

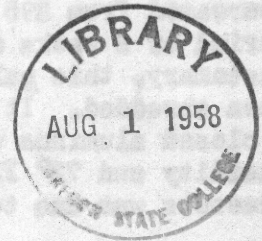
CHANGE IN THE FREENESS OF THREE MOIST STUFFS DURING STORAGE

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CHANGE IN THE FREENESS OF THREE MOIST STUFFS DURING STORAGE¹

By

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Abstract

The freeness of two softwood mechanical pulps and of a beaten hardwood sulfite pulp was determined at intervals for 32 weeks. The pulps were stored at a moisture content of about 75 percent in closed opaque containers in a room in which the relative humidity and temperature were maintained at 65 percent and 24° C., respectively. At the end of 23 weeks the freeness values of the mechanical pulps were unchanged, but at the end of 32 weeks they had decreased about 12 percent. The freeness of the sulfite pulp remained constant for 13 weeks; thereafter, it increased gradually a total of 10 percent in the next 19 weeks.

Introduction

During a study of the effect of electrolytes on the freeness of stuffs², the freeness (Schopper-Riegler) of one of the mechanical pulps used decreased from 295 to 260 cc. The change occurred at some time during a period of 2 weeks after the pulp was approximately 3 months old. As was customary, this pulp had been put over the wet machine after grinding and then shredded. It was stored during the course of the experimental work in a closed aluminum container in a room maintained at 65 percent relative humidity and 72° F. There was no evidence that the observed decrease in freeness was due to the action of bacteria or fungi.

To examine this occurrence more fully, the stability of the freeness of three other stuffs under the same storage conditions was checked over a period of 32 weeks. The results comprise the present report.

¹This report was originally written in May, 1940.

²The Effect of Acids and Other Electrolytes on Freeness. Adams, S. R., Simmonds, F. A., Baird, P. K., Tech. Assn. Papers 22:482 (1939).

Wood Used³

Groundwood pulps were prepared from Wisconsin spruce and Arkansas loblolly pine. Both samples had been cut during the current winter and stored out-of-doors until prepared for grinding. The material was, therefore, green and sound. The spruce wood was cut from two 8-foot logs and the pine from one 4-foot log. The average physical properties of the woods are given in table 1.

Table 1.-- Physical properties of spruce and loblolly pine used in freeness tests

	: Spruce	: Loblolly
	: (Shipment:	: pine
	: No. 1523):	: (Shipment
	: :	: No. 1519)
Diameter of logs (inches)	: 8.3	: 15.8
Age (years)	: 58	: 50
Growth rate (rings per inch)	: 14.2	: 6.3
Dryness (percent)	: 51.7	: 47.8
Density (O.D. weight-green volume)	: :	: :
(Lb. per cu. ft.)	: 24.8	: 26.5
Volume of heartwood (percent)	: 49	: 8.1
Volume of summerwood (percent)	: ---	: 35.6

It was not possible to determine the volume of summerwood in the spruce, as it appeared only as a fine line with no definite demarkation between it and the adjacent springwood of the same year's growth. It was estimated that the springwood content was at least 90 percent by volume.

Procedure and Results

Both woods were ground on a dull stone surface and at a high pit temperature, i.e., under conditions previously found favorable for southern pine. The different pressures employed for the two species were guessed at in advance since the runs were too short to make adjustments while grinding. The results summarized in table 2 showed that the pine pulp was somewhat below average in strength and energy consumption, indicating a lower pressure might have been employed. The spruce pulp, on the other hand, was exceptionally strong, with an energy consumption normal or slightly above normal.

³Schafer, E. R., Engineer, and Pew, J. C., Associate Engineer, prepared the spruce and loblolly pine groundwood pulps used in this study.

Table 2.--Grinding of spruce and loblolly pine,
and properties of the pulps

	: Spruce :	: Loblolly
	: (Grinder :	: pine
	: run No. :	: (Grinder
	: 342) :	: run No.
	: :	: 341
<u>Grinding Conditions</u> ¹	-----	
Pressure of wood on stone (Lb. per sq. in.)	: 32	: 16
Pit temperature (°F.)	: 190	: 190
Consistency (percent)	: 4.2	: 5.7
Dry wood ground per 24 hours (tons)	: 1.69	: 1.18
Energy consumption (H.p. days per tons wood ground)	: 70.4	: 54.2
	: :	: :
<u>Properties of the Pulp Suspension</u>	: :	: :
Freeness, Schopper-Riegler (in tap water)	: 320	: 519
Freeness, Schopper-Riegler (in distilled water)	: 259	: 462
Screen analysis:		
Retained on 24-mesh screen (percent)	: 23.8	: 15.8
Retained between 24-mesh and 42-mesh (percent)	: 7.0	: 12.0
Do..... 42-mesh and 80-mesh (percent)	: 14.3	: 22.1
Do..... 80-mesh and 150-mesh (percent)	: 12.4	: 12.4
Passing 150-mesh (percent)	: 42.5	: 37.7
Average screen opening (mm.)	: 0.100	: 0.107
	: :	: :
<u>Properties of Pulp Test Sheets</u>	: :	: :
Bursting strength, per pound per ream		
(25 x 40 - 500) -- (points)	: 0.36	: 0.15
Tearing strength, per pound per ream		
(25 x 40 - 500) -- (grams)	: 0.94	: 0.54
Tensile per square inch (pounds)	: 1985	: 834
Solid fraction	: 0.28	: 0.24

¹Other conditions were:

Stone, Norton 3760/5 N 7, surface dressed with a 10-cut, 1-1/2 inch lead spiral burr, previous service 152 hours based on three-pocket operation at 3150 feet per minute.

peripheral speed, 3150 feet per minute.

Number of pockets in simultaneous operation, 2.

Stone immersion, 9.5 inches.

Shower and dilution water, zeolite softened.

Experimental Part

Three stuffs were used. Two of these were mechanical pulps, G. R. No. 342 being made from spruce and G. R. No. 341 from loblolly pine. The grinding conditions and wood and pulp properties for these two pulps are presented elsewhere in this report. The third stuff was an unbleached red gum sulfite pulp, Digester No. 3849, which had been beaten to a freeness of 553 cc., Schopper-Riegler.

The original pulps were screened and put over the wet machine in the usual way, after which the wet webs contained approximately 23 percent of pulp calculated on the oven-dry basis. The sulfite pulp was beaten in the No. 3 (Noble and Wood) beater for 2 hours at a consistence of 2.7 percent, with the roll at about 0.003 inch above the bed plate for 1 hour and at about 0.0015 inch for 1 hour. Distilled water and the equivalent of 3.6 pounds of moisture-free pulp comprised the beater charge. At the end of the beating the stuff was dewatered by means of a centrifuge. The initial effluent from each batch centrifuged was returned to preclude the loss of fines.

All three stuffs were broken up by hand, and two master samples of each were stored in aluminum containers coated on the inside with a chlorinated rubber paint. One master sample of each pulp was treated with xylene as a preservative. All six master samples were stored in the research relative humidity room.

Prior to the beginning of the testing schedule, 40 gallons of distilled water in glass carboys were stored in the humidity room. This quantity was sufficient for the entire testing period. The hydrogen-ion concentration of the water was approximately 5.

All hydrogen-ion concentration determinations were made with the Beckman pH meter; the freeness, with a Schopper-Riegler apparatus. These tests were made in the research relative humidity room. No temperature control of test suspensions in addition to that used to maintain conditions in this room was exercised.

Standard freeness suspensions from each master sample, to which no preservative had been added, were titrated with standard hydrochloric acid to obtain pH-acid volume curves at the beginning and end of the total storage period. This was done because in the electrolyte-freeness study² it had been observed that after the freeness of the groundwood had decreased during the period the pulp was in storage less of either aluminum sulfate or chloride was required to adjust a suspension of the pulp in distilled water to a given pH value. It, therefore, appeared possible that whatever change was responsible for the decrease in the acid demand of the pulp in this one instance might always occur with like freeness changes.

When weighing samples for testing, the container was first well shaken and then a sample taken as quickly as possible and the can closed. Any excess was discarded to help maintain a constant moisture content in the master

sample. In the case of the pine and spruce groundwoods, the sample of approximately the right weight was wadded in the weigher's hand (not squeezed) and the excess was picked off of this wad with tweezers. This was found to be a more accurate means than just heaping the pulp on the watch glass, in that less moisture loss occurred during weighing. All freeness determinations were made in triplicate.

Since the master samples were at an oven-dry fiber content of 23 percent, it was necessary to agitate each test suspension with a high speed impeller type of mixer to insure a uniform suspension free from fiber aggregates. The time of this agitation was exactly 4 minutes in all cases.

At the beginning of each period of testing all of the apparatus with which the suspensions were to come in contact was washed carefully with the standard supply water. The pH value of this water also was found to remain constant at 5.1 for all carboys as long as they remained full and unopened. After, however, a carboy had been opened and stood, for example, half full for a few days, the pH increased to about 5.4 due to the loss of carbon dioxide.

Standard procedure was followed in the freeness determination, with the exception that higher temperatures obtained which varied from 24° to 25° C.

Previous experience had shown that a temperature variation of 1 degree does not have a measurable effect on the freeness value. The top meniscus was read in adjusting the suspension volume to 1,000 cc.

Discussion of Results

The results in table 3 show that at the end of about 31 weeks the freeness of the two mechanical pulps decreased. The decrease amounted to 11 percent for the spruce pulp and 12.6 percent for the loblolly pine. This confirmed the decrease of 11.8 percent observed in the freeness of the mechanical pulp used in the electrolyte-freeness study. For all three, the average decrease in freeness during storage under the specified conditions was approximately 12 percent.

The freeness of the beaten, unbleached red gum sulfite pulp also changed during storage but, unlike the groundwood, the change was an increase. On a percentage basis, it amounted to 9.8 percent.

The hydrochloric acid-pH titration curves for these three stuffs both at the beginning and end of the storage period are in figure 1. No significant difference was observed in the amount of acid required to obtain the same pH value before and after storage. It is of interest that the titration curves for the two mechanical pulps coincided and also that, down to a pH of about 3.5, the acid demand of the sulfite pulp was a little less than that

Table 3.--Schopper-Riegler freeness values of stuffs at intervals during storage in opaque closed containers stored at 65 percent relative humidity and 72° F.

Storage: time	Mechanical pulp suspensions						Chemical pulp suspensions		
	Spruce		Loblolly pine		Red gum sulfite				
	Temper- :	H-ion :	Free- :	Temper- :	H-ion :	Free- :	Temper- :	H-ion :	Free- :
	ature :	concen- :	ness :	ature :	concen- :	ness :	ature :	concen- :	ness :
	tration :	tration :	tration :	tration :	tration :	tration :	tration :	tration :	tration :
Weeks	°C.	pH	Cc.	°C.	pH	Cc.	°C.	pH	Cc.
0	: 24	: 6.5	: 236	: 24	: 6.5	: 450	: 24	: 6.2	: 553
1	: 24	: 6.4	: 230	: 24	: 6.4	: 447	: 24	: 6.4	: 540
2	: 24	: 6.4	: 234	: 24	: 6.3	: 449	: 24	: 6.4	: 552
3	: 24	: 6.4	: 231	: 24	: 6.5	: 452	: 24	: 6.4	: 542
4	: 24	: 6.4	: 234	: 24	: 6.5	: 453	:	: 6.5	: 556
5								
6	: 25	: 6.5	: 232	: 25	: 6.5	: 452	: 25	: 6.5	: 550
7	: 24	: 6.5	: 230	: 24	: 6.5	: 457	: 24	: 6.4	: 558
8	: 24	: 6.4	: 230	: 24	: 6.5	: 461	: 24	: 6.4	: 556
9	: 24	: 6.4	: 228	: 24	: 6.4	: 457	: 24	: 6.4	: 557
10	: 24.5	: 6.6	: 225	: 24.5	: 6.5	: 455	: 24.5	: 6.4	: 555
11	: 25	: 6.6	: 230	: 25	: 6.6	: 448	: 25	: 6.6	: 560
12	: 24	: 6.5	: 223	: 24	: 6.5	: 451	: 24	: 6.5	: 555
13	: 24	: 6.5	: 220	: 24	: 6.5	: 447	: 24	: 6.5	: 554
19	: 24.5	: 6.5	: 233	: 24.5	: 6.5	: 468	: 24.5	: 6.2	: 585
19	: 24.5	: 6.2	: 238	: 24.7	: 6.4	: 467	: 24.5	: 6.1	: 600
19								
23	: 25	: 6.2	: 239	: 25	: 6.5	: 445	: 25	: 6.0	: 605
27	: 23.5	: 6.3	: 215	: 24	: 6.5	: 419	: 24	: 6.3	: 614
31-32	: 23.5	: 6.5	: 210	: 23.5	:	: 393	: 23.5	: 6.2	: 607

¹Suspensions prepared with a new supply of distilled water.

²Determinations made on samples to which the xylene was added as a preservative.

of the mechanical pulps. The fact that there was no change in the acid requirement of the mechanical pulps after the freeness had changed was contrary to expectation.⁴

Conclusions

The freeness of chemical and mechanical pulps when stored at a moisture content of about 75 percent and moderate temperature in closed containers will very likely remain essentially constant for at least 2 months even when the containers are opened occasionally for sampling.

The length of storage before a freeness change does occur varies among pulps as does the amount of change.

The observed freeness changes were not accompanied by any increase in the acidity of the moist pulps.

⁴Since the experimental work on this problem was completed it has been observed in a rate-of-flow study that dewatering a stuff by suction filtration but with no loss of fines increases the freeness value. It is, therefore, likely that, had the stuffs used in the present study been stored at the consistence at which they were processed (approximately 2 percent), an even greater change in freeness would have been observed. The results indicated that, once a stuff has been dewatered to about 25 percent fiber content on the oven-dry basis, the property of freeness is stable for a period of approximately 3 months. This appeared to be the upper limit of safety, however, and obviously refers only to freeness.

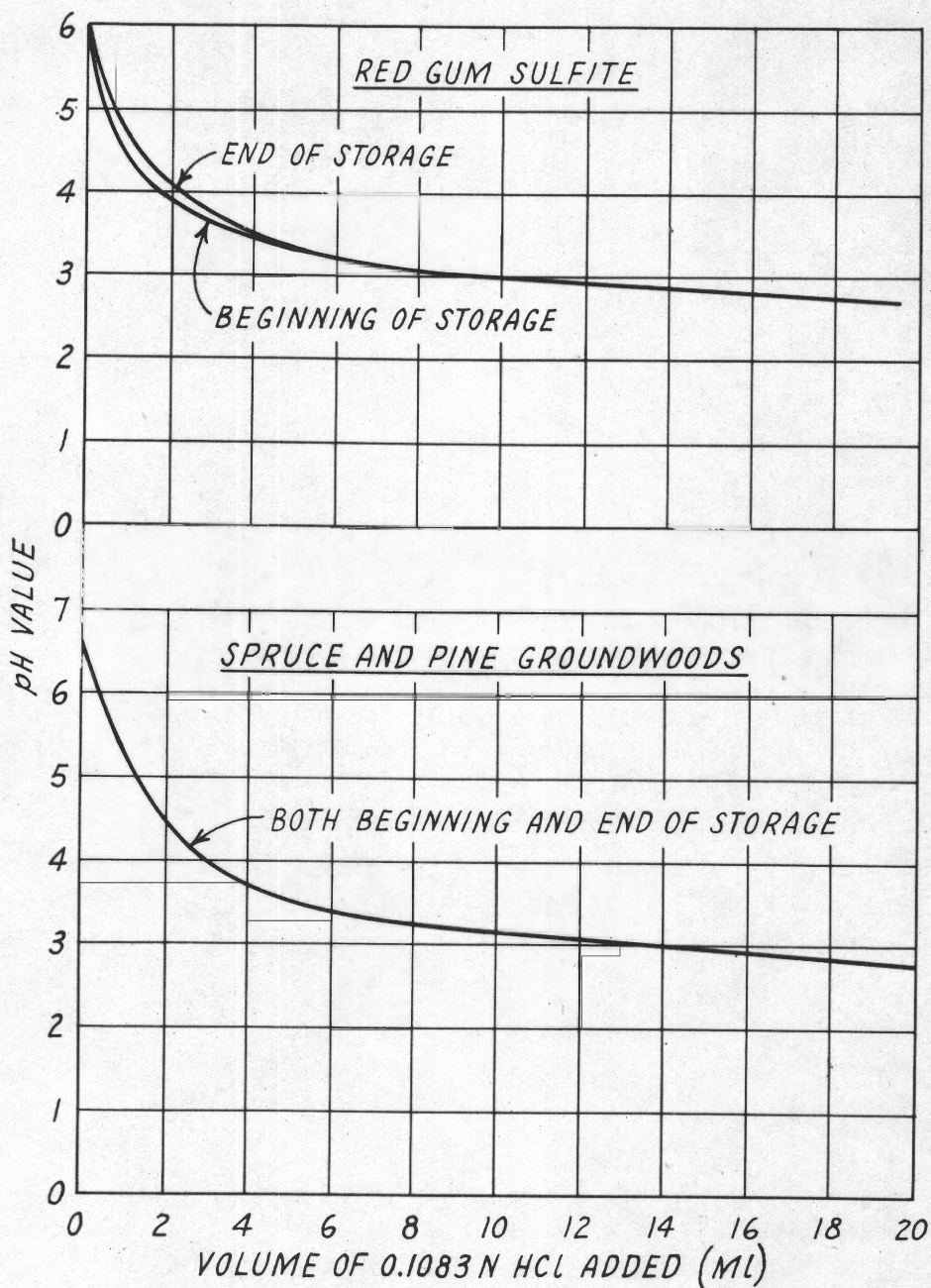


FIG. 1
 pH-ACID VOLUME CURVES FOR SUSPENSIONS
 OF THREE STUFFS CONTAINING 2 GM. OF FIBER PER LITER
 Z M 43803 P