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The Effect of the Cold Soda Pulping Process on the Various Constituents of Aspen Wood

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THE EFFECT OF THE COLD SODA PULPING PROCESS

ON

THE VARIOUS CONSTITUENTS OF ASPEN WOOD /

A

dissertation

submitted to the faculty

of

Western Michigan University

by

William A. Foster

In partial fulfillment of
the prerequisites for the degree

of

Bachelor of Science

June, 1958

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LITERATURE SURVEY

The commercial Cold Soda Process for wood is a relatively new pulping process developed at the United States Forest Products Laboratory at Madison, Wisconsin, for high-yield pulping of hardwoods.

The development of this process has been due to the increasing shortage of softwood papermaking fibers. This shortage of softwood fibers has made necessary the need for a suitable process for pulping hardwoods. The Cold Soda process is thought to meet some of the economic and strength requirements of this need.

Most of the research done on this subject has been done in the 85 to 95 yield range at the Forest Products Laboratory. This naturally follows from the fact that the Cold Soda process is a high-yield process and almost all interest to date has been in this range.

It should be noted that the few articles published have been concerned almost wholly with strength and economic characteristics, which are of primary concern to industry. Only one article has shown the effect of the process on chemical composition of the pulp.

Brown and McGovern have done some interesting work in the 85 to 95 yield range (1, 2). They varied time, temperature and chemical concentration in search of the optimum point for strength and yield. The treatments were made with caustic concentrations of 24 and 63 grams per liter, reaction periods of from 15 minutes to 5 hours, and temperatures of from 25 to 90 degrees centigrade. The optimum condition for treatment variables was found to be a 2-hour treatment at 25 degrees with a caustic concentration of 24 grams per liter. In this treatment the chemical consumption was about 5.5 per cent on the weight of the wood and the yield was about 91 per cent.

They also found that within the range of their experiments there was some hemi-cellulose removal but little or no loss of lignin or alpha-cellulose (2). This would tend to indicate that loss of yield was due primarily to loss of hemi-cellulose. They have shown that with the same time and temperature, an increase in caustic concentration of from 24 to 63 grams per liter produced a corresponding decrease in yield of from 90.4 to 88.4 per cent. This change produced a slight increase in both tear and mullen (1).

The energy requirement for fiberizing this type pulp was within the range of commercial practicability

and usually fell within a range of from 20 to 30 horsepower-days per ton of air dry pulp (1).

Brown and Monsson have done some work on forced-liquor penetration in the Cold Soda Process (3). They found that the optimum treatment variables for this modification of the process are a temperature of 25 degrees, a time of 1 hour, and a chemical concentration of 50 grams per liter. The important point shown here is the cut in the time factor over the atmospheric steep method.

Brown, in further studies on the Cold Soda Process, found that the amount of caustic soda liquor absorbed by aspen and southern red oak woods was increased by decreasing the moisture content of the wood, by decreasing the particle size, by increasing the hydrostatic pressure in the treating vessel, or by removing the air from the wood prior to treatment (10).

The forementioned articles show the mullen for the atmospheric steeped pulp to be approximately 0.45 points per pound and the tear to be approximately 0.98 grams per pound for a 25 x 40-500 sheet.

Villars has used variables of a time of 4 hours, a temperature of from 15 to 20 degrees C., and a caustic solution of 1 part NaOH to 10 parts wood with a liquor

to wood ratio of 4 to 1 (4). He was concerned primarily with hydration and reached 53 to 70 degrees S. R. with yields of from 70 to 85 per cent. He stated that an increase in temperature produced some improvement in tear resistance.

To date the major uses of Cold Soda Process pulp has been for corrugating medium. This pulp has also been used as a raw stock component for coated papers.

It should be noted that this discussion is limited to the Cold Soda Process. The soda process and the kraft process are both basically NaOH processes, but they are used primarily with higher temperatures and pressures and the reactions are, therefore, different.

A word is now in order as to the nature of the chemical action of caustic soda on the various components of wood in the 25 degree temperature range. The components referred to will be lignin, hemi-cellulose, and alpha-cellulose.

The mechanism by which lignin goes into solution in the alkaline process appears to involve three separate steps (5):

1. Absorption of alkali at the lignin-liquor interface by acidic phenolic groups in the lignin complex.
2. A chemical combination between the lignin and the absorbed alkali.

3. Chemical hydrolysis and separation of the
alkali-lignin complex from the lignin surface.

This mechanism occurs under conditions prevailing in alkaline pulping with formation of new hydroxyl groups. However, it appears that the single phenolic or enolic hydroxyl group in the highly polymerized lignin complex is not sufficient to bring about solution in NaOH at atmospheric conditions of pressure and temperature (5).

The hemi-cellulose fraction of wood is almost wholly soluble in dilute caustic solution, although it is less soluble in cold than in hot solution (7). In alkaline pulping the hemi-celluloses which are removed in the black liquor appear to be changed into saccharinic acids.

Alkali hydroxides act as swelling agents for all forms of cellulose and as solvents for modified or degraded forms of cellulose. A slight amount of alkaline hydrolysis takes place in the cellulose when it is subjected to attack by NaOH solution (8). However, alpha-cellulose, the more stable portion of cellulose, is defined on the basis of its insolubility in 17.5 per cent NaOH at 20 degrees centigrade. Repeated treatments with alkali increase the amount of cellulose that goes into solution, showing that alkali has some degrading action on cellulose (9).

EXPERIMENTAL PROCEDURE

As the title indicates, the objective of this work is to determine the effect of the Cold Soda pulping process on the chemical composition of pulp from quaking aspen wood.

To do this a sample of the wood was treated by this process and a sample of this pulp was analyzed chemically. A sample of the wood was also analyzed chemically and the difference between the two analyses noted.

A Beater run was made on the pulp and handsheets were made and tested. These physical strength data were used to characterize the pulp.

The following specialized equipment was used in this thesis:

Rotary bicycle-wheel digester. This consisted of a bicycle wheel placed on an axle and mounted on a wooden frame. This wheel was connected through a belt drive to a one-sixth HP motor in such a manner that it rotated eight revolutions per minute. Four two-quart jars were mounted on this wheel with wire and the chips and treating liquor were placed in these jars.

A laboratory Bauer refiner with both breaker plates and fine plates. This refiner must be adjustable to give various degrees of refining.

A motor-driven shaker screen made by Valley Iron Works. This screen handled pulp in the consistency range of approximately one half per cent. It was also equipped with a pump.

A laboratory beater, British sheet mold, handsheet press, and a Canadian Standard freeness tester for the beater run and handsheet formation.

The standard paper mill laboratory paper testing devices.

EXPERIMENTAL WORK.

Chemical Treatment.

The treatment was made on quaking aspen wood chips. These chips had been obtained from a normal, 10-blade, industrial chipper and were of a reasonably uniform size. However, to assure uniformity the chips were screened further. The chips accepted were ones which would pass through three-quarter inch square holes upon shaking but would not pass through one-half inch square holes.

The apparatus used for the digester consisted of a bicycle wheel mounted on a wooden frame and driven by a one-sixth HP motor in such a way that it would rotate eight revolutions per minute. Four two-quart jars were then mounted on this wheel with wire. The chips and caustic liquor were then put into the jars and rotated on the wheel for two hours. At the end of this time the wheel was stopped and the jars were taken off. The spent liquor was then decanted off into a crock for determination of chemical consumption and the chips were put into a larger crock for washing.

At the start of the treatment each jar was filled with 300 grams of air dry chips. As the moisture content of the chips was 10.35 per cent and there were four jars the total oven dry weight of the batch was 1075 grams. Into each jar of chips were poured 900 ml. of sodium hydroxide at a concentration of approximately 50 grams per liter. This liquor was tested later, however, and found to have a concentration of 53.1 grams per liter.

The treated chips which were put into a crock and were washed with cold water until the water appeared clear. This consisted in filling the crock which contained the chips with water and swirling the chips for about one minute. This water was then decanted off. The wash water was also poured into another crock and

saved for further measurement. This procedure was repeated for a total of four times.

Physical Action on Treated chips.

The washed chips were given one pass through the breaker plates in a laboratory Bauer refiner. The coarse refined chips were dewatered in a laboratory tub screen. These chips were then put through the fine plates of the Bauer. With the fine plates in place the Bauer was set so that it pulled just three amperes with the normal amount of water running through it. After two passes through the fine plates the pulp was watered down to approximately one-half per cent consistency and started through the laboratory shaker screen.

After approximately half of the pulp had been put through the screen it was noticed that the rejects amounted to about 50 per cent. At this point the screening was stopped and the rejects and the rest of the pulp were sent through the fine plates of the Bauer again. This time, however, the plates were set so that the refiner pulled 3.5 amperes with water alone.

The pulp was again screened and this time there were very few rejects. The pulp from the shaker screen was pumped into the tub screen for dewatering. After the pulp had all been screened and dewatered it was

placed in a crock and brought back up to about 1.5 per cent consistency for a yield determination.

Yield.

After the pulp had been placed in the crock and diluted for the yield determination the volume of the pulp in the crock was calculated. The consistency of the pulp was then found by weighing a definite amount of the pulp and determining its oven dry content after drying to constant weight.

The total weight of oven dry pulp was calculated, and this value was used to ascertain the per cent yield.

Chemical consumption.

The spent liquor and the wash liquor were measured and samples of these two liquids were titrated with sulfuric acid to determine the amount of caustic soda present. The total weight of unused caustic was thus obtained.

A sample of the treating liquor was titrated in a similar manner. The total weight of the original was thus determined. The difference between the original and residual caustic represented the caustic used by the wood. The per cent chemical consumption based on the weight of wood was then calculated.

Beater run.

The consistency at which the yield was measured was too low for use in the beater. Therefore, the stock was dewatered and another consistency taken. The correct amount of stock was then weighed out (360 grams, oven dry) and placed in the beater.

The beater run was made according to TAPPI Standard T 200 m-45. Handsheets were made according to TAPPI Standard T 205 m-53 and conditioned for seven days at 73 degrees Fahrenheit and 50 per cent relative humidity.

The following tests were run on the handsheets:

Basis Weight (used to convert strength tests
to a standard weight sheet)

Mullen

Tear

Breaking length (tensile strength)

Fold

Preparation for Chemical tests.

After the beater run was made the remainder of the pulp was spread out to air dry. This air dry pulp was then put through the fine plates of the Bauer in its dry form. This dry refining served to break up the dry clumps of pulp for more uniform sampling. The dry refined pulp was then put in a

glass stoppered bottle and allowed to come to equilibrium with respect to moisture content.

The air dry wood chips used for testing were put through the breaker plates of the Bauer refiner and then twice through the fine plates. This action gave a wood powder with a visably uniform particle size. The wood was not screened as it was thought that this might remove more of certain constituents than of others. This wood powder was also put into a glass stoppered bottle and let come to equilibrium.

After the wood and the pulp had come to equilibrium the moisture content of each was measured accurately.

The same tests which were run on the pulp were also run on the wood to assure consistency of results.

The following tests were run on the wood and on the pulp in accordance with the Institute Methods of The Institute of Paper Chemistry:

Lignin in Pulp (Institute Method 422)

Alpha-cellulose in Pulp (Institute Method 421)

And the following test in accordance with TAPPI:

Alcohol-Benzene Solubility of Wood

(TAPPI 54)

Lignin.

The wood sample used for the lignin determination was first extracted for three hours with the standard

alcohol-benzene mixture referred to in TAPPI standard T-6-m. This sample was then treated with 72 per cent sulfuric acid according to Institute Method 428.

The pulp sample to be tested for lignin was not extracted with the alcohol-benzene mixture before the acid treatment.

Alpha-cellulose.

The wood sample used for this test was also extracted with the alcohol-benzene mixture. The wood sample was then treated with acidified sodium chlorite as given in the procedure for unbleached pulp in Institute Method 421, the test for alpha-cellulose. The holo-cellulose obtained after treatment with the acidified sodium chlorite was then treated with 17.5 percent sodium hydroxide to obtain the alpha-cellulose.

The pulp sample was treated the same as the wood sample with the exception that it was not extracted with the alcohol-benzene mixture.

Hemi-cellulose.

The weight of the holo-cellulose from the alpha-cellulose determination was recorded for both wood and pulp. When the weight of the alpha-cellulose was subtracted from the weight of the holo-cellulose for the given sample the weight of hemi-cellulose was

obtained.

Alcohol-benzene extraction.

A sample of approximately two grams of wood was placed in a Soxhlet extractor and extracted with the alcohol-benzene mixture for a period of three hours. The extraction thimble and sample were then taken out of the extractor and the sample dried in an oven to obtain the oven dry weight. The weight of the extractives was then determined by difference.

The sample of pulp was extracted in exactly the same manner.

EXPERIMENTAL DATA.

The yield of the pulp was 84.7 per cent.

The chemical consumption was 3.6 per cent.

The values for the Canadian Standard freeness test have been corrected for temperature and consistency variation and the values for the physical strength tests have been corrected to the average basis weight of all the handsheets. This basis weight is equivalent to 45.8 pounds for a 25 x 40-500 ream. These physical strength values are given in Table I.

A representation of the change in test values with beating time may be seen in graphs one through five.

The actual experimental values for the various constituents in the wood and pulp are given in Table II. However, due to the failure of the acidified sodium chlorite to remove all of the lignin from the holo-cellulose, it was found necessary to determine the hemi-cellulose by difference. This, in turn, made necessary the determination of the hot water extractives. The percentage of hot water extractives both in the wood and in the pulp was taken from the literature. The corrected summary of values for the various wood constituents may be found in Table III.

A comparison of the various constituents in the wood and in the pulp can be seen in Chart I.

TABLE I

Test	Beating Time (minutes)					
	0	10	20	30	40	50
Freeneas (Canadian Standard)	503	406	333	240	175	121
Mullen (lbs. per sq. in.)	12.3	15.5	20.7	23.8	29.4	32.9
Tear (Grams)	37.9	31.3	33.8	31.0	31.0	34.3
Breaking length (meters)	3741	4753	6111	6459	7283	7910
Fold (F. I. T..)	3.4	8.6	17.5	37.8	69.9	100.7

TABLE II
EXPERIMENTAL VALUES FOR CONSTITUENTS IN
WOOD AND PULP

Fraction	In Wood	In Pulp	
	(lbs per 100 lbs original wood)	(lbs per 100 lbs original wood)	(lbs per 100 lbs pulp)
Alcohol-Benzene Extractives	3.4	0.5	0.6
Lignin (acid)	21.1	17.4	20.5
Holo-cellulose	90.3	71.7	84.7
Alpha-cellulose	50.3	49.3	58.2
Hemi-cellulose (by difference between holo- cellulose and alpha-cellulose)	40.0	22.4	26.5

TABLE III
CHANGE IN VARIOUS WOOD CONSTITUENTS ON PULPING
(corrected and accepted summary)

Fraction	In Wood	Loss on Pulping	In Pulp	
	(lbs per 100 lbs original wood)	(lbs per 100 lbs original wood)	(lbs per 100 lbs original wood)	(lbs per 100 lbs pulp)
Hot Water Extractives (from literature)	3.0	2.0	1.0	1.2
Alcohol-Benzene Extractives	3.4	2.9	0.5	0.6
Lignin (acid)	21.1	3.7	17.4	20.5
Alpha-cellulose (in holo-cellulose)	50.3	1.0	49.3	58.2
Hemi-cellulose (by difference)	22.2	5.6	16.6	19.6

DISCUSSION OF DATA.

The normal yield range of Cold Soda pulp is from 85 to 90 per cent. In view of this fact the experimental yield in this report is a little low. This may be accounted for by the fact that the pulp was refined to a freeness of 500 before the yield determination was made. It is thought that some of the pulp was lost through a shattering effect because it was somewhat brittle from the raw treatment. If some of the pulp shattered, the small fragments were lost through the screen during refining.

It will be noted that in the washing operation following the treatment, the pulp was washed only until the wash water looked clear. This could conceivably leave a little caustic in the pulp. However, it was presumed that this amount was negligible in comparison with the total amount that was removed.

The physical strength tests serve only to characterize the pulp. No attempt will be made in this paper to correlate the physical strength values with the chemical composition of the pulp. However, it should be noted that the tear values are quite erratic and do not serve as a good characterization of the pulp. Nevertheless, the curve of the tear

values with beating time show a slight downward trend. It should be noted, further, that the degree of variation of these points from the curve may be due to the small range on the scale which they are plotted. If the scale were to be shortened so that the coordinate line ran from zero to 50 rather than 30 to 40, these points would be less erratic.

The graphs of the other physical strength tests show a rise in the strength value with increase in beating time.

It will be seen from Tables II and III that the experimental values for the alcohol-benzene extractives and for the lignin have been accepted as they are reported. These values came out consistently and in literature.

In view of these lignin values the holo-cellulose values seem to be out of line. It is believed that the strength of the acidified sodium chlorite given in the Institute Method test was not great enough to remove all of the lignin present in the wood. This would give a high value for the holo-cellulose. It is also thought that this might apply to the pulp as well because very little of the lignin is removed in this process. However, more of the lignin is removed from the pulp by the acidified sodium chlorite

than from the wood.

While the sodium hydroxide used in the alpha-cellulose test is not sufficient to remove the residual lignin in the wood and in the pulp, the action of the acidified sodium chlorite in the holo-cellulose test chlorinated the lignin in such a manner that the sodium hydroxide could remove the chlorinated lignins along with the hemi-celluloses. This action occurred much the same as the alkaline extraction in pulp bleaching. The experimental values for the alpha-cellulose were accepted.

Due to the faulty values for the holo-cellulose, the hemi-cellulose values were unable to be determined from the difference between alpha-cellulose and holo-cellulose. Because of this discrepancy it was decided to determine the hemi-cellulose by the difference between the total weight of the wood or the chips, whatever the case may be, and the sum of the other constituents. However, to do this, the weight of the hot water extractives had to be known. These values were obtained from the literature. It will be noticed that the hemi-cellulose values thus obtained fell in a range compatible with those in the literature. Thus, these latter hemi-cellulose values were accepted also. From Table III it can be seen that the hemi-celluloses were removed to the greatest extent.

Appreciable amounts of lignin were removed and as well as some alcohol-benzene extractives and some hot water extractives. However, little of the alpha-cellulose was removed. This follows from the fact that alpha-cellulose is defined by its insolubility in 17.5 per cent sodium hydroxide. This is a rather concentrated caustic, whereas the strengths used in the Cold Soda Process are much more dilute.

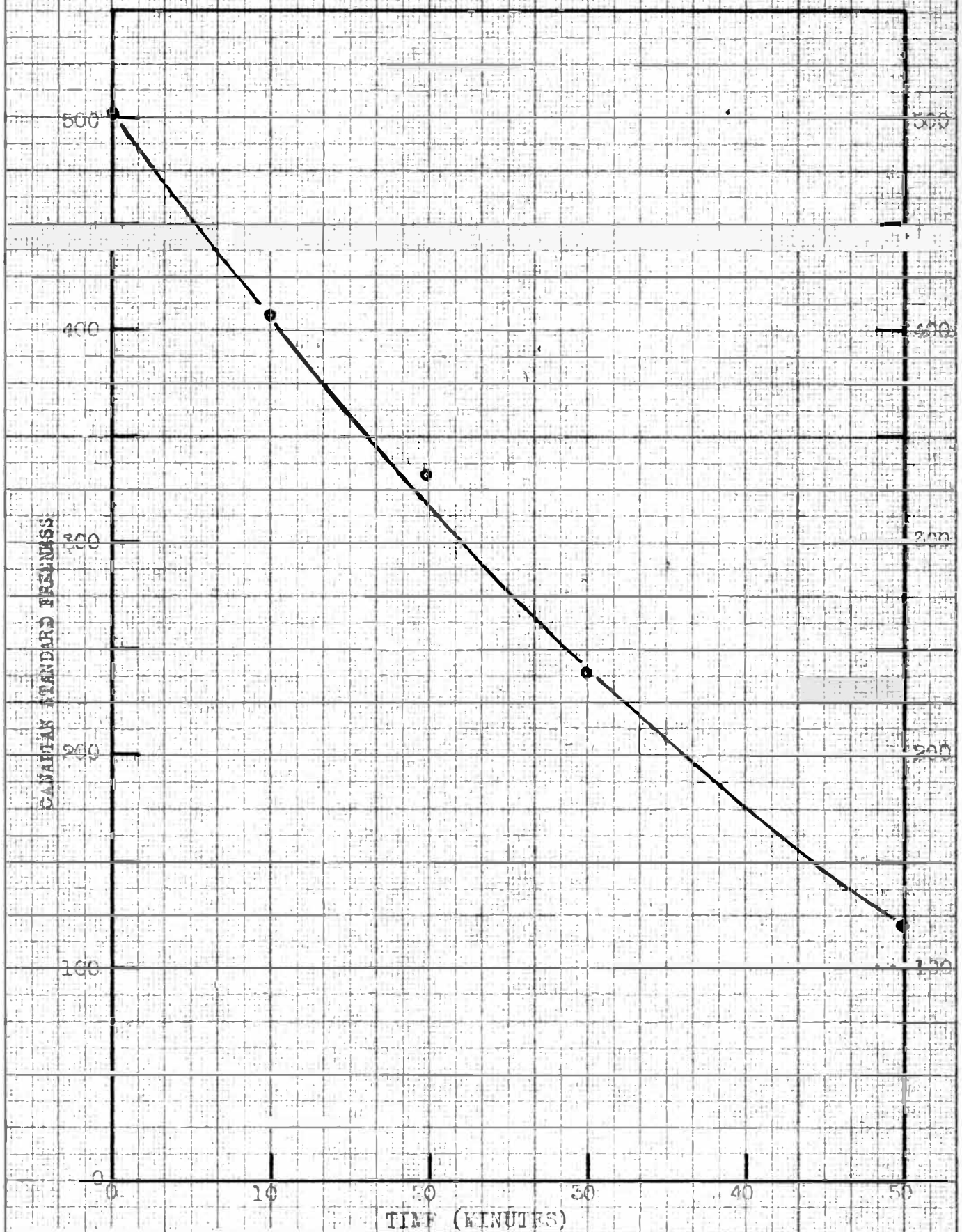
SUMMARY

The treatment of aspen chips for two hours at a room temperature of approximately 25 C. with a sodium hydroxide solution of a 53.1 grams per liter concentration was found to remove mostly hemi-celluloses. These hemi-celluloses were removed to the extent of 5.6 parts per 100.0 parts of original wood. Lignin was removed to the extent of 3.7 parts per 100.0, alcohol-benzene extractives were removed to the extent of 2.9 parts per 100.0, and hot water extractives were removed to the extent of 2.0 parts per 100.0.

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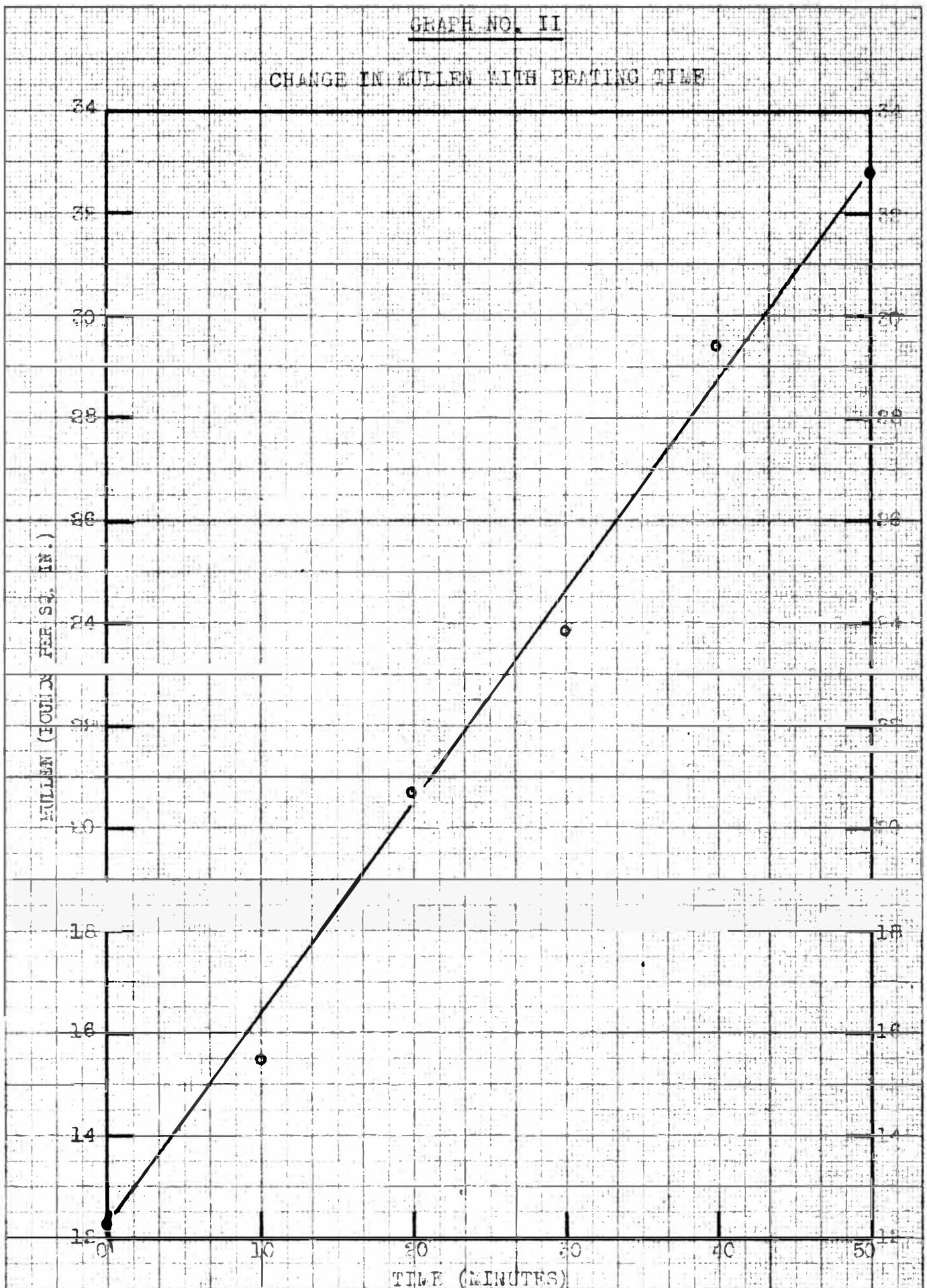
GEAPE NO. 1

CHANGE IN FIRMNESS WITH HEATING TIME



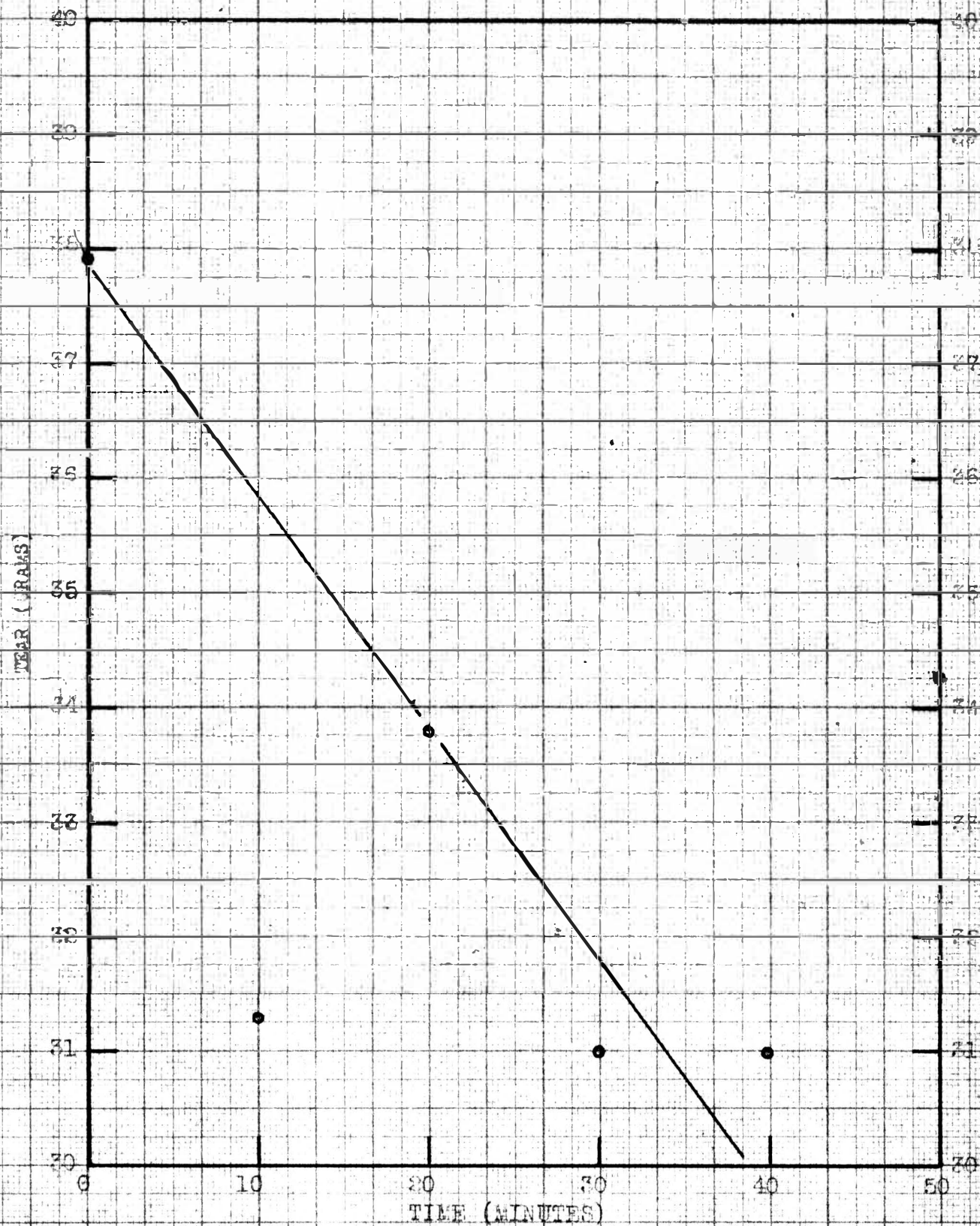
GRAPH NO. II

CHANGE IN MULLEN WITH BEATING TIME



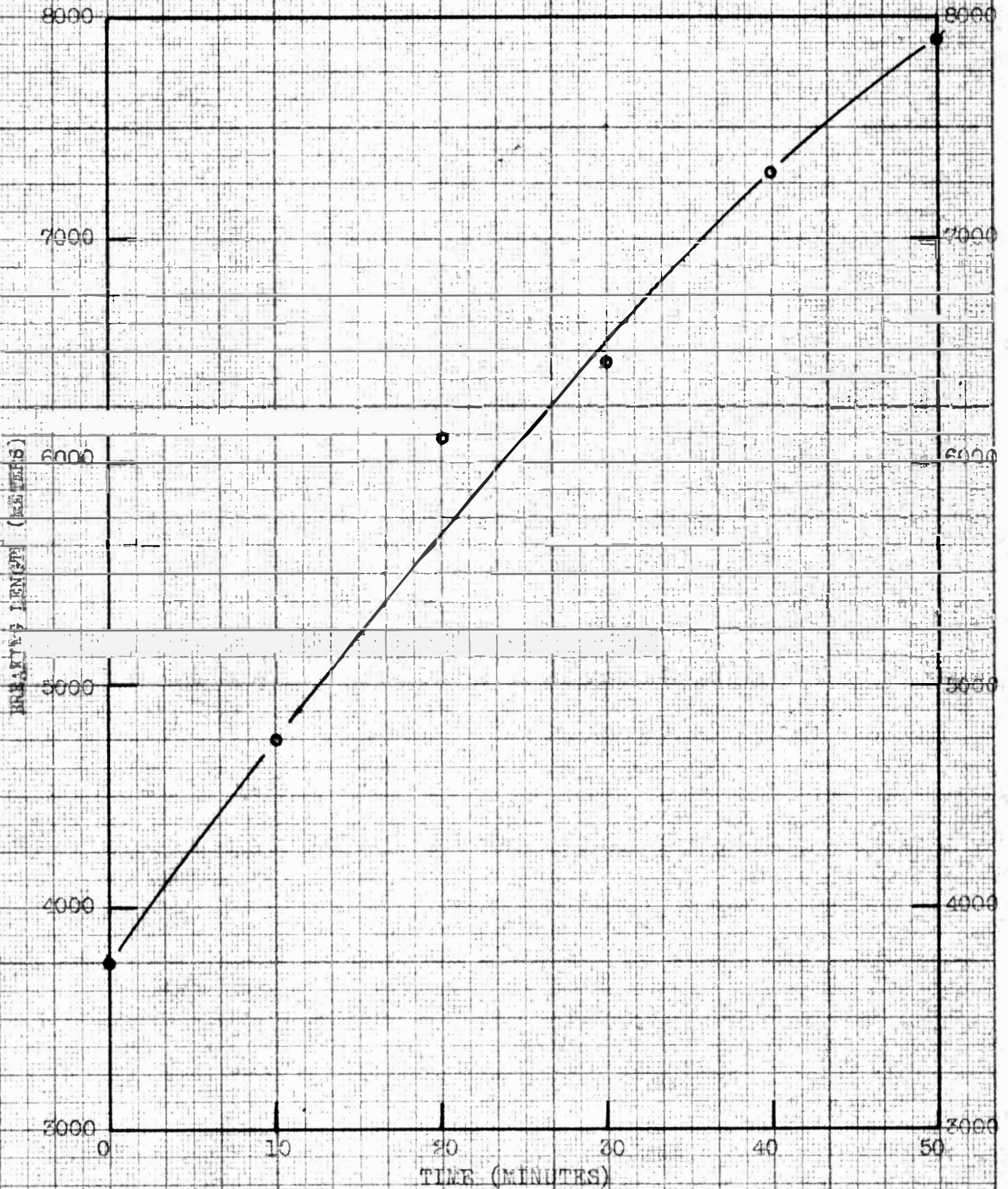
GRAPH NO. III

CHANGE IN TEAR WITH BEATING TIME



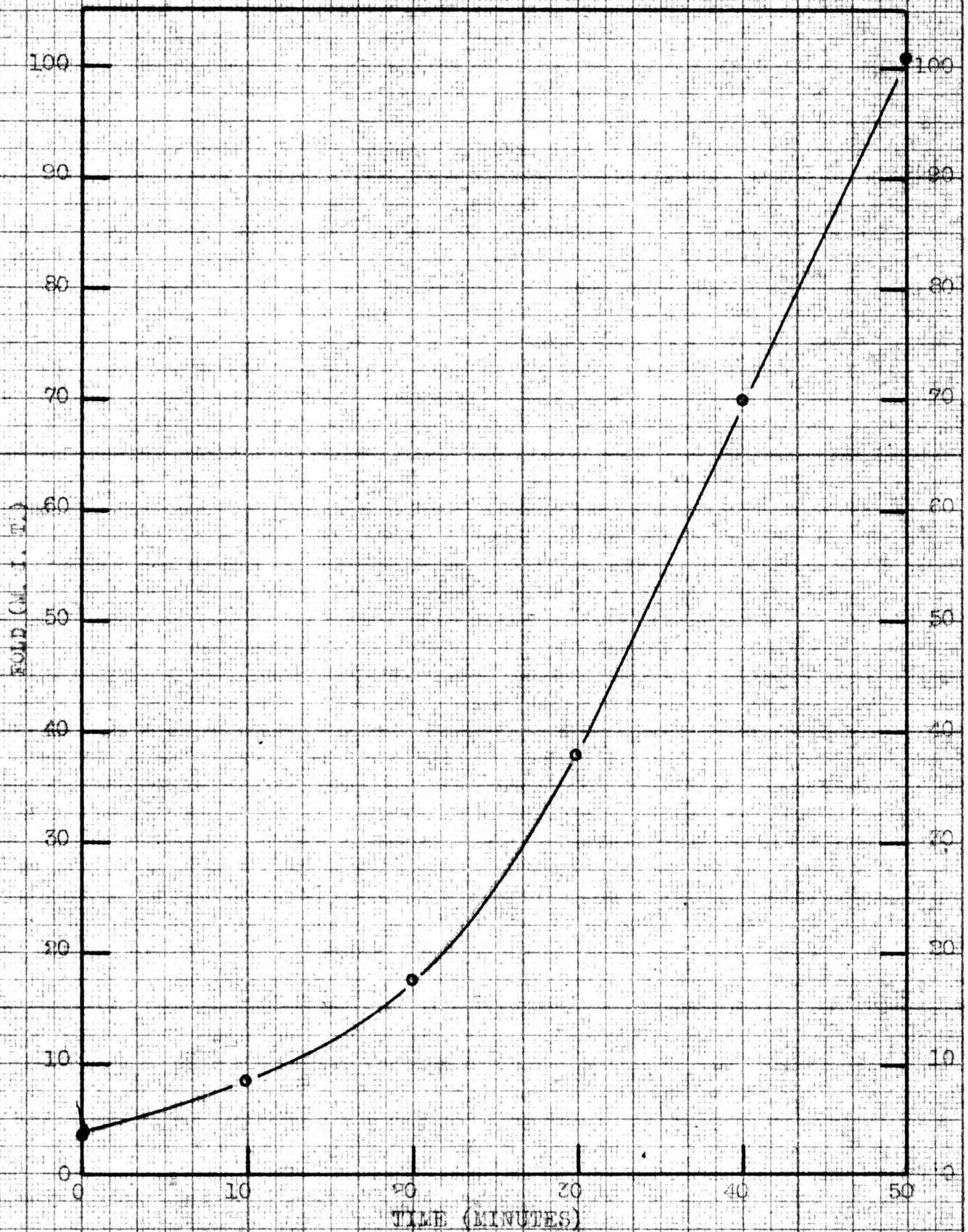
GRAPH NO. IV

CHANGE IN BREAKING LENGTH WITH BEATING TIME



GRAPH NO. V

CHANGE IN FOLD WITH BEATING TIME



CHANGE IN VARIOUS WOOD CONSTITUENTS ON PULPING

NOT WATER
EXTRACTIVES
(FROM LITERATURE)

ALCOHOL-~~SOLUBLE~~
EXTRACTIVES

LIGNIN (ACID)

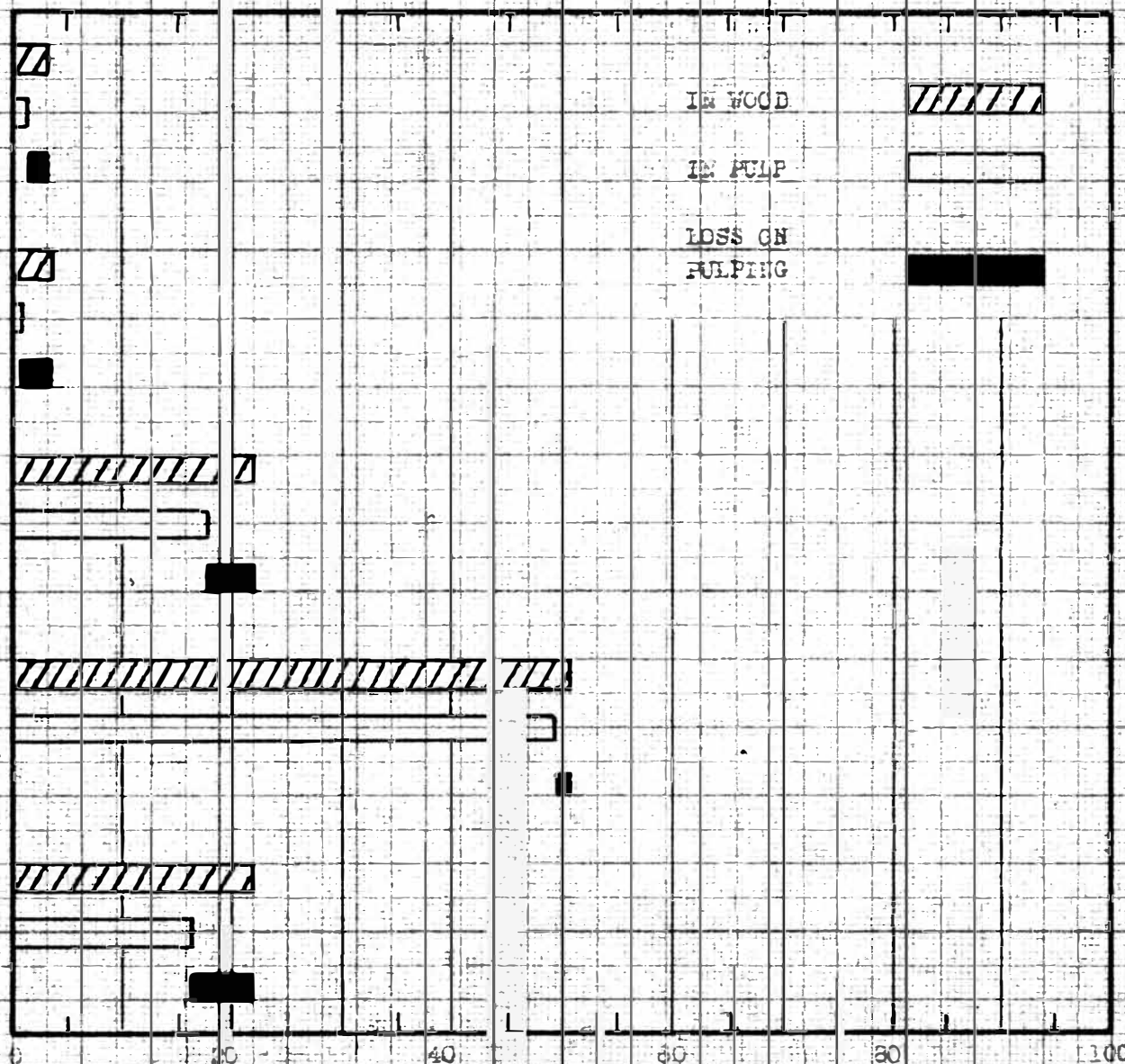
ALPHA-CELLULOSE
(IN HOLF-CELLULOSE)

HEMI-CELLULOSE
(BY DIFFERENCE)

IN WOOD

IN PULP

LOSS ON
PULPING



PARTS PER 100 PARTS ORIGINAL WOOD

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