





NBSIR 77-1286

Characterizing the Interfiber Bond Strength of Paper Pulps in Terms of A Breaking Energy: Effect of Beating

Jack C. Smith and Edmond L. Graminski

Polymers Division Institute for Materials Research National Bureau of Standards

December 15, 1976

Progress Report Covering the Period July 1, 1976 through September 30, 1976

Prepared for U. S. Energy Research and Development Administration Washington, D.C. 20234

CHARACTERIZING THE INTERFIBER BOND STRENGTH OF PAPER PULPS IN TERMS OF A BREAKING ENERGY: EFFECT OF BEATING

Jack C. Smith and Edmond L. Graminski

Polymers Division Institute for Materials Research National Bureau of Standards

December 15, 1976

Progress Report Covering the Period July 1, 1976 through September 30, 1976

Prepared for U. S. Energy Research and Development Administration Washington, D.C. 20234



U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, Secretary

Dr. Sidney Harman, Under Secretary Jordan J. Baruch, Assistant Secretary for Science and Technology NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Acting Director



TABLE OF CONTENTS

SUMMARY AND	CONCLUSIONS	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	٠	•	•	•	•		i
INTRODUCTION	N	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		1
EXPERIMENTA	Б	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	2
RESULTS AND	DISCUSSION	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	3
REFERENCES		•	•	•	•	•	•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•		•		6

Page



SUMMARY AND CONCLUSIONS

An energy parameter, E, is being investigated for possible use in characterizing the adhesion between two paper fibers. A handsheet in the form of a thin open web is made from the pulp fibers to be evaluated, and E is determined from a tensile test that records the force and number of bond breaks, as a specimen from this web is elongated. Details of the method for measuring E and preliminary tests to determine its applicability in characterizing Northern and Southern softwood kraft pulps have been described in a previous report [1]. E was shown to have approximately the same value for both unbeaten pulps. Beating to 10,000 revolutions in a laboratory beater increased this value ninefold. Although it was not possible to distinguish between the two pulps by means of the E values alone, some differences were noted in the mechanical behavior of handsheet specimens from the two different pulps. Webs from the unbeaten Northern pulp had a tighter structure with more bonds between fibers than was the case for comparable webs made from Southern pulp.

In the work reported here the effect of beating has been examined in more detail. Handsheet specimens were prepared from Northern and Southern softwood kraft pulps beaten to 1,000, 2,000 and 5,000 revolutions. The method of preparation was the same as that used previously except that a sheet of filter paper was used as a forming medium, and the webs were pressed at a lower pressure (44 kPa for 5 min).

The values of E for both unbeaten pulps were comparable, approximately 0.8×10^{-7} J/break. The effect of beating to 5,000 revolutions was to increase E for both pulps fivefold to a value of approximately 4×10^{-7} J/break. E for the Northern pulp increased rapidly with beating to 2,000 revolutions, and then increased more slowly. In contrast E for the Southern pulp increased slowly with beating to 2,000 revolutions, and then increased more rapidly. There is, however, some uncertainty to these latter results, because of imprecision in the value of E.

If the elongation of a specimen versus the number of bond breaks is plotted, the initial slope of this curve, or initial

i

elongation per break, can be used as a measure of the tightness of the web structure; i.e., the density of interfiber bonds per unit area of web. Large values of the initial elongation per break thus indicate a loose web in which the area density of fibers is small and in which many of the crossing fibers are not bonded together. It was found that the unbeaten Northern pulp sample had an initial elongation per break of 0.16 %, and that subsequent beating produced only a slight decrease to a value of 0.12 %/break. Thus the beating treatment only resulted in a slight tightening of the web. In contrast the initial elongation per break for the unbeaten Southern pulp was 0.52 % indicating a relatively loose web. Beating to 1,000 revolutions brought the value down to 0.18 %/break, and further beating produced a small decrease to 0.16 %/break.

The observed effects of beating on the strength of the interfiber bonds and on the tightness of the fiber web are consistent with the known morphologies of the two pulps. The unbeaten Northern pulp fibers form tighter webs because, unlike Southern pulp fibers, these fibers tend to collapse to a ribbon-like structure and thus become more conformable. Beating induces the formation of ribbonlike structures, and increases the number and area of the interfiber bonds in the web.

Webs formed from Southern pulp were looser than webs of the same basis weight (weight per unit area of web) formed from Northern pulps, even after the pulps had been beaten 5,000 revolutions. As Southern pulp fibers are coarser than Northern pulp fibers, webs of the Southern pulp would have fewer fibers per unit area in webs of the same basis weight, and hence fewer bonds on this account. It seems that better comparisons between pulps might be obtained if the webs tested had the same number of fibers per unit area rather than the same basis weight. It is planned in future work to test this idea by making measurements of E on a series of webs of different basis weights in order to obtain a closer match between webs from different pulps.

The webs used in this work were formed at low pressures. When they were tested it was difficult to measure E with satisfactory certainty. It therefore will be necessary to experiment with webs formed at various pressures, in order to develop webs that are more suitable for these measurements, but are still easy to manufacture.

ii

INTRODUCTION

If a very thin web of pulp fibers is elongated to break in a sensitive tensile tester, a force-elongation curve containing numerous jags is obtained. Each jag is caused by the breakage of a bond between fibers constituting the paper network. From a plot of work of extension as a function of the number of bond breaks, the average energy stored in the fibers previously connected by the broken bond can be found. This energy parameter E is being investigated for possible use in characterizing the adhesion between two paper fibers.

Applicability of the energy parameter E in the characterization of Northern and Southern softwood kraft pulps is being investigated . Preliminary results are given in a previous report [1]. It was shown that the parameter E had the same value for the unbeaten Northern and Southern pulps, and almost the same value when both pulps were beaten to 10,000 revolutions. However the beating treatment produced an increase in the value of E from 0.8×10^{-7} J/break for the unbeaten pulps up to 7×10^{-7} J/break for the beaten pulps. In the work reported here the effect of beating has been examined in more detail.

EXPERIMENTAL

Handsheets of basis weight 2.5 g/m^2 were prepared from the batches of Northern and Southern softwood kraft pulps used previously. The pulps were unbeaten and beaten to 1,000, 2,000 and 5,000 revolutions in a PFI laboratory beater. The method of preparation was the same as that reperted previously except for the following changes: It was found that filter paper (Whatman No. 541) could be substituted for the cotton linters pulpsheet previously used as a forming medium. It was usually not necessary to treat the filter paper with a fluorocarbon release agent. Pressing for five minutes in a hydraulic press was carried out at 44 \times Pa (6.4 $1b/in^2$) instead of the previous 0.66 Mpa (96 $1b/in^2$), as this produced a coherent web that was easy to separate from the ferrotype backing plate.

The procedure for preparing, mounting, and testing of specimens from the handsheet samples was unchanged. Specimen dimensions were 1 cm width by 2 cm length. The specimens were tested at a cross head speed of C.2 cm/min and a chart speed of 20 cm/min. Load cell sensitivities ranged between 0.02 N (2g) full scale for handsheets of unbeaten pulp and O.1 N (10 g) full scale handsheets of pulp beaten to 5000 revolutions.

RESULTS AND DISCUSSION

The force-elongation curve obtained by extending fiber web specimens in a tensile tester can be analyzed to provide two types of data. Data for a curve of work of extension versus number of bond breaks can be obtained by integrating under the force-elongation curve and counting the discontinuities in slope caused by the bond breaks. The characteristic energy E is estimated from the minimum slopes of this curve in accordance with guiding principles given previously [1]. A curve of percent elongation of the specimen versus number of bond breaks can also be plotted. The initial slope of this curve, or initial percent elongation of the specimen per break, is a measure of the tightness of the web structure, as explained later.

The results from testing five specimens of each of the handsheet samples are given in tables 1 through 8. It can be seen from these tables that values of E for both unbeaten pulps are comparable, about 0.8×10^{-7} J/break. The effect of beating to 5,000 revolutions is to increase E for both pulps fivefold to a value of approximately 4×10^{-7} J/break.

In rigure 1 the average values and standard deviations of the characteristic energy E are plotted to facilitate comparison. This plot suggests that adhesion between Northern pulp fibers, as measured by E, improves most rapidly at the start of beating, and that increased beating produces a lower rate of improvement. In contrast, adhesion between fibers of Southern pulp does not increase as rapidly with beating initially, but increases more rapidly as beating is continued. The standard deviations associated with the values of E are rather large, however, so this apparent behavior must be confirmed by further measurements.

The relative tightness of the web structures can also be determined, according to the following rationale. A tightly bonded structure is one that has a large number of interfiber bonds per unit area. The number of these bonds depends

upon the number of fiber crossings and on the number of crossings that bond together. If the structure is tightly bonded, it can be seen intuitively that on the average only a small percent extension is required before the force applied to some one bond is sufficient to break it. For a loosely bonded network a larger percent extension on the average is required. Thus a small value of average percent extension per break would be characteristic of a tight network, and a larger value of average percent extension would be characteristic of a looser network. The average percent extension per break for a given web specimen is equal to the initial slope of the curve of percent extension versus number of breaks obtained by a tensile test.

Values of the initial percent elongation per break are plotted in Figure 2. It is apparent from this figure that the unteaten Northern pulp sample has an initial elongation per break of 0.16 %, and subsequent beating produces only a slight decrease to a value of 0.12 %/break. Thus the beating treatment only resulted in a slight tightening of the web. In contrast the initial elongation per break for the unbeaten Southern pulp is 0.52 %, indicating a relatively loose web. Beating to 1,000 revolutions, however, brings the value down to 0.18 %/break, and further beating to 5,000 revolutions produces a small decrease to 0.16 %/break.

The effects of beating on the strength of the interfiber bonds and on the tightness of the fiber web can be explained in terms of the morphologies of the two kinds of pulp fibers. It is known that unbeaten Northern pulp fibers have the shape of thinwalled tubes, most of which have collapsed to form a ribbon-like structure. Therefore most of the junctions between fibers of unbeaten Northern pulp will have large areas of contact, will bond easily, and produce a tight web. The effect of beating is to make the fibers more pliable and to reduce more fibers to a collapsed ribbon-like state. The increase in pliability should produce more intimate contacts between fibers and increase the bon. strength. The increased number of ribbon-like structures should provide more bonds and make the web tighter.

Fibers from unbeaten Southern pulp are coarser than those from unbeaten Northern pulp. They have the shape of thick-walled round hollow tubes, fewer of which have collapsed to a ribbon-like shape. Therefore fewer fiber junctions will have sufficiently large areas of contact to produce an effective bond, and the resultant web will be looser. Only a little beating is needed to collapse more of the tubes, thus producing more bonds and providing an appreciably tighter web, but more beating is required to make the fibers pliable and increase the bond strength.

Webs from Northern pulp beaten to 5,000 revolutions have an initial elongation to break of 0.13 %, and webs of the same basis weight made from Southern pulp with the same degree of beating have an initial elongation per break of 0.16 %. Although specimen variation prevents good resolution, this difference in value should be expected because of the difference in coarseness of the two pulps. A web made from the coarser Southern pulp has fewer fibers and fiber crossovers per unit area than a web of the same basis weight made from the Northern pulp.

As stated in the experimental section, the handsheet samples prepared for these experiments were formed at low pressures (44kPa for 5 min). Coherent webs were obtained that were easily separated from the forming medium and backing plate. However, the derived curves of work of extension versus number of bond breaks had a scalloped appearance with many changes in slope. As a rule the minimum slopes extended over only a few bond breaks, so the value of the slope was uncertain, and it was difficult to obtain a good estimate of E. This difficulty was not so prevalent in previous measurements on the webs which were formed at a higher pressure. It therefore will be necessary to experiment with webs formed at various pressures, in order to develop webs that are more suitable for these measurements, but are still easy to manufacture.

REFERENCES

 J. C. Smith and E. L. Graminski, Characterizing the Inter-Fiber Bond Strengths of Paper Pulps in Terms of a Breaking Energy, Progress report covering the period Jan. 1, 1976 to June 30, 1976, NBSIR - 1148. Available from National Technical Information Service PB 264,689. Table 1. Tests on Specimens of Northern Pulp, Unbeaten

Specimen	Ē	Init. Elong.
	_	per break
	10 ⁻⁷ J/break	%/break
l	0.75	0.12
2	0.64	0.14
6	0.70	0.22
8	0.77	0.16
13	0.92	0.18

Average \pm standard deviation E (0.76 \pm 0.10)x10⁻⁷ J/break. Initial elongation per break 0.16 \pm 0.04 %/break.

Table 2.

Tests on Specimens of Southern Pulp, Unbeaten

Specimen	Ē	Init. Elong.
	_	per break
	10 ⁻⁷ J/break	%/break
3	0.76	0.28
4	1.28	0.47
5	0.55	0.31
10	1.12	0.74
15	0.69	0.80

Average ± standard deviation

E $(0.88 \pm 0.31) \times 10^{-7}$ J/break.

Initial elongation per break 0.52 ± 0.24 %/break

Table 3. Tests on Specimens of Northern Pulp, Beaten 1,000 Revolutions

Specimen	E	Init. Elong.
	-	per Break
	10 ⁻⁷ J/break	%/break
2	1.42	0.12
3	1.44	0.11
9	1.92	0.14
10	1.32	0.14
11	0.96	0.12

Average ± standard deviation E (1.41 ± 0.34)x10⁻⁷ J/break Initial elongation per break 0.12 ± 0.01 %/break

Table 4.

Tests on Specimens of Southern Pulp, Beaten 1,000 Revolutions

Specimen	E	Init. Elong.
		per break
	10 ⁻⁷ J/break	%/break
1	1.40	0.21
2	1.24	0.22
8	1.50	0.18
9	1.00	0.17
10	1.32	0.15

Average ± standard deviation

 $E (1.29 \pm 0.19) \times 10^{-7} \text{ J/break}$

Initial elongation per break 0.18 ± 0.03 %/break

Table 5. Tests on Specimens of Northern Pulp, Beaten 2,000 Revolutions

Specimen	E	Init. Elong.
	10 ⁻⁷ J/break	per Break
	10 0/ DI CAR	joj bi cak
1	2.70	0.12
2	1.80	0.12
9	2.10	0.12
10	2,64	0.12
11	2.24	0.12

Average \pm standard deviation E (2.30 \pm 0.38)x10⁻⁷ J/break

Initial elongation per break 0.12 ± 0.00 %/break

Table 6.

Tests on Specimens of Southern Pulp, Beaten 2,000 Revolutions

Specimen	E	Init. Elong.
		per Break
	10 ⁻⁷ J/break	%/break
2	1.80	0.19
5	1.42	0.22
9	1.88	0.16
11	1.72	0.18
12	1.42	0.21

Average ± standard deviation

 $E (1.65 \pm 0.22) \times 10^{-7} \text{ J/break}$

Initial elongation per break 0.19 ± 0.02 %/break

Table 7.

Tests on Specimens of Northern Pulp, Beaten 5,000 Revolutions

Specimen	E	Init. Elong.
	_	per Break
	10 ⁻⁷ J/break	%/break
1	4.76	0 17
1	1.10	0.T!
2	3.16	0.13
9	3.10	0.13
10	2.86	0.13
13	3.02	0.11

Average ± standard deviation E (3.36 ± 0.79)x10⁻⁷ J/break Initial elongation per break 0.13 ± 0.02 %/break

Table 8.

Tests on Specimens of Southern Pulp, Beaten 5,000 Revolutions

Specimen	Ē	Tnit. Elong.
		per Break
	10 ⁻⁷ J/break	%/break
3	4.38	0.17
9	4.04	0.18
10	4.04	0.18
12	3.28	0.15
13	4.70	0.12

Average ± standard deviation

 $E (4.09 \pm 0.53) \times 10^{-7} \text{ J/break}$

Initial elongation per break 0.16 ± 0.02 %/break





Characteristic Energy E as a Function of Degree of Beating. Open circles, Northern pulp; solid circles, Southern pulp.



BEATING, REVOLUTIONS

Figure 2.

Initial Elongation per Break as a Function of Degree of Beating. Open circles, Northern pulp; solid circles, Southern pulp.

NBS-114A (REV. 7-73)			
U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBSIR 77-1286	2. Gov't Accession No.	3. Recipient's Accession No.
4. TITLE AND SUBTITLE Characterizing the	Inter-Fiber Bond Strength	of Paper	5. Publication Date
Pulps in Terms of	a Breaking Energy: Effect	of Beating	6. Performing Organization Code
7. AUTHOR(S) Jack C. Smith	and Edmond L. Graminski		8. Performing Organ. Report No.
9. PERFORMING ORGANIZAT	ION NAME AND ADDRESS		10. Project/Task/Work Unit No.
DEPARTMEN WASHINGTON	T OF COMMERCE		11. Contract/Grant No.
12. Sponsoring Organization Nat	me and Complete Address (Street, City, S	tate, ZIP)	13. Type of Report & Period Covered
			14. Sponsoring Agency Code
15. SUPPLEMENTARY NOTES			
16. ABSTRACT (A 200-word or bibliography or literature su Handsheets in from Northern and beaten to 1,000, 2 density of bonding estimated from ten equal, but density Bond density and s beating to approxid differences were co	less factual summary of most significant rvey, mention it here.) the form of a thin web of Southern softwood kraft pul ,000 and 5,000 revolutions between fibers and the rel sile test data. Bond stren of bonding was greater in trength for both pulps incr mately the same value for p bserved in the rate of chan	information. If docume basis weight 2.5 ps. The pulps w in a laboratory ative strength o gths of the two the web made fro eased with incre- ulps beaten 5,00 ge of these quar	2 9 g/cm ² were prepared vere unbeaten and beater. The relative of the bonds were unbeaten pulps were wm the Northern pulp. eases in the degree of 10 revolutions, but htities.

7. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)

Adhesion of paper fibers; bonding of paper fibers; paper; paper fibers, bonding; paper pulps, characterization; paper, tensile tests.

8.	AVAILABILITY [X] Unlimited	19. SECURITY CLASS (THIS REPORT)	21. NO. OF PAGES
	For Official Distribution. Do Not Release to NTIS	UNCL ASSIFIED	17
	Order From Sup. of Doc., U.S. Government Printing Office Washington, D.C. 20402, <u>SD Cat. No. C13</u>	20. SECURITY CLASS (THIS PAGE)	22. Price
	X Order From National Technical Information Service (NTIS) Springfield, Virginia 22151	UNCLASSIFIED	\$3.50

USCOMM-DC 29042-P74

