.87, averaging 0.85.

When this calculation was applied to similar hysteresis loops available from unpublished data the ratio was found to fall within the rather narrow limits of 0.85 to 0.88 for a variety of chemical wood pulps and stuffs. In the case of sulphuric acid lignin and cuprammonium lignin the values were, respectively, 0.78 and 0.79. The hysteresis ratio, in the case of chemical wood pulps, thus appears approximately a constant, independent of species, amount of springwood or summerwood, pulping process, degree of beating, and degree of bleaching.

Urquhart (18) attributed sorption hysteresis of cellulose to the free secondary-valence bonds of the hydroxyl groups. When cellulose is wet the hydroxyl groups are practically all satisfied by water molecules and as drying proceeds these molecules are removed. As shrinkage occurs pairs of proximate hydroxyl groups are drawn together to satisfy the secondary-valence bonds. During adsorption not all these hydroxyl to hydroxyl linkages are again broken apart thus reducing the number of points of adsorption and consequently reducing the amount of moisture held.

Since the number and reactivity of the hydroxyl groups found in cellulose and lignin are very different, and since the degree and mechanics of shrinking and swelling are probably different for the two materials, it was thought that the magnitude of sorption hysteresis of cellulose and lignin would likely be different. It was found, however, that although the hysteresis ratios of the two materials showed some difference they were still of the same order of magnitude and the difference was much less than expected. Unless the observed difference between the hysteresis ratios of cellulose and lignin can be proved significant it is unlikely that hysteresis can be explained solely by Urquhart's theory of the coupling and uncoupling of the hydroxyl groups.

Effect of Physical and Chemical Processing

The continuity of the moisture sorption isotherms has been used (1, 12, 13) to establish the fact that beating (physical processing) does not effect the formation of a true cellulose hydrate. Although the results obtained in these investigations indicate a tendency of the beating effect to increase the hygroscopicity of pulps, the small and somewhat irregular differences observed apparently preclude a significant difference.

The study of the beating effect (11) was continued with the apparatus of improved sensitivity using highly beaten sulphate and sulphite pulps. The beaten pulps attained consistently higher equilibrium moisture contents than in the unbeaten state over the entire humidity range. A typical example is afforded by Figure 1. The average increase in hygroscopicity was 6 percent on the basis of the moisture content of the unbeaten pulp, which is probably near the maximum attainable by beating for the specified humidity range. Since, however, the hygroscopicity increase was greater at the higher relative humidities, it was deemed advisable to extend such determinations to a higher humidity.

Bleaching was found to reduce hygroscopicity, the reduction increasing with increasing severity of bleaching. In the case of a sulphite pulp a practically linear reduction in hygroscopicity resulted over a range of 8 to 25 percent bleach ratio where the upper limit represented definite overbleaching. Representative adsorption isotherms for wood and the lignin and sulphite pulp isolated from it are illustrated in Figure 2.

Irreversible Reduction of Original Hygroscopicity by Drying

The effect of drying on the properties of cellulosic materials is of both fundamental and practical importance. This can be readily appreciated by reference to relevant literature, a survey of which is available (14). The adsorption hysteresis following original drying has been discussed previously as illustrative of fundamental value. In the case of cotton, Urquhart and Eckersall (19) found this reduction in hygroscopicity to persist after soaking the material in hot water. An essentially practical example is the effect on the strength of a pulp. Some have found that air drying caused an irreversible reduction in the strength whereas others, notably Harrison (2), found the contrary. There are, however, practically no data available relevant to the effects of drying on the moisture relations of pulps. For this reason the following study of these effects was made at the Forest Products Laboratory.

Apparatus Used for the Determination of Hygroscopicity

The apparatus used for determining the moisture contents at the various humidities is shown in a previous paper in this series (11). It consists essentially of a thermostatic air bath in which the samples of pulp are suspended from sensitive calibrated quartz spirals in sorption chambers through which air, humidified by passing through different saturated salt solutions to give the desired relative humidity, is circulated. The change in length of the quartz spirals as the samples gained or lost moisture, depending on whether the humidity was increased or decreased, was measured by means of a cathetometer mounted outside of the air bath. In this way the moisture content of the samples, calculated from the cathetometer readings and the calibration data of the quartz spirals, was determined without disturbing the temperature and humidity conditions of the sorption chambers.

Prehumidifying towers delivered air with a relative humidity from 5 to 10 percent above that desired for the sorption experiments. This prehumidified air was then brought to the desired humidity by passage through one of the several saturated salt solutions within the thermostatic air bath.

The saturated salt solutions used and the respective relative humidities obtained thereby are listed in Table 1. The salts marked with superscript a give the most constant relative humidities and are the only ones now used. All stopcocks within the bath affecting the flow of conditioned air were operated by extensions outside the bath.

Table 1.--Equilibrium relative humidities obtained by 12 saturated salt solutions

Salt solution	Relative humidity : 25 degrees C., : percent	Salt solution	Relative humidity : 25 degrees C., : percent
LiCl . H ₂ O ^a	12.0	:NaNO ₂ ^a	63.5
MgCl ₂ . 6H ₂ O ^a	33.0	:NaCl ^a	74.0
Zn(NO ₃) ₂ . 6H ₂ O.....	40.0	:KCl ^a	85.0
Na ₂ Cr ₂ O ₇ . 2H ₂ O.....	53.4	:K ₂ CrO ₄	87.0
MnCl ₂ ^a	55.0	:KNO ₃ ^a	93.0
NaHSO ₄ . H ₂ O.....	56.5	:K ₂ HPO ₄	95.3

^aGave most uniform relative humidities.

The samples were dried by air from a series of towers containing, in the order named, anhydrous calcium chloride, concentrated sulphuric acid, and phosphorus pentoxide suspended on glass wool.

The air flow was distributed evenly throughout eight sorption tubes by controlling the pressure through side-arm outlets projecting from the base of the tubes through the air-bath wall and into a trough of water at constant level. The depth of immersion of each side-arm tube was adjusted to obtain a rate of flow of approximately three bubbles per second from a 4-mm. bore tube.

The quartz spirals, to which the pulp and stuff samples were attached in sheet form, gave, on an average, a deflection of about 30 cm. with 100-mg. load.

The sorption samples ranged in weight from 30 to 75 mg., the lighter samples being used with the more sensitive spirals. All samples came to equilibrium in 24 hours except in relative humidities near 90 percent in which instances the time required was frequently 48 hours. When drying the samples a 48-hour period was used to insure equilibrium, although the sample weight at the end of 24 hours usually was the same as that after 48 hours of drying.

The apparatus is capable of being operated with high precision. In a typical sorption experiment, the average arithmetical difference in percentage moisture content among 93 pairs of check determinations was 0.10. At very high humidities the check determinations did not agree as well, so if the 10 pairs of check determinations at 93 percent relative humidity were omitted from the calculations, the average spread among the remaining 83 pairs was reduced to 0.07.

Wet samples, too heavy to be carried by the spirals, must be supported otherwise until after partial drying. The device used, a modification of Russell's (8) ring support, is illustrated in Figure 3. It consists of a hook at the end of a glass rod which passes through and is supported by a rubber stopper at the top of the sorption chamber. The part of the rod projecting above the stopper is bent to form a handle for turning the hook out of the way from outside of the bath after the spiral has assumed the load of the drying sample. Considerable vertical adjustment is possible to accommodate the length and stretch of the spiral and the hook can also be turned completely out of the way after the spiral has assumed the load.

With highly sensitive quartz spirals a correction had to be made for the moisture adsorbed by the spirals themselves above 65 percent relative humidity. No change in length of the spirals was observed below 55 percent relative humidity. At 65 percent relative humidity a deflection was observed, but was within the error of the cathetometer readings. Above 65 percent relative humidity the deflection increased with increasing humidity until it became appreciable at the higher

humidities. The spirals were calibrated at 65 percent relative humidity and the correction applied was for the weight of moisture adsorbed in excess of that adsorbed at 65 percent relative humidity. The corrections range from an average of 0.07 to 0.16 mg. for relative humidities of 74 to 93 percent, respectively. These amounts are, in general, greater than can be accounted for by a monomolecular adsorption on the geometric surface of the spirals. This may indicate surface irregularities bordering on porosity.

Materials and Procedure

The material selected for determination of the effect of drying on hygroscopicity comprised both unbleached and bleached unbeaten western hemlock sulphite pulps. These materials were maintained above the fiber-saturation point at all times from the digestion of the wood to the determination of the original desorption isotherms under the conditions of the sorption apparatus. After the samples had reached dryness through the course of original desorption and the weight of dry material determined, they were removed from the sorption apparatus and soaked in water at room temperature for 24 hours. A modification of this treatment was made in the case of the fifth isotherm by boiling the samples in water for 4 hours and then soaking them in water at room temperature for 20 hours prior to the determination of the isotherm. After each desorption, the samples were carefully placed between alternate layers of lintless blotters and wrapped with rubber bands to keep them firmly in place during soaking. At intervals during soaking the submerged samples were subjected alternately to a vacuum and atmospheric pressure to remove entrapped air. After such treatment the samples were touched gently with blotters to remove enough excess water so that they could be replaced on the spirals without danger of falling apart. The next desorption isotherm was then determined. The moisture content values of each isotherm were calculated on the basis of the weight of dry material found at the end of each run, thus avoiding error due to loss of fibers in the soaking treatment. The alternate soaking and drying procedure was continued until nine desorption isotherms were obtained. After the ninth desorption a closed hysteresis loop, between dryness and 93 percent relative humidity, was determined for each pulp.

Discussion of Results

The desorption isotherms in Figures 4 and 5, plotted from the data in Table 2, illustrate that the drying of pulps under the conditions of the experiment resulted in a reduction of their original hygroscopicity over the range of 93 to approximately 50 percent relative humidities. Below this range the isotherms were coincidental, differences, if any, being too small to measure.

Table 2.--Effect of alternate drying and soaking on the desorption equilibrium moisture contents of unbeaten western hemlock sulphite pulp

Relative: humidity: at 25 : degrees: C.	Moisture contents: Averages of four determinations (Percent on dry basis)									
	Desorption isotherms									
	:Original:	Second:	Third :	Fourth:	Fifth :	Sixth :	Seventh:	Eighth:	Ninth	
	<u>Unbleached</u>									
93	: 27.69	: 23.51	: 21.61	: 20.73	: 20.77	: 19.66	:	: 19.67	: 19.66	
85	: 19.84	: 18.43	: 17.78	: 17.01	: 16.84	: 16.50	: 16.49	: 16.55	:	
74	: 14.89	: 14.33	: 14.13	: 13.67	: 13.60	: 13.20	: 13.43	: 13.25	: 13.23	
63.5	: 11.43	: 11.31	: 11.07	: 10.87	: 10.61	: 10.65	: 10.71	: 10.71	: 10.71	
55	: 9.54	: 9.57	: 9.40	: 9.19	: 9.10	: 9.19	: 9.10	: 9.19	: 9.18	
33	: 6.03	: 6.05	: 6.10	: 5.97	: 5.82	: 5.95	: 5.95	: 6.03	: 5.89	
12	: 3.27	: 3.21	: 3.25	: 3.13	: 3.04	: 3.20	: 3.19	: 3.17	: 3.14	
	<u>Bleached</u>									
93	: 26.24	: 22.15	: 20.44	: 19.52	: 19.62	: 18.73	:	: 18.75	: 18.65	
85	: 19.47	: 17.85	: 17.14	: 16.28	: 16.10	: 16.40	: 16.12	: 15.98	:	
74	: 14.55	: 14.10	: 13.19	: 13.33	: 13.26	: 13.01	: 13.29	: 12.92	: 13.01	
63.5	: 11.20	: 11.22	: 11.04	: 10.69	: 10.32	: 10.61	: 10.57	: 10.46	: 10.55	
55	: 9.44	: 9.46	: 9.34	: 9.13	: 8.92	: 9.09	: 9.05	: 8.93	: 9.07	
33	: 6.06	: 6.04	: 6.09	: 5.93	: 5.77	: 5.97	: 5.94	: 5.81	: 5.89	
12	: 3.33	: 3.26	: 3.25	: 3.14	: 3.02	: 3.25	: 3.20	: 3.13	: 3.18	

The reduction continued in decreasing increments with each successive drying until the pulps had been dried six times. The reduction was not reversible by the intermediate soaking or boiling in water as applied. It is true, however, that the treatment in boiling water applied after the fourth desorption did prevent a reasonably expected reduction in the fifth as compared with the fourth. This means, of course, a partial restoration of hygroscopicity was effected.

One interpretation of the results is afforded by comparing the range of percentage reduction in the hygroscopicity of the unbleached and bleached pulps. These percentage values are given in Table 3.

The reduction in hygroscopicity due to these successive dryings appears to be independent of the bleaching effect since the percentage reduction was the same after bleaching as before. It follows from the absence of lignin in the bleached pulp that the hygroscopicity reduction in the case of the unbleached pulp did not involve mechanical structural relations between

either cellulose or hemicelluloses and the residual cell wall lignin. Likewise, extraneous materials other than lignin, but removable by bleaching, were obviously not involved. Since cotton, very low in pentosan content, exhibits a similar initial reduction it seems unlikely that these components are an important factor. In other words, the phenomenon appears to be related strictly to the cellulose.

Table 3.--Range of percentage reduction in hygroscopicity

Condition of pulp	Percentage reduction ¹ between isotherm numbers			
	1 and 2		1 and 7 or 9	
	63.5 percent relative humidity	93.0 percent relative humidity	63.5 percent relative humidity	93.0 percent relative humidity
Unbleached.....	1	15	6	29
Bleached ^{2, 3}	0	16	6	29

¹Values on basis of original desorption moisture content.

²Bleached in a single stage with 5.3 percent chlorine as calcium hypochlorite to a white of about 86 percent.

³The original hygroscopicity of the bleached pulp was 5 percent less than the unbleached at 93 percent relative humidity. This difference remained the same after the ninth desorption.

Other factors that must be considered when interpreting the results are: first, the adsorption isotherms for the unbleached and bleached pulps determined after the ninth desorption; and, second, the adsorption isotherm determined as a part of a closed hysteresis loop, immediately following a first desorption isotherm of the material. As was mentioned in the details of procedure, adsorption isotherms, as parts of closed hysteresis loops, between dryness and 93 percent relative humidity, for both the unbleached and bleached pulps were determined immediately after the ninth desorption. The equilibrium moisture values for these adsorption isotherms are recorded in Table 4. They are expressed graphically in Figures 4 (unbleached pulp) and 5 (bleached pulp). An original closed hysteresis loop (adsorption to 93 percent relative humidity, then desorption, after the original desorption from above fiber saturation) was obtained for the unbleached pulp only. The equilibrium moisture content values for these closed hysteresis loops and the corresponding hysteresis ratios are shown in Table 5. In the case of the unbleached pulp the two adsorption isotherms were coincidental, so that the adsorption isotherm

Table 4.--Sorption hysteresis between the average final desorption from above fiber saturation and subsequent adsorption of an unbleached and bleached western hemlock sulphite pulp

Relative humidity 25 degrees C.	Moisture content (percent on dry basis)			Hysteresis ratio ¹
	Average of sixth, seventh, eighth, and ninth desorptions from above fiber saturation			
	Subsequent adsorption			
	:			
Unbleached	:	:	:	:
93	19.66	16.64	:	0.85
85	16.51	13.62	:	.83
74	13.28	11.20	:	.85
63.5	10.70	9.15	:	.86
55	9.17	8.01	:	.87
33	5.95	5.34	:	.90
12	3.17	2.99	:	.94
Average.....				0.87
Bleached	:	:	:	:
93	18.70	15.72	:	.84
85	16.17	12.93	:	.80
74	13.06	10.64	:	.89
63.5	10.55	8.84	:	.84
55	9.04	7.72	:	.86
33	5.90	5.20	:	.88
12	3.19	2.88	:	.90
Average.....				0.86

¹ Adsorption moisture content divided by desorption moisture content.

Table 5.--Sorption hysteresis values for closed hysteresis loops of unbleached and bleached western hemlock sulphite pulps

		Unbleached				Bleached			
		Moisture content after original ¹ desorption:		Hysteresis ratio ²		Moisture content after original ¹ desorption:		Hysteresis ratio ²	
		from above fiber saturation:	ratio ²	from above fiber saturation:	ratio ²	from above fiber saturation:	ratio ²	from above fiber saturation:	ratio ²
		Percent		Percent		Percent		Percent	
<u>Adsorption from dryness</u>									
12	:	2.86	:	0.87	:	2.99	:	0.94	:
33	:	5.41	:	.89	:	5.34	:	.92	:
55	:	8.04	:	.88	:	8.01	:	.90	:
63.5	:	9.15	:	.86	:	9.15	:	.89	:
74	:	11.35	:	.89	:	11.20	:	.92	:
85	:	13.33	:	.93	:	13.62	:	.94	:
93	:	16.40	:	:	16.64	:	:
Average hysteresis ratio		.89				.92		.90	
<u>Desorption from 93 percent relative humidity</u>									
85	:	14.32	:		:	14.48	:	13.95	:
74	:	12.76	:		:	12.20	:	11.83	:
63.5	:	10.60	:		:	10.33	:	10.03	:
55	:	9.15	:		:	8.89	:	8.75	:
33	:	6.10	:		:	5.81	:	5.76	:
12	:	3.31	:		:	3.17	:	3.15	:

¹Separate sample run independently of rest of samples.

²Adsorption moisture content divided by desorption moisture content.

in Figure 4 represents both the original and final adsorptions. The desorption isotherms of the closed hysteresis loops are not shown in Figures 4 and 5.

The original and final adsorption isotherms began from a dry condition which had previously been reached after contact with liquid water hence they are on a common basis. For this reason and also because they are coincidental, the adsorption moisture relations of this pulp appear to be independent of the effects produced by the alternate drying and soaking treatments on the desorption moisture relations. If, in this instance, adsorption were not independent of the observed reduction in desorption moisture content, it seems reasonable to expect the occurrence of a more or less corresponding reduction in the adsorption moisture contents determined immediately following the ninth desorption.

The fact that the desorption reduction extended only to 40 percent relative humidity (as a likely lower limit) in conjunction with its apparent lack of influence upon the adsorption isotherm cannot be explained on the basis of current theories of sorption mechanism.

The average hysteresis ratios of the unbleached and bleached pulps in Table 4 were calculated from the open hysteresis loop formed by the average moisture content values of the sixth, seventh, eighth, and ninth desorption isotherms from above fiber saturation and the subsequent adsorption isotherms. These average ratios are somewhat lower than the average ratios in Table 5 calculated from the closed hysteresis loops between dryness and 93 percent relative humidity. As the limits of the extremes of humidity of a closed hysteresis loop are extended, the magnitude of hysteresis increases, and, therefore, the average ratio of adsorption to desorption decreases. These facts indicate that the open hysteresis loop (formed by the ninth desorption isotherm) is probably coincidental over the range of 0 to 93 percent relative humidity, with the corresponding isotherms of a hypothetical closed hysteresis loop between dryness and some relative humidity higher than 93 percent, probably 100 percent. The dotted line in Figures 4 and 5 are extrapolations and represent possible points of juncture of the adsorption and desorption isotherms at 100 percent relative humidity. The moisture contents at which these isotherms are shown to close, are purely arbitrary and in no sense quantitative, since they were obtained by extrapolating curves of rapidly changing slope. The greater rate of change of slope of the adsorption isotherm is further indication of the possibility of juncture.

The drying and soaking treatments did not affect the desorption moisture content below 50 percent relative humidity. It is therefore unlikely that moisture contents of these pulps obtained from further desorptions from above fiber saturation would fall below this isotherm. The fact that there was but a negligible difference between the moisture content values of the sixth and ninth desorption isotherms is experimental evidence that the theoretical stable minimum was attained.

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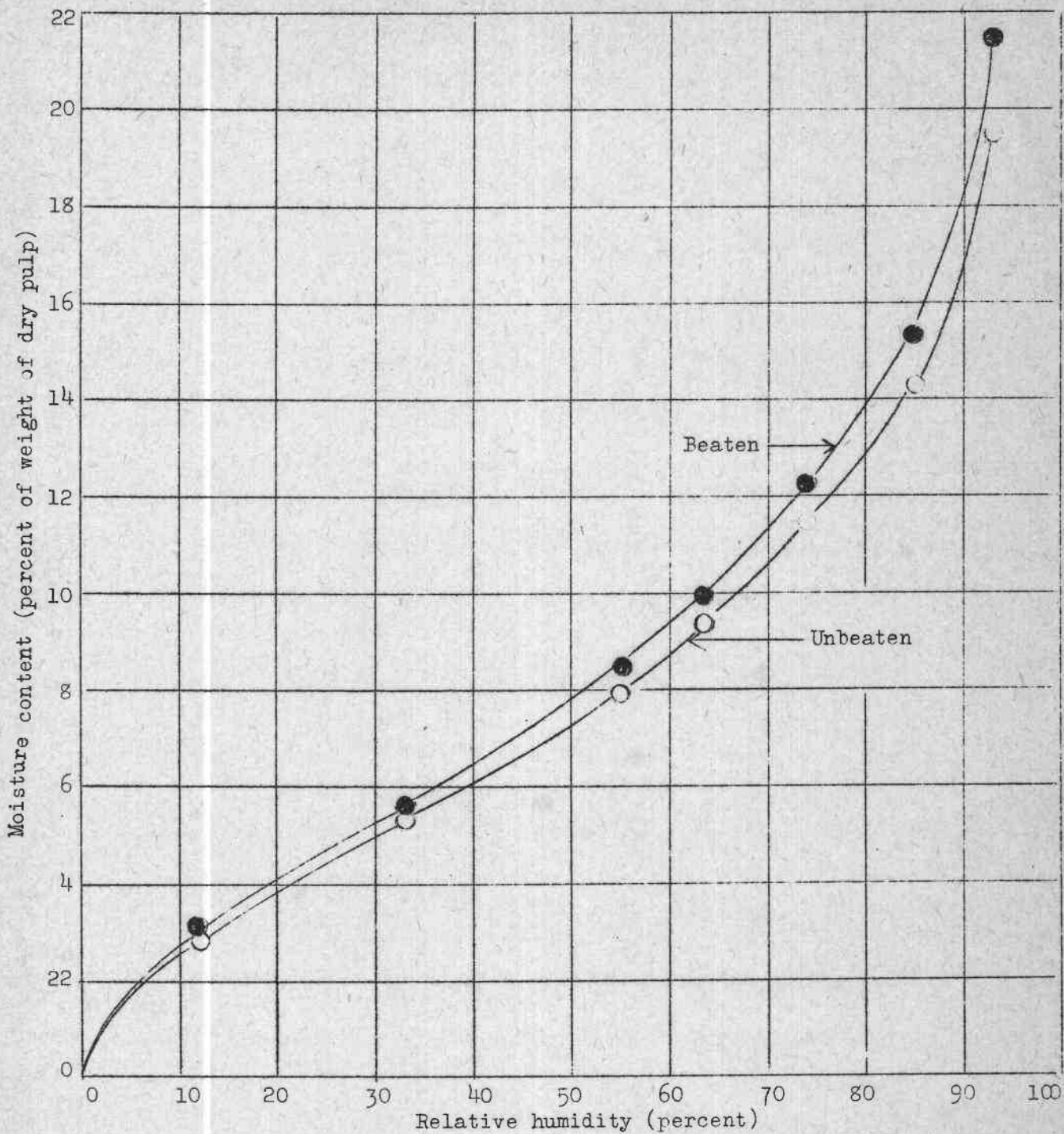


Figure 1.--Effect of extreme beating on the adsorption equilibrium moisture content of a silver fir sulphite pulp.

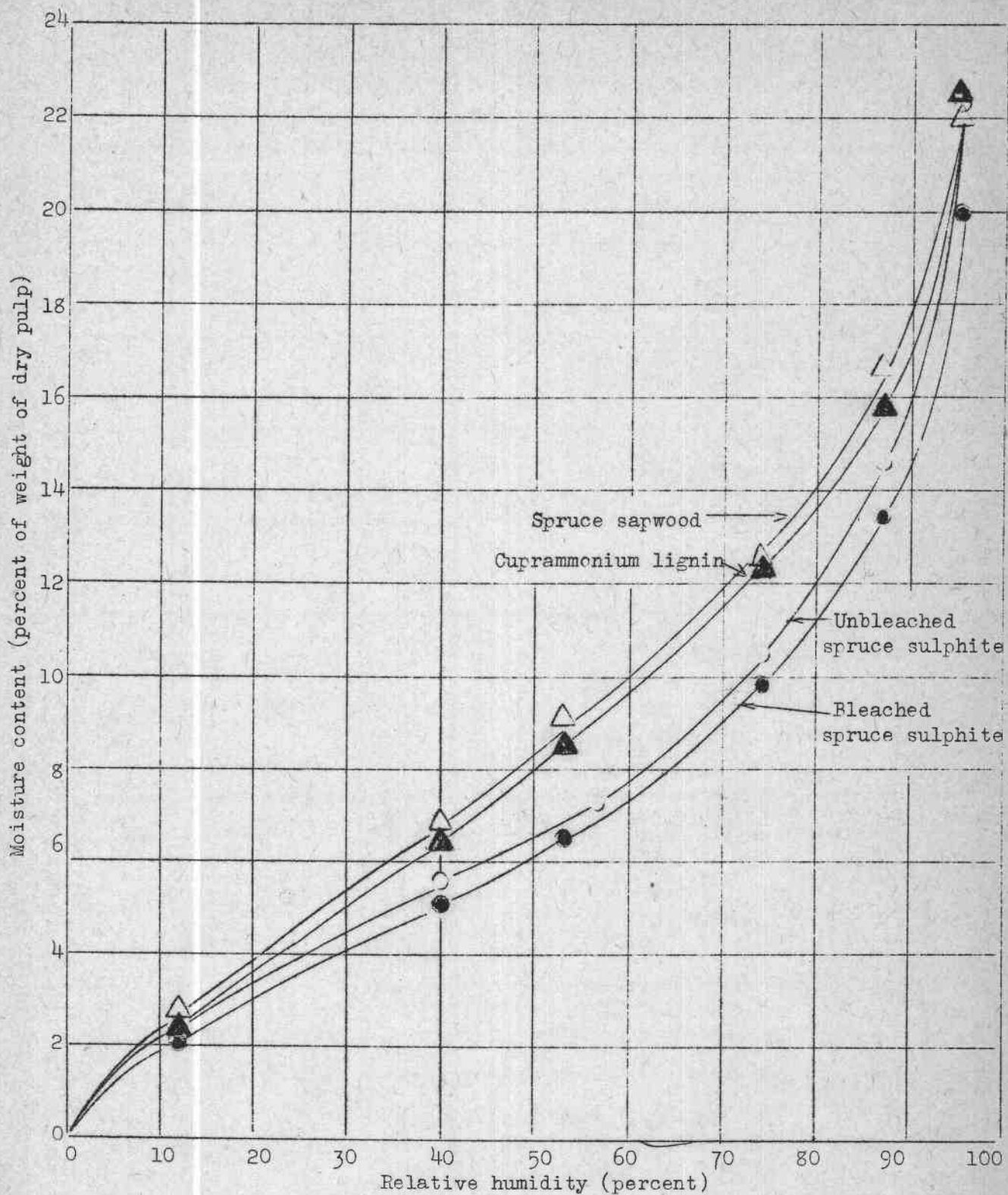
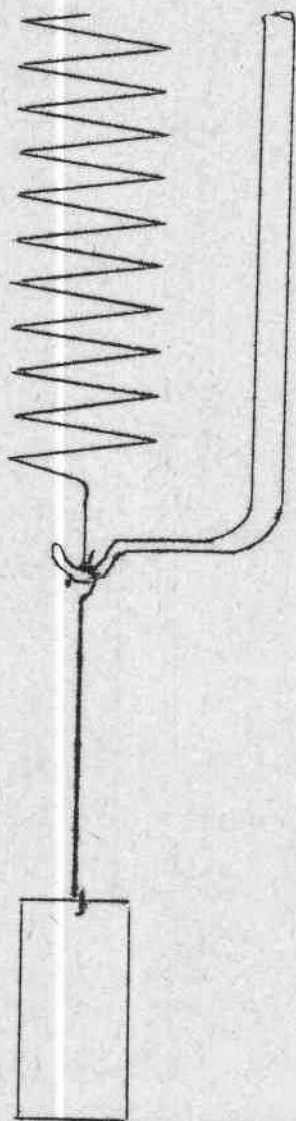
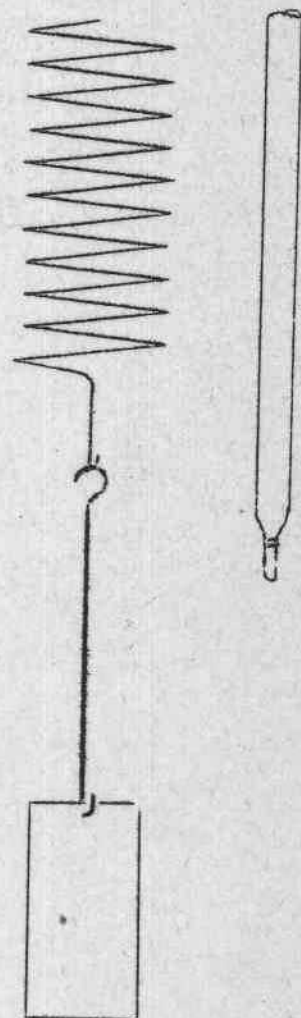


Figure 2.—Adsorption isotherms for spruce sapwood and the lignin and sulphite pulp isolated from it.



Glass hook supports
Excess weight of sample.



Spiral supports partially dried sample.
Glass hook turned out of way at 90
degrees,

Figure 3.--Hook support for wet sample.

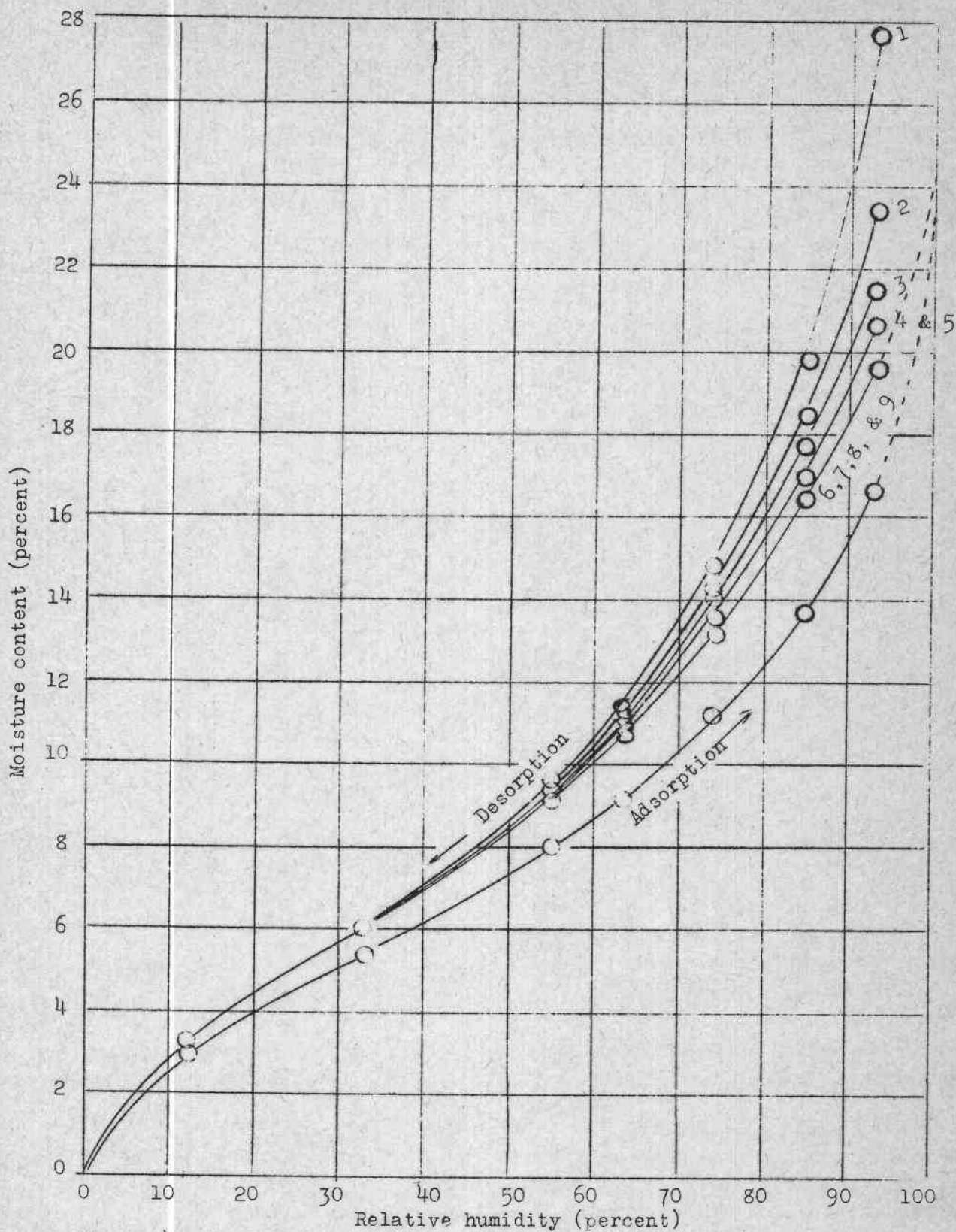


Figure 4.--Desorption isotherms from above fiber saturation of unbleached unbeaten western hemlock sulphite pulp.

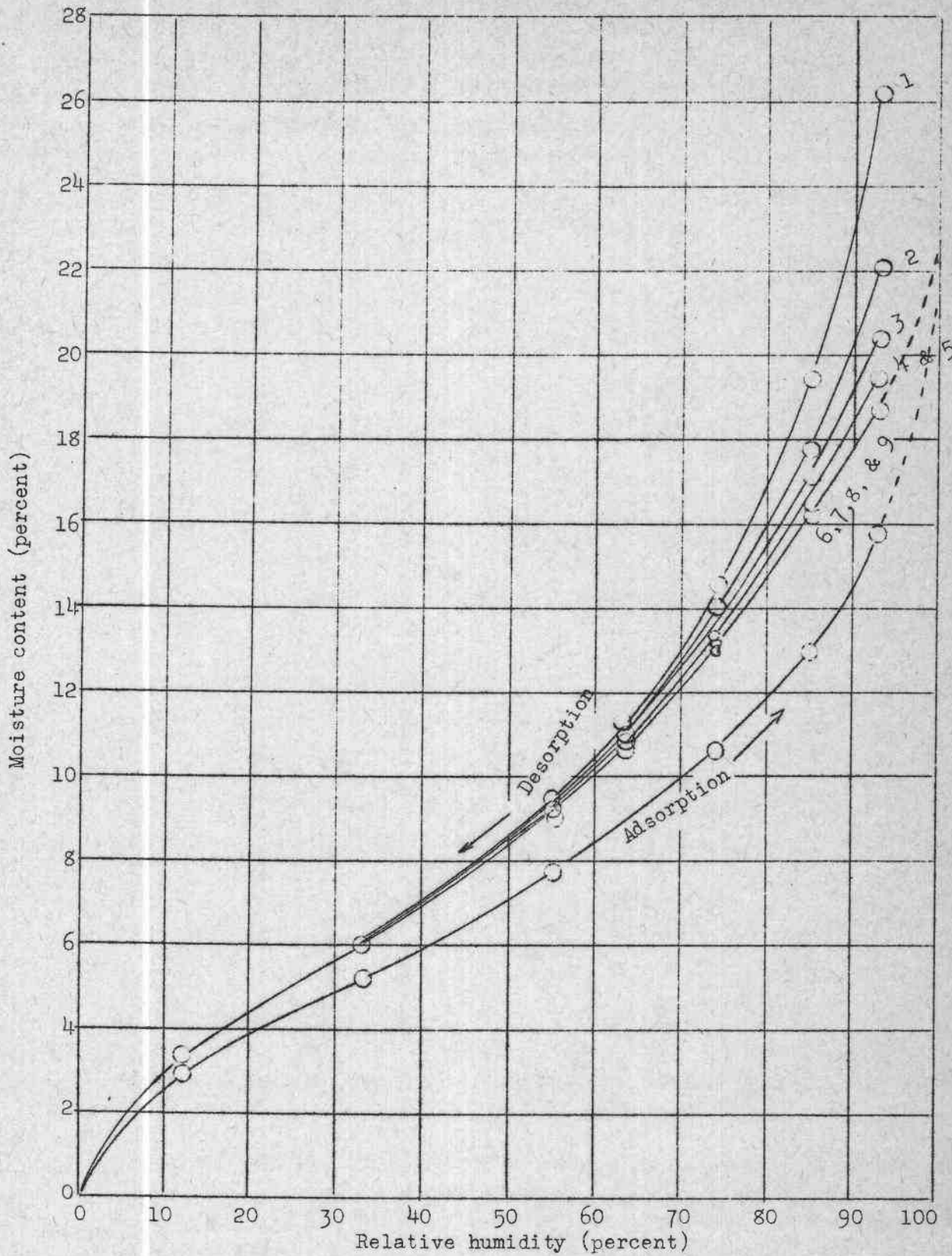


Figure 5.--Desorption isotherms from above fiber saturation of bleached unheaten western hemlock sulphite pulp.