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Variables Affecting Impregnation during Kraft Pulping

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VARIABLES AFFECTING IMPREGNATION

DURING KRAFT PULPING

by

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INTRODUCTION

The purpose of this report was twofold. First, a pulping aid was evaluated for its effects on pulp properties. Then, the pulping aid was used in conjunction with a reduction in the times to temperature for several cooks, to see if the pulping aid could be used to reduce the total cooking time for kraft cooks.

To gain some background as to how the pulping aid might affect the cook, variables affecting impregnation are presented and discussed.

PART 1: THEORETICAL

REASONS FOR WANTING IMPROVED IMPREGNATION

There can be numerous benefits from improving impregnation. Perfect impregnation can never be attained, but approaching perfect impregnation can have some of the following effects: (1)

Increased Yield: Perfect impregnation implies that all parts of a chip are cooked under the same conditions. In real situations, however, the cooking of the inside of the chip always lags behind the cooking of the outside. To cook the inside of the chip enough so that rejects are minimized, the outside must be overcooked. The degradation caused by this overcooking sometimes cannot be tolerated, so the middle of the chips may be undercooked, resulting in rejects. Both degradation of the outside of the chip and rejects cause the effective yield to be reduced. Improving the impregnation of the chips before and during a cook results in a more uniform cook, and the yield will improve due to reduction of the above problems.

Decreased Chemical Consumption: Increasing the uniformity of a cook by improving the impregnation will help cut chemical consumption. A more uniform cook implies that a greater percentage of the chemicals consumed are reacting with lignin, as opposed to reacting with cellulose and hemicelluloses. At the same kappa number, therefore, less chemical will be consumed, and the yield will be higher.

Reduction in Cooking Time: Improving impregnation reduces the lag between the cooking of the outside of the chip and the

cooking of the inside of the chip. As this lag time is reduced, so is the time needed to cook the inside of the chip. Therefore, a cook can be completed faster by improving impregnation.

Reduction in Cooking Temperature: If steam output is limited, the cooking temperature for a pulp can be reduced if impregnation is improved. All other factors being equal, improving impregnation should reduce energy requirements for pulp production.

PENETRATION AND DIFFUSION THEORY

At the onset of this section, it is important to distinguish between and understand the terms penetration, diffusion, and impregnation. Penetration is liquid flowing into a chip under driving forces such as pressure and surface tension. Diffusion is the movement of ions through the liquid into the chip due to a concentration gradient. Impregnation is a general term which describes a combination of these two operations. Good chip impregnation is a combination of getting liquid into the chip rapidly and then getting the cooking ions into the chip quickly. Liquor penetration is the important starting point for impregnating and cooking. (3) Liquor penetration starts slowly and then increases rapidly over 100 C. (4) But by the time lignin removal begins, (at around 130-140 degrees C), 75% of the original cooking chemicals in the liquor have been consumed by neutralization of carbohydrate degradation products. (5) Therefore, chemicals necessary for lignin removal must then be transported into the chip by dif-

fusion.

Liquor penetration into the chip takes place in softwoods mainly through the lumens of the tracheids. (6) Liquid flows from fiber to fiber through bordered pits, and liquor penetration is thus much quicker in the transverse direction than in the tangential or radial directions. However, at pulping conditions, the rate of diffusion is approximately equal in all three planes. (7) It is for this reason that chip thickness can becone a critical variable as far as overall penetration is concerned. (8)

Many variables can affect penetration and/or diffusion to give a subsequent effect on impregnation. Variables important to the impregnation process, along with some of the reasons that they affect the process, are listed in the following section.

VARIABLES AFFECTING IMPREGNATION

Chip Shape and Dimensions: As previously mentioned, chip shape and dimensions are important factors that must be considered when studying impregnation. The chip will be longest in the fiber direction, and an average chip is around 30 mm in length. (9) The chip must be thin in one of the other two directions, in order to facilitate rapid duffusion. (10) Due to the way that chips are made, this is usually in the radial direction.

As far as penetration is concerned, the thinner the chip, the better. However, once a chip is reduced to a certain

thickness, the strength of the subsequent pulp will suffer. (11) There is disagreement in industry as to what the best compromise thickness is. Researchers usually find that a thickness of 2-3 mm is not harmful to pulp strength. (12),(13) However, most mills cannot use a chip this thin and still get an adequate pulp. The reason for the disagreement is probably that researchers tend to use very uniform chips, while in industry this is not practical.

The last dimension to be considered is the width. As long long as the other two dimensions are set, the width is considered to be insignificant to impregnation. However, the width is important to other aspects of cooking, such as digester packing. The optimum width for this is about 30 mm. (14)

Wood Species: Softwoods and hardwoods are the two major classifications of wood species, and the large anatomical differences between them cause significant differences in the way that they are impregnated. (15) Although the anatomical differences do not cause major differences in the rate or mechanism of diffusion, the dissimilar biological structures result in different rates and modes of penetration. The hardwood vessels are more susceptable to liquid flow than are the tracheids of the softwoods. (16) This results in better penetration at the beginning of a cook for hardwoods.

Differences between species among the hardwoods and among the softwoods are not as substantial as those between these classes, but they can be important. For example, differences between oleoresin contnet of the softwoods can have an impor-

tant effect on penetration, since these substances block capillaries and thereby inhibit liquid flow. (17)

Wood Moisture: High chip moisture can be detrimental to impregnation in two ways. If there is a large relative amount of liquid in the chips, there is no room for the penetration of the liquor, and all impregnation must be done by diffusion. (18) Also, moisture swells wood, (19) constricting possible flow paths for penetrating liquor. Above 100 C, the effect of wood moisture is not very significant.

Air Content of Chips: A high air content in chips is detrimental to penetration early in a cook. (20) Like moisture, however, it becomes less important at high temperatures.

Temperature, Viscosity, and pH of Liquor: The temperature has a threefold effect on liquor impregnation. (21),(22) Raising the temperature lowers the viscosity, thus facilitating better penetration. However, raising the temperature causes the wood to swell, which inhibits penetration. A third effect of raising the temperature is to raise the rate of diffusion. Diffusion increases proportionally to an increase in absolute temperature.

The pH of cooking liquor also has an effect on the impregnation. Under pulping conditions, the high alkali content of the pH14 liquor causes considerable swelling of a chip, which of course inhibits penetration into the chips. (23)

Pressure: Pressure will cause an obvious increaes in impregnation. Increasing pressure forces the cooking liquor to penetrate the chip. (24)

All of the aforementioned variables have an effect on the impregnation of a chip by cooking liquor during a kraft cook. During the experimental part of this study, every effort was made to keep these variables constant. In this way, the true effects of the experimental parameters were sought.

PART 2: EXPERIMENTAL

OVERVIEW

Two studies were done for the experimental part of this thesis. The first experiment was a study of the effect of different amounts of a pulping aid on kraft cook. A second experiment was done to determine the effect of successively decreasing the normal time to temperature for three cooks. These cooks with the lower time to temperature were done at the medium level of addition of pulping aid. (Fig. 1)

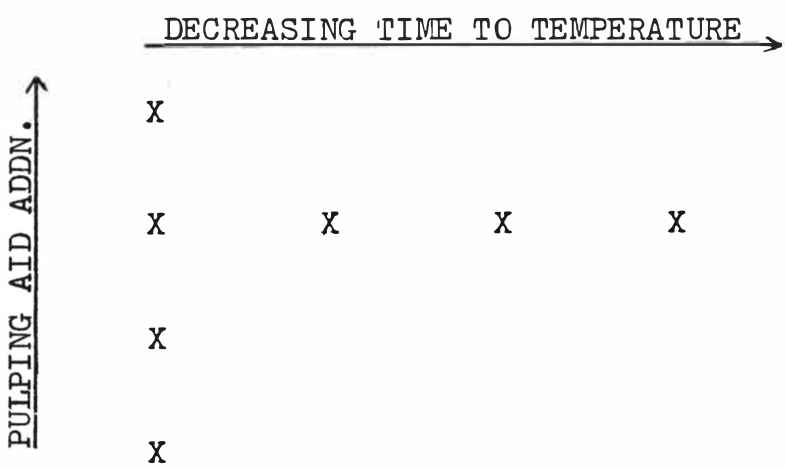


FIGURE 1

The purpose of running these two experiments together was to see, first of all, if the pulping aid was effective, and secondarily, if the pulping aid could possibly be used to reduce the necessary time to temperature for a cook while retaining acceptable pulp properties. Reducing the time to temperature would be beneficial to a mill that had a pulp production bottleneck at the digester.

PULPING PARAMETERS

Varying Time to Temperature: By changing the time taken to bring a cook to temperature, the amount of initial impregnation for the chips will be reduced. By taking the cook to temperature slowly, an ideal penetration can be approached. At the other extreme, cooking liquor at cooking temperature can be put on the chips, and the effects of this "worst" penetration can be studied. Two levels of intermediate time to temperatures were also studied.

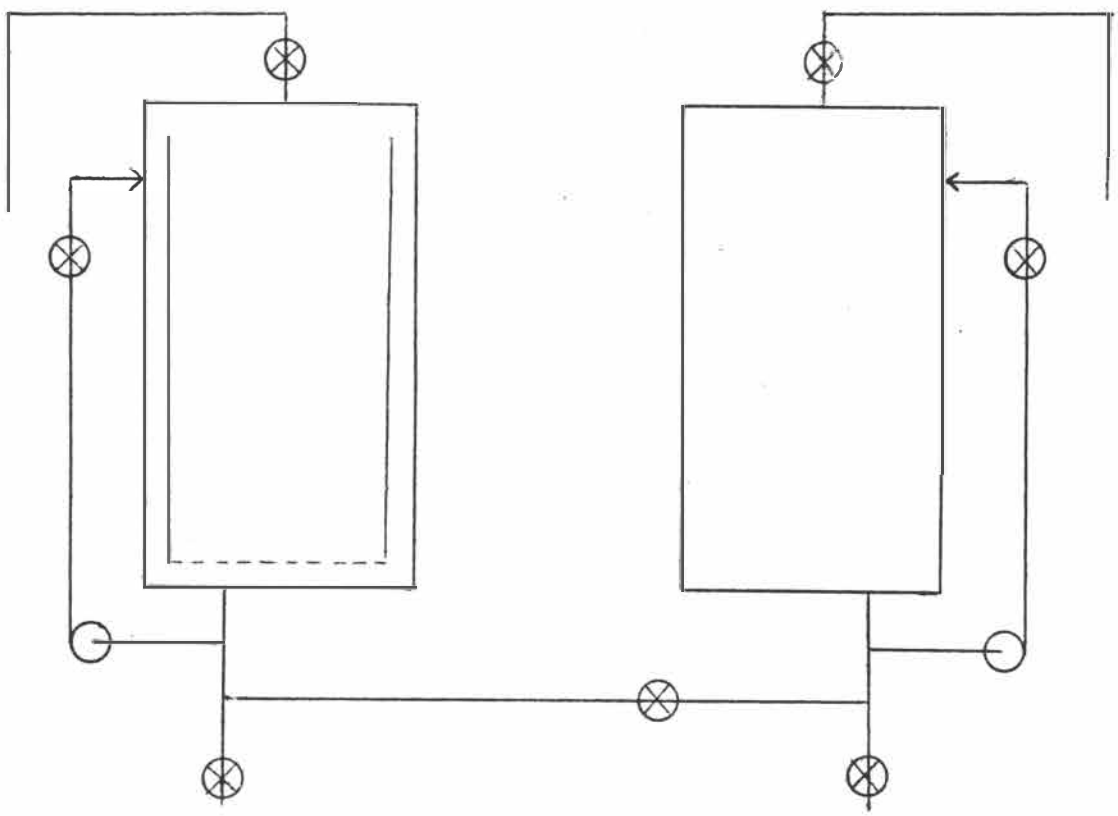
Pulping Aid Concentration: The second parameter that was varied was the concentration of a pulping aid. The chemical is an N,N dimethylamide of a fatty acid. Some of the possible benefits that can be obtained with this chemical are reduced shives, chemicals and/or time, and increased yield and brightness. To realize how these changes might come about, it is necessary to understand what this chemical does to the cooking liquor.

The chemical acts as a pulping aid by acting as an impregnation aid. N,N dimethlyamides of fatty acids are surface acting agents that are also good solvents. Because the chemical is a surfactant, it acts as a wetting agent for the capillary walls and facilitates liquid flow, thus increasing penetration. Since the chemical is a good solvent, it helps dissolve resinous materials from plugged capillaries and also helps dissolve and remove delignigication products. In this way, the chemical works both as a penetration and diffusion aid.

Another possible mechanism of the chemical that has not been reported but may be a factor is the action of the chemical on the liquor itself. Since it is a surfactant, it may help remove small bubbles from the liquor, which would help increase the rate of the initial penetration into the chip.

EXPERIMENTAL

All of the cooks were done in a loboratory model (M & K) digester. The digester consists of two pressure vessels, equiped with heaters, liquor recirculation pumps, and temperature and pressure guages. The vessels are connected by a valved line at the bottom. A cook is carried out in one of the vessels, and the vessel is relieved by blowing the liquor through the bottom line into the other cold vessel.



Once the cook is relieved, the vessel is opened and the basket of chips is removed. Water is run through the basket for three minutes, after which the chips are disintigrated for one minute at low speed in a one gallon Waring blender. Washing and screening were combined as water was continuously added with pulp to a slotted laboratory flat screen.

After screening, the pulp was refined to three different freenesses in a PFI mill. Each cook was refined for 3000, 6000, and 9000 revolution. Handsheets were then made in a Noble and Wood sheet mold, and were conditioned for one week at 50% humidity and 23 C.

Physical tests run on the handsheets were Mullen, Elmondorf tear, and Instron tensile. Strength values were expressed as factors, to eliminate possible basis weight variations. (See Tappi T220)

$$\text{Burst Factor:} \quad \frac{70.3 \times \text{burst in psi}}{\text{OD basis weight}}$$

$$\text{Tear Factor:} \quad \frac{100 \times \text{g. to tear one sheet}}{\text{OD basis weight}}$$

$$\text{Tensile Factor:} \quad \frac{200{,}000 \times \text{kg. tensile for 15 mm}}{\text{OD basis weight}}$$

All of the cooks done were done with kraft cooking liquor. The analysis of the liquor used is as follows:

Total Alkali....	93.9 g/l as Na_2O
Active Alkali...	87.0 " " "
NaOH............	68.3 " " "
Na_2S............	18.4 " " "
Na_2CO_3..........	6.4 " " "
Sulfidity.......	21.1%

For all of the cooks, the following constants were used:*

20 % Alkali on chips

5:1 liquor to wood ratio (higher than normal because the laboratory digester is not heated with direct steam.)

170 C

2.00 hrs. at temp.

61.5 ± 2.5 min to temp (first four cooks only)

Wood was 40 year old white pine from the eastern part of the Upper Peninsula of Michigan

Chip charge was 500g. AD, with about 200g. blocks

In general, all cooks went of with no problems. Some variations were noticed in the time to temperature for the first four cooks. The time to temperature ranged from 59 to 64 minutes. This is a significant difference that may have to be considered in the final evaluation of the data.

An important complication factor in this study was the fact that the yields were lower than desired. Liquor calculations were based on the total amount of wood in the digester. Some of this wood was in block form, and since the size of the block (1½ x 1½ x 1") did not permit total penetration, actual liquor ratios were higher than desired. However, although the yields were lower than expected, conditions were the same for all cooks, and relative differences, if any, should show up.

One other difficulty encountered with this study was that the cooks were run with a rather small amount of chips. The screening rejects did not show any pattern throughout any of

*Details in appendix

the studies, and it is felt that this is due to the uncertainty involved with using a small charge of chips.

STUDY #1 EFFECT OF PULPING AID

In this first experiment kraft cooks were done with the following additions of pulping aid:

	TARGET ADDITION	ML	ACTUAL
Cook #1	No Pulping Aid	0	0
Cook #2	$.1 \frac{kg.}{metric\ ton\ OD\ pulp}$	1.3	$.13 \frac{kg.}{ton}$
Cook #3	$.3 \frac{kg.}{metric\ ton\ OD\ pulp}$	3.8	$.35 \frac{kg.}{ton}$
Cook #4	$.5 \frac{kg.}{metric\ ton\ OD\ pulp}$	6.3	$.64 \frac{kg.}{ton}$

The pulping aid was first diluted 100:1 in a 1000 ml volumetric flask, and was added in the quantities shown above. The addition was calculated on an assumed 49% yield. Since the actual yields were lower, the actual levels of addition were somewhat higher than targeted.

The results of the cooks in the first study are presented in Table I . It can be seen that the pulping aid caused an initial increase in the yield, which subsequently dropped off as the level of addition increased. (Graph #1) The kappa number decreased in a very linear fasion with level of pulping aid. (Graph #2)

As may be expected, this combination of higher yield and lower kappa numbers resulted in generally higher strength values for the cooks with the pulping aid. Although the

"best" level of addition is subjective and not really clear cut, the medium level of addition seemed to give a good all around combinations of properties.

At 9000 revolutions in the PFI mill, the burst and tensile showed quite similar graphs when plotted against pulping aid concentration. (Graphs 3 & 4) A second similarity between these two tests is that for both, the highest strength was not reached until the highest level of refining. To see how these may be related, the freeness were consulted.

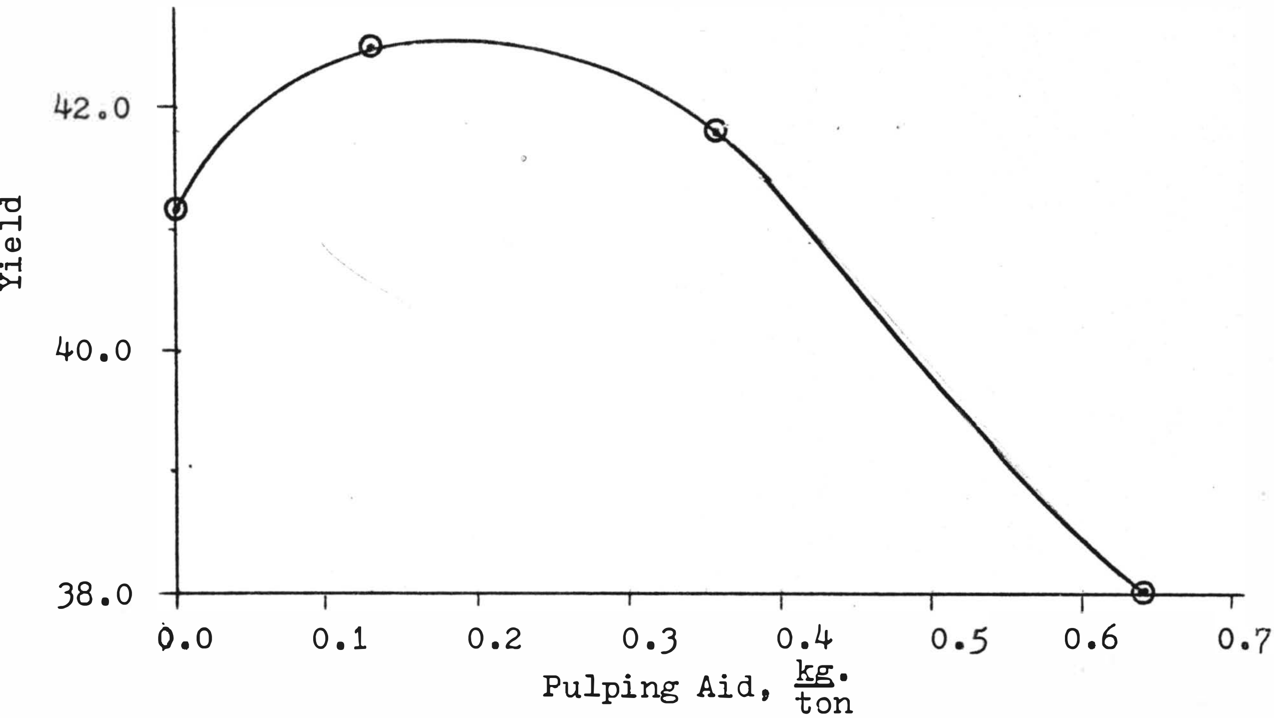
The freenesses for cooks two through four did not drop as fast or as far with successive refining as did the cook with no pulping aid. The freenesses for cook number three (medium level of addition) were generally the highest. This seems to verify what has already been indicated by the yields and kappa numbers: that the pulping aid causes a higher fraction of celluloses in the resultant in the pulp. (The freeness values must not be relied on too heavily, however, as there is a relatively large error involved with doing a freeness test.)

There is one other observation that is of interest with this data. The cooks at the low level of refining all had about the same tear strength. It seems that the reduction in kappa number caused by the pulping aid is being accomplished without a decrease in fiber length. This is born out by generally higher strength.

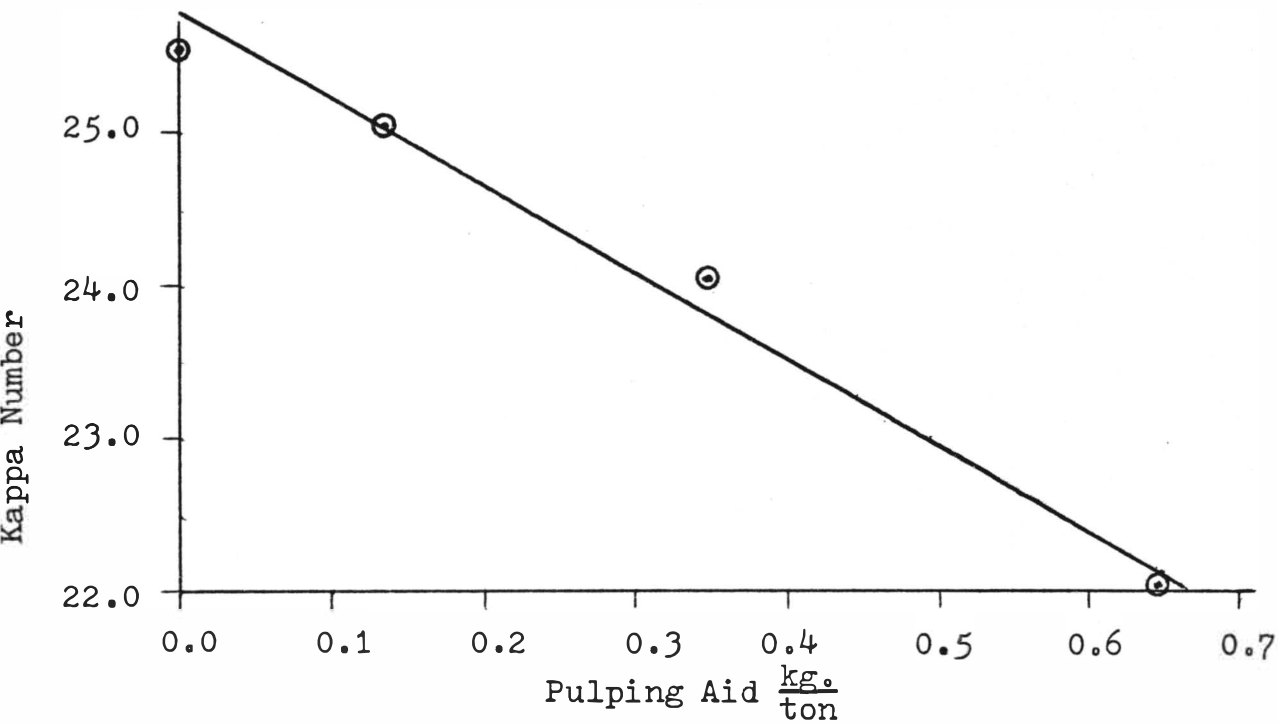
TABLE I

Evaluation		COOK 1	2	3	4
YIELD		41.1	42.5	41.8	38.0
PULPING AID ADDITION		NONE	LOW	MEDIUM	HIGH
KAPPA NUMBER		25.6	25.0	24.0	22.0
STRENGTH					
Burst Factor	3000	64.6	64.5	70.7	69.1
	6000	73.7	74.5	75.2	74.3
	9000	73.2	74.5	78.0	75.3
Tear Factor	3000	116	115	118	115
	6000	98.7	106	104	104
	9000	92.9	104	97.0	100
Tensile Factor	3000	4590	4370	4300	4640
	6000	4700	4810	4500	4990
	9000	4570	4670	5370	4950
Freeness	3000	615	626	629	615
	6000	400	447	402	409
	9000	266	291	315	319

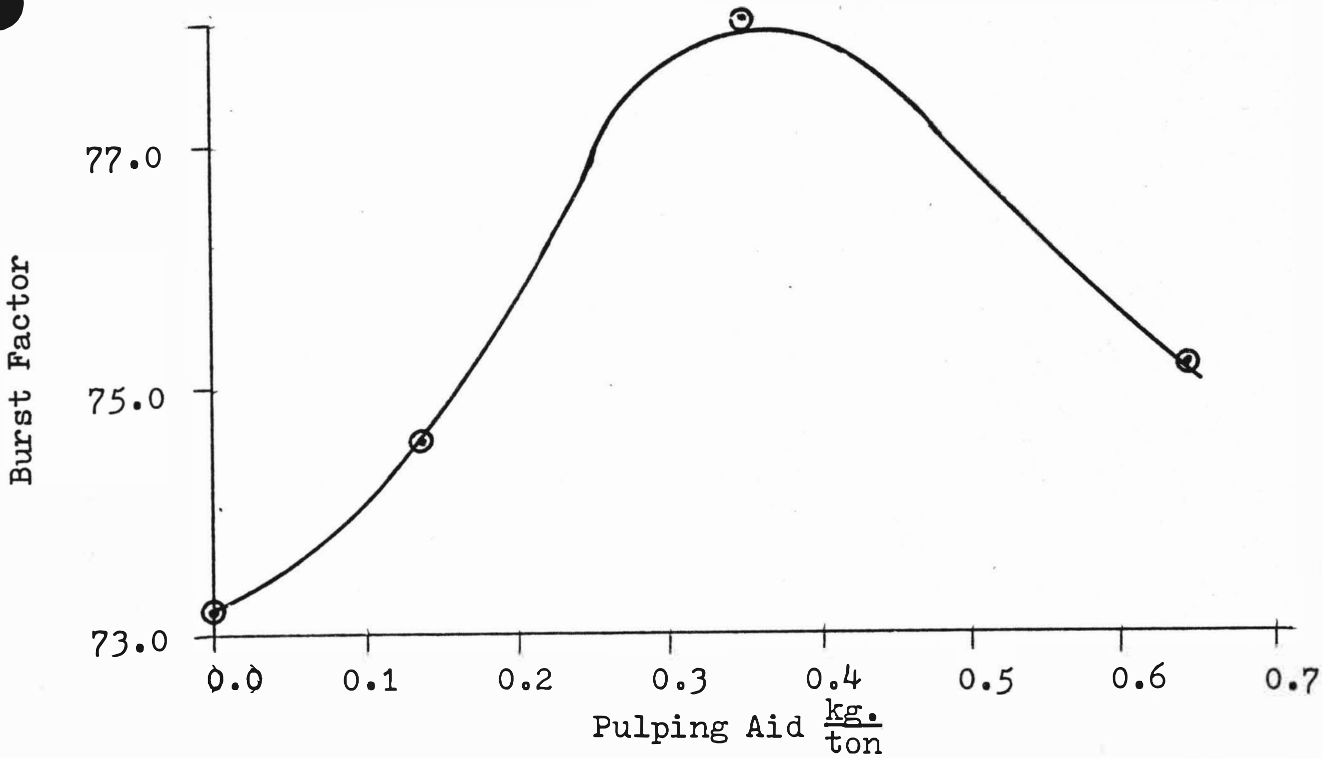
GRAPH #1: EFFECT OF PULPING AID ON YIELD



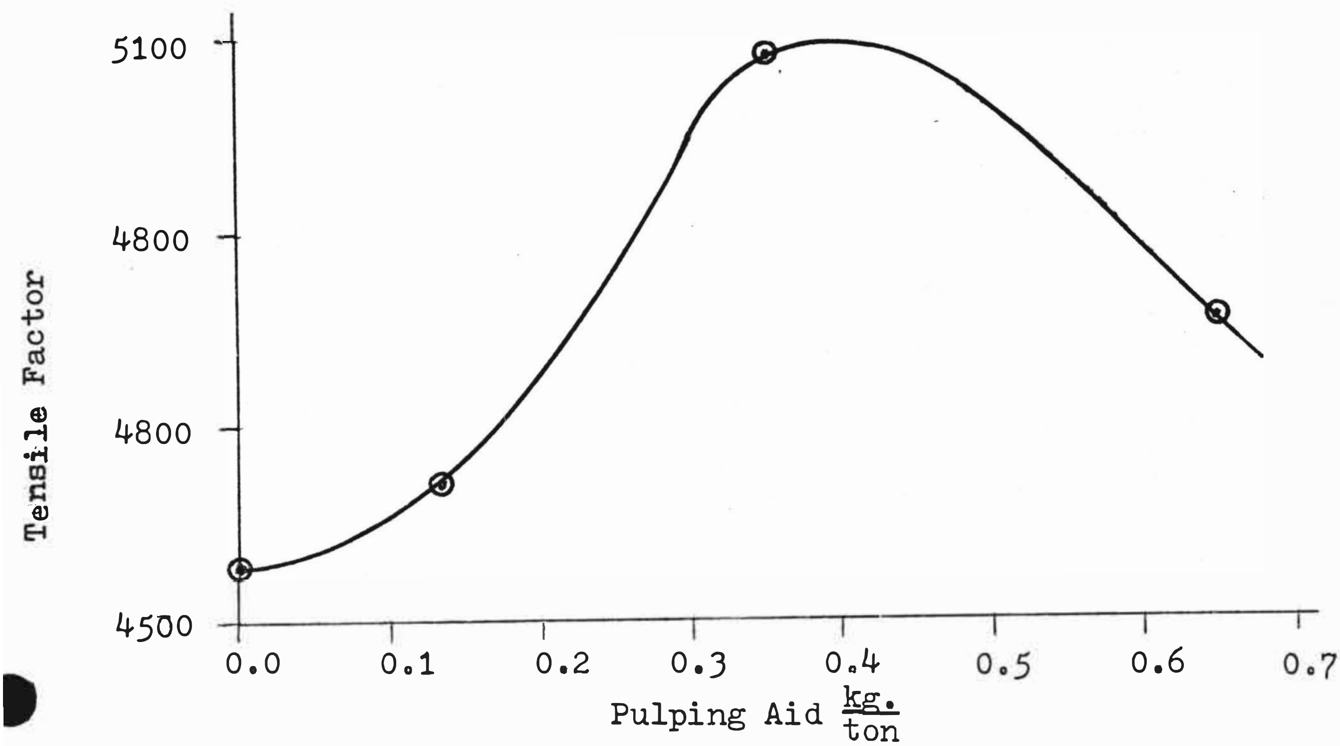
GRAPH #2: EFFECT OF PULPING AID ON KAPPA NUMBER



GRAPH #3: EFFECT OF PULPING AID ON BURST



GRAPH #4: EFFECT OF PULPING AID ON TENSILE



STUDY #2: EFFECT OF DECREASING TIME TO TEMPERATURE

The data for this study is presented in Table 2. An encouraging result is that all of the yields came out about the same. Thus the data can be evaluated at a nearly constant yield.

Not surprisingly, the kappa numbers were seen to rise as the time to temperature for the cooks was reduced. However, the first rise was not as steep as the subsequent rise. This initial pause before the following sharp increase resulted in pulp properties that were as good or better than were the results from cook #1. (Table 1) In other words, the cook with only 48 minutes time to temperature and a medium addition of pulping aid was as good or better than the cook with 62 minutes to temperature and no pulping aid.

The last two cooks with the even lower times to temperature were not as effective in holding acceptable pulp properties. They are substantially lower in both burst and tensile than the first two cooks in this study. The kappa number and strength of these last two cooks are quite similar, indicating that these properties may be approaching nearly constant values.

There are two possible reasons why cook #1 (62 min to temp, no aid) and cook #5 (48 min to temp, med. aid) are comparable. One is that the penetration was improved with the pulping aid. The other is that the cook would have been good anyways, with or without the aid. To speculate as to which may be the cause of this, the strength tests were again con-

T A B L E II

Evaluation		COOK 1	3	5	6	7
TIME TO TEMP		62	60	48	39	32
PULPING AID		NONE	MED	MED	MED	MED
YIELD		41.1	41.8	41.9	42.0	41.8
KAPPA NUMBER		25.6	24.0	26.2	32.0	31.7
STRENGTH						
Burst Factor	3000	64.6	70.7	76.4	66.0	69.3
	6000	73.7	75.2	79.1	67.1	67.3
	9000	73.2	78.0	76.5	63.9	62.9
Tear Factor	3000	116	118	103	105	109
	6000	98.7	104	102	96.3	96.2
	9000	92.9	97.0	88.4	92.5	92.0
Tensile Factor	3000	4590	4300	4940	4465	4110
	6000	4700	4500	5160	4590	4300
	9000	4570	5370	4910	4480	4100
Freeness	3000	615	629	594	577	604
	6000	400	442	390	410	434
	9000	266	315	234	277	297

sulted.

The strength tests for cook #5 (48 min to temp, med. aid) were higher than those for cook #3 (60 min to temp, med. aid). If the only variable change was decreasing time to temperature, it would seem logical that the strength would be lower for cook #5. This indicates that the pulping aid played a positive role in the cooking of the pulp.

BLOCKS

At the onset of this thesis it was determined that blocks might be useful to help evaluate some of the variables in the study. Uniform rectangles were cut from the same tree as the chips, and nine blocks ($1\frac{1}{2}$ x $1\frac{1}{2}$ x 1") were cooked with each cook.

The block were to be layered and analyzed for impregnation in each direction. The blocks would be cut to determine the impregnation in one direction only by removing edges after cooking to eliminate impregnation effects from the other two directions. They were to be analyzed for lignin and residual liquor.

When the blocks were cooked, they retained their shape well. However, it became apparent that they were not firm enough to microtome. Freezing the blocks caused them to warp. The blocks could be shaved by hand, but not to a satisfactory degree of accuracy. Therefore the decision was made, with some regret, not to use the blocks in the analysis of the variables in this thesis.

CONCLUSIONS

The pulping aid seemed to have a positive effect on pulp properties at low and medium levels of addition.

When the time to temperature for a cook was reduced from 62 to 48 minutes, the pulp properties did not suffer appreciably when the pulping aid was used at a medium level of addition.

Larger quantities of chips should have been cooked with each batch, to reduce errors from random variations in the chips.

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APPENDIX

COOK # 1 PULPING AID: None TIME TO TEMPERATURE: 64 min

GRAMS A. D. WOOD IN COOK: 712.6 GRAMS A. D. WOOD IN CHIPS: 500.1

GRAMS A. D. WOOD IN BLOCKS: 212.5 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 365.6

GRAMS O. D. WOOD IN COOK: 347.0 GRAMS O. D. WOOD IN CHIPS: 233.8

GRAMS O. D. WOOD IN BLOCKS: 113.2 ML LIQUOR IN COOK: 797.8

ML WATER ADDED: 571.6 ML PULPING AID ADDED: 0

CONCENTRATION OF PULPING AID IN COOK: 0

COOK # 2 PULPING AID: Low TIME TO TEMPERATURE: 59 min

GRAMS A. D. WOOD IN COOK: 693.4 GRAMS A. D. WOOD IN CHIPS: 499.7

GRAMS A. D. WOOD IN BLOCKS: 193.7 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 356.6

GRAMS O. D. WOOD IN COOK: 336.8 GRAMS O. D. WOOD IN CHIPS: 233.6

GRAMS O. D. WOOD IN BLOCKS: 103.2 ML LIQUOR IN COOK: 774.3

ML WATER ADDED: 553.1 ML PULPING AID ADDED: 1.3

CONCENTRATION OF PULPING AID IN COOK: .13 kg/ton

COOK # 3 PULPING AID: Med. TIME TO TEMPERATURE: 60 min

GRAMS A. D. WOOD IN COOK: 709.4 GRAMS A. D. WOOD IN CHIPS: 500.3

GRAMS A. D. WOOD IN BLOCKS: 209.1 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 364.1

GRAMS O. D. WOOD IN COOK: 345.3 GRAMS O. D. WOOD IN CHIPS: 233.9

GRAMS O. D. WOOD IN BLOCKS: 111.4 ML LIQUOR IN COOK: 790.1

ML WATER ADDED: 568.5 ML PULPING AID ADDED: 3.8

CONCENTRATION OF PULPING AID IN COOK: .35 kg/ton

COOK # 4 PULPING AID: High TIME TO TEMPERATURE: 63 min

GRAMS A. D. WOOD IN COOK: 695.2 GRAMS A. D. WOOD IN CHIPS: 499.7

GRAMS A. D. WOOD IN BLOCKS: 195.5 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 357.5

GRAMS O. D. WOOD IN COOK: 337.3 GRAMS O. D. WOOD IN CHIPS: 233.6

GRAMS O. D. WOOD IN BLOCKS: 104.1 ML LIQUOR IN COOK: 770.1

ML WATER ADDED: 554.6 ML PULPING AID ADDED: 6.3

CONCENTRATION OF PULPING AID IN COOK: .65 kg/ton

COOK # 5 PULPING AID: Med TIME TO TEMPERATURE: 48 min

GRAMS A. D. WOOD IN COOK: 700.4 GRAMS A. D. WOOD IN CHIPS: 500.6

GRAMS A. D. WOOD IN BLOCKS: 199.9 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 360.0

GRAMS O. D. WOOD IN COOK: 340.6 GRAMS O. D. WOOD IN CHIPS: 234.1

GRAMS O. D. WOOD IN BLOCKS: 106.5 ML LIQUOR IN COOK: 782.8

ML WATER ADDED: 559.7 ML PULPING AID ADDED: 3.8

CONCENTRATION OF PULPING AID IN COOK: .35 kg/ton

COOK # 6 PULPING AID: Med TIME TO TEMPERATURE: 39 min

GRAMS A. D. WOOD IN COOK: 710.4 GRAMS A. D. WOOD IN CHIPS: 499.2

GRAMS A. D. WOOD IN BLOCKS: 211.2 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 364.5

GRAMS O. D. WOOD IN COOK: 345.9 GRAMS O. D. WOOD IN CHIPS: 233.4

GRAMS O. D. WOOD IN BLOCKS: 112.5 ML LIQUOR IN COOK: 795.3

ML WATER ADDED: 569.7 ML PULPING AID ADDED: 3.8

CONCENTRATION OF PULPING AID IN COOK: .35 kg/ton

COOK # 7 PULPING AID: Med TIME TO TEMPERATURE: 32 min

GRAMS A. D. WOOD IN COOK: 704.4 GRAMS A. D. WOOD IN CHIPS: 500.0

GRAMS A. D. WOOD IN BLOCKS: 204.4 MOISTURE OF CHIPS: 53.25%

MOISTURE OF BLOCKS: 46.74% GRAMS TOTAL WATER IN WOOD: 361.7

GRAMS O. D. WOOD IN COOK: 342.7 GRAMS O. D. WOOD IN CHIPS: 233.8

GRAMS O. D. WOOD IN BLOCKS: 108.9 ML LIQUOR IN COOK: 787.9

ML WATER ADDED: 563.9 ML PULPING AID ADDED: 3.8

CONCENTRATION OF PULPING AID IN COOK: .35 kg/ton