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COURSE X - LI

M. I. T.

*Chem. Engg
Thesis*



PAPER PULP FROM CHINESE

BAMBOO BY THE SODA PROCESS.

Thesis submitted to the secretary of the faculty, Massachusetts Institute of Technology, as a partial fulfillment for the degree of Bachelor of Science in the Department of Chemical Engineering.

By:

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I. INTRODUCTION

With increase demand for paper and limited supply of wood the far-sighted Paper Manufacturers today are naturally in search of the new materials suitable for Paper making. Among the many possible substitutes of wood, bamboo stands out as the best qualified one. Its rapidity of growth and ease of cultivation can not be surpassed with any other kind of wood. Yet the fact that very few modern factories in the world now are using bamboo as a raw material for paper making may lead us to doubt, Whether bamboo will make a high quality paper as the other paper making woods.

Advantages
of bamboo
over the
woods.

Past investigator's on this subject have cleared out this doubt for us, and the concensus of opinions today is that bamboo yields high quality paper just as well as wood if not better. But the question *that* remains is whether the production of this high grade paper from bamboo is economically possible and whether the modern process of paper manufacture for woods can be applied to bamboo. Early workers in this field like Raithledge and Raitt firmly maintained the affirmative. The reason that bamboos are not much used in the modern factories today is probably due to the fact that the great paper producing and consu^ming country, United Sates, is not gifted with the growth of

^{agree}
Authorities.
on high
quality of
paper from
bamboo

bamboo and the conditions of the paper producing forests in this country do not yet warrant cultivation of this plant, and that countries like India, where the bamboo grows almost every where, are lacking in paper consuming powers. However, Bamboo seems destined to hold an increasingly important place as a raw material for paper making.

(a) Chinese Method for Making Paper from Bamboo

The use of bamboo as a raw material for paper, although not much practised in modern factories, is very common among the Chinese who prepare the plant for paper by the native process. The stems are cut into lengths, made into bundles and immersed into concrete pits, being weighed down and kept under water by heavy stones. After 3 months, they are removed, opened up and thoroughly washed. Next they are restacked in layers, each layer being well sprinkled with lime and water, holding potash salts in solution. After 2 months they are well settled. The fibrous mass is then washed to remove the lime, steamed for 15 days, when it is removed, thoroughly washed and again placed in concrete tanks. The mass is next reduced to fine pulp with wooden rakes and is then ready for conversion into paper. A quantity of the pulp is put into tough with cold water and mucilage. An oblong bamboo frame, the size of the desired sheet of paper, having a fine

Native
Chinese
method

mesh, is held at the two ends by a workman, and drawn endways and diagonally into the liquid contents, which are kept constantly stirred in trough. It is then gently raised to the surface and the film which has collected on the top is deposited as a sheet of moist paper when the frame is turned over. After the surplus water has drained away from the mass of moist sheets, the whole is submitted to pressure and it is then dried either in kilns or in the sun, according to the quality, the sun dried being its inferior. The species of bamboos used are several, but the most common is *Phyllostachys Hetroclea*.

From the above description, it is clear that the native Chinese process is the same as the lime process as is used in the modern factories in producing paper from rags, only the reaction is allowed to be carried on at ordinary temperature and hence is exceedingly slow. It should also be noted that native Chinese paper are not bleached nor ~~loaded~~ and naturally its ash content is very low. These paper ranges from pale yellow to pure white in color depending upon quality of the bamboo used.

This process, although workable and perhaps was the best when the Chinese first started to make paper centuries ago, is certainly very ^{un-}economical, in

the present day world. The working out of a modern process for its substitute in China is badly wanting today and it is with this need of the people in mind that the writer chose this subject as Thesis, although he is perfectly in aware of the fact that his Thesis will barely touch ^{one} every corner of the subject owing to such limited time. Aim underlying the Thesis

PAST INVESTIGATION ON MAKING BAMBOO

PAPER BY THE MODERN PROCESSES

The investigation of making paper from bamboo by the modern processes was started first by Thomas Routledge in 1875. Followed him, there are many others who worked for the Indian Government for the disposal of her rich forests of bamboo, especially in lower Burma where 20,000 square miles are covered with this plant. Notably among these workers is Mr. Wm. Raitt whose experience of more than 30 years in the investigations of this field has gained him the title as authority on bamboo as paper making material. He claims⁴ that not all species of bamboo (which amounted to about 500) are suitable for paper manufacturing and among the Indian species, only five are the best suitable.

Summary of
Wm. Raitt's
work.

⁴ These numbers indicate the number of article in the Bibliography.

He found difference in composition between the nodes and internodes* of the bamboo and suggested preliminary crushing before digestion as a means of eliminating this difficulty and also at the same time destroy the effect of capillary air in the stem of the bamboo causing it to float. Raitt claims that sulphite process is not fit to make bamboo pulp due to the formation of yellow coloration which is difficult to bleach. Soda process has no ill effect on bamboo, only the bleaching consumption of the subsequent pulp is still high. He favors the sulphate process because it decreases the bleach consumption decidedly while the sulphate used in making up the loss in recovery is a much cheaper raw material than NaOH which is used in the soda process.

Crushing of bamboo eliminates many difficulties

Sulphate process is considered best for bamboo.

Raitt's system of investigation consists in first analyzing the bamboo for its water extracts (which are mainly starch and starch secondary products.) pectose and lignin contents. By some method, which is not published in his books, he figured out the % NaOH necessary to react with 1% of each of the above three substances as found in the analysis. These values being:

% NaOH necessary to dissolve 1% of W.E.	=	.22
" " " " " Pectose	=	.32
" " " " " lignin	=	.66

Calculating the % NaOH necessary for bare digestion

From these values he figured the percentage of NaOH necessary for bare digestion of a particular specie of bamboo. Starting from this, and after ascertained

* The nodes of bamboo are the solid rings which intermittantly placed among the internodes, or hollow stem of the plant.

of the best temperature, pressure and duration of cooking, he increased the % of NaOH each time and found that increase of caustic causes decrease of yield which, on bleaching, takes up less bleach. Although he did not state whether the amount of NaOH used each time is the amount added or the amount consumed, but from the regularity of the increase in step of 2%, it can be inferred that they are on the amount added,

By making a series of runs of this sort, and calculate the unit ~~of~~ cost of each run*, the most economical condition can be obtained. For although increase of NaOH gives ~~increase~~ through digestion and consequently the pulp produced requires less bleach, but the cost of raw material and NaOH soon off set the saving from the bleach consumption. The best conditions can only be found by comparing the cost necessary to produce a unit of bleached pulp. Then after conditions, which Raitt found that will affect the quality of the pulp are temp. or pressure and duration of cooking. He found the best conditions for

Unit ^{cost} ~~increase~~
of obtaining
best cooking
conditions

$$* \text{ Unit cost} = \frac{(100\% \text{ Bamboo})A + (\% \text{ NaOH})B + (\% \text{ B. Powder})C}{\text{Bleach Yield}}$$

A, B and C are cost per unit of the three substances respectively.

these are: cook for 1 hour at 177°C or 120# and another 4 hours at 162°C or 80#. He claims that at 177°C, the destruction of ^{fibres} ~~it~~ by NaOH is serious, but it helps to overcome penetration resistance and improve color, also ~~it~~ shortens the time of digestion from 6½ hours to 5 hours. He found that these cooking conditions are applicable to nearly ^{all} bamboos except the amount of NaOH consumed varies ~~with~~ the different species according to their compositions.

With the sulphate process, the time, temperature and pressure remain the same, while instead of increasing the % of NaOH every time during the series of runs to find out the best amount of alkali, he increased the % Na₂S keeping the % NaOH the same, which equals to the amount necessary for bare digestion. By similar process of comparing the unit cost, the most economical amount of the ~~total~~ ⁱⁿ alkali that should be added can be found. The difference results between the sulphate process ~~and~~ the soda process lies, as stated before, in the reduction of bleach consumption and a little increase ^{of yield.} For example, ~~with~~ Bambusa Polynorpha, the following table shows the results by the ~~time~~ process.*

The sulphate process reduces bleach consumption and increase yield.

*It should be noted that in both digestions, the starch content is first extracted ~~with~~ water.

	Soda	Sulphate
NaOH	19.5 %	20.5 % *
Unit Yield	45.5 %	46 %
B.P.	11.4 %	7.82%
B. Yield	43 %	44%
Unit Cost	6.82	5.85

All Percentages are on basis of raw material

* NaOH & Na₂S calc on NaOH

Other workers on bamboo are Messrs. Sindell, Pearson and Richmond who contributed to the literature on this subject while still quite a few others hold patents regarding pulping of bamboo. For detailed discussion of these past investigators, refer to the appendix.

(c) SCOPE AND PURPOSE OF THE PRESENT INVESTIGATION

Although Raitt's work seems to be very systematic and trustworthy but other workers of the same field do not entirely agree with him. For instance, Schmidt⁷ found sulphite process is suitable for the reduction of bamboo, while Richmond¹⁰ found that the soda process is very successful for Philippine bamboo. Realizing the difference of species of bamboo that grow in different countries, it is not surprising that one process which applicable to one species, may be useless to another. Therefore the purpose

Authorities do not agree on the best process

of the present investigation is to study particularly on Chinese Bamboo which has so far not been investigated, and to find out whether it is suitable for paper making. Present investigation deals only with Chinese bamboo

With the many ^{angles} that such investigations could be directed, it is a little perplexing as to which route should be followed. Of course, it is plain that the nature of the investigation is very evolved and a thorough study may take years of work. However, to make a start, it was thought to be well by starting on the soda process and then continue with the sulphite process, ^{if} time permits, to make some comparison. The first ~~thought~~ ^{thought} of digesting the bamboo with all the four processes, namely, sulphite, soda, sulphate and lime, was ~~omitted~~ ^{abandoned} owing to the fact that ~~several~~ apparatus had to be installed for the different methods of treatment and time is apparently lacking. Having decided on the Soda Process, the writer thought it will be well to follow Raitt's procedure since his system sounds reasonable and see whether the results obtained check up with his observations. Before going on to the detailed discussion of the experimental work of this Thesis, it is certainly not out of place to state in brief the principles of the Soda Process and its varying factors as applied to the manufacture of paper from wood. Select soda process as starting point.

(a) DISCUSSION OF THE SODA PROCESS

The soda process, as it is known today, may be considered as a modification of the old Watt and Burgess process, first practised in 1853 in producing chemical pulp from wood. It originally consisted in digesting suitably prepared wood in a larger boiler with a strong solution of caustic soda under a pressure of about 90 lbs. per square inch for 10 or 12 hours. The wood was then washed to remove the alkali and treated with chlorine gas or an oxygenous compound of chlorine. The partially digested wood was then washed to free it from the hydrochloric acid formed and again treated with a small quantity of caustic soda solution. The pulp produced was then washed, bleached and beaten in a beater Engine, after which it was ready for the paper machine.

The old
Watt and
Burgess
Process

In the modern factory practise the bark is first taken off from the wood, which is then chipped and charged to the digester. The digester is either rotary or stationary, but the present tendency is towards stationary, vertical, cylindrical digestors heated by live steam which enters at the bottom of the digester in such a manner as to carry the cooking liquor through a pipe to the top of the vessel and spray it over the chips to insure good circulation. The chips and cooling liquors are charged through the main hole at the top of the digester, the bottom of which is provided

Modern
factory
practice

with a "blow off" pipe and valve for discharging the pulp after ~~the~~ cooking is complete. As soon as the charging of the chips and caustic soda cooking liquors is complete, steam is turned into the digester until a certain cooking pressure or temperature is reached. This temperature varies at different mills, but one corresponding to 110 pounds steam pressure per square inch is the most common.

The cooked pulp, after it is blown out from the digester is drained from the black liquor and washed thoroughly with hot water. The black liquor which contains the great part of alkali cooking chemicals are run to evaporators, concentrated and later calcined in furnaces. The burned ash, containing Na_2CO_3 is leached with water. The resulting solution is treated with quicklime (CaO) which change the carbonate to NaOH .

Recovery
of alkali
from black
liquor

Efficiency of recovery in modern plants is from 88 to 92%. The pulp, after washing, is screened to remove uncooked portions, bleached with bleaching powder and again washed.

Although the process of cooking wood with caustic soda to form paper pulp sounds very simple, yet the best cooking conditions are not at all easy to determine and the effect of varying the conditions on the pulp produced is quite considerable. For example, investigation on varying certain cooking conditions in the soda process led Beveridge²⁰ to draw the following conclusions:

1. Increase of pressure resulted in diminuation of yield, the quantity of pulp obtained being reduced considerably.

Beveridge's
conclusions

2. Excess of caustic soda caused rapid diminuation of yield of cellulose.

3. Gradual exhaustion of the caustic soda by prolonged digestion prevented such serious diminuation of yield.

Aside from the character of the wood, the principal cooking conditions affecting yields and properties of pulps, consumption of cooking chemicals, and the general efficiency and costs of cooking operations are:¹⁹

Cooking
conditions

1. Preliminary treatments
2. Character of cooking apparatus, especially the manner of heating and kind of mechanical agitator.
3. Proportions of the charges.- the amounts of wood and chemicals and the amount of water in digester from softwood and steam.
4. Character of cooking liquors when charged such as causticity, initial temperature, impurity and concentration.
5. Duration of cooking treatment
6. Pressures and temperatures.
7. Manner of admitting steam, "relieving" and "blowing" the digester.

The many factors are more or less inter-dependent, and any change in one results in unavoidable changes in others. The more fundamental of these factors are:-

1. Amount of caustic soda charged per pound of wood.
2. Duration of working at maximum temperature.
3. Maximum temperature and pressure of cooking
4. Initial concentration of a cooking chemicals.

Four fundamental factors

From the investigation made by the Forest Service of the United States Department of Agriculture on Aspen the following conclusions were obtained.

Effect of varying these factors

1. The varying of the four fundamental cooking conditions influence the yield and properties of pulp by influencing the severity of the cooking reactions.

2. Severity of cooking is an effect mainly of the amount of caustic soda consumed per unit of wood.

Increasing the amount or concentration of the chemicals or the pressure of cooking produces a quicker reaction, while increasing the duration results in a more complete reaction.

3. Great severity of cooking is accompanied by a decrease in yield of crude pulp and usually of screened pulp.

4. The properties of the pulp which are influenced by greater severity of cooking are as follows:

Properties of pulp influenced by severity of cooking

a. Shives are decreased in number or eliminated.

b. Bleaching is ~~rendered~~ more easily and the loss on bleaching becomes less.

c. The strength may either decrease or increase depending upon which cooking condition is varied and the degree of variation.

d. The color of the unbleached pulp becomes lighter within certain limits, beyond which, under certain conditions, becomes darker.

Viewing from the above, while the amount of bleach required decreases with increasing severity of cooking, a point is soon reached where the decrease in bleach required is not commensurate with the decrease in yields and increase of caustic consumption. Therefore it is clear that in ~~work~~ on the soda process, the best quality of paper pulp and the amount yield can only be obtained by experimental determination ~~by~~ varying the cooking conditions, and it is also apparent that the ~~best~~ cooking conditions for one kind of wood are not good for the other kinds of woods.

(e) OUTLINE OF THE PRESENT EXPERIMENTAL WORK

From the knowledge gained from ~~the~~ past investigations on bamboo and on Soda Process, and realizing the limited amount of the time the writer had, the following schedule was planned and carried out.

The species of bamboo selected is first analyzed by the method adopted by Wm. Raitt and from the percentage of water extract, pectose and lignin found by analysis, it was expected to be able to calculate the % NaOH necessary for bare digestion by using Raitt's figures of the amount of NaOH increasing for dissolving each of the three constituents. Cooks was then started. By keeping the other factors constant, the amount of NaOH put in was increased by 2% for each cooking. After the different cooks were made, a sample of unbleached pulp from each cooking was then bleached under fixed conditions and amount of bleach consumption determined. The fibers from the pulp were then examined under the microscope with microphoto taken of the fibers, and the dimension of the fibre measured. Of course, before digestion could begin, the setting of apparatus, crushing of bamboo, and making of different solutions used were made. In brief, the following is a outline of the experimental work of the Thesis. Detailed description of the procedure and the discussion of results will appear later in this report.

Outline of Experimental Work.

1. Selection of species of bamboo.
2. Crushing of bamboo.
3. Setting up of apparatus.
4. Analysis of bamboo.
5. Determination of cellulose by digestion with excess of caustic under atmospheric pressure.
6. Digestions of bamboo with varying amount of caustic in different cookers.
7. Bleaching of the unbleached pulps.
8. Microscopic examination of fibres.

II. DESCRIPTION OF THE EXPERIMENTAL WORKS

I. PRELIMINARY WORK

(a) Selection of Raw Materials.

Since the purpose of this Thesis is to find out the characteristics of Chinese Bamboo towards digestion with caustic soda, and since no Chinese Bamboos were available in this country, an order was sent to China for some Chinese Bamboo. Samples of six species were received, totally about 50 lbs of raw material.

Order
Bamboo from
China

The species are markedly different in size. The biggest has a diameter of 4" while the smallest species have diameters of only .5 inches. The first thought was to crush all the bamboos together and treat them as a mixture, but the second thought revealed the fact that judging from the sizes, different species must have different compositions and they will not behave the same under similar conditions of cooking. The small ones will undoubtedly be attached first by the alkali at high temperatures, and they will be very much overcooked when the big ones are dissolved. Thus, cooking with different species will not be uniform and yields will naturally be lower. Having abandoned the thought of mix cooking, the attention was turned to the selection of a species that will be a good representative of Chinese Bamboo. With some consideration, one

Species are
different
in size

Different
species can-
not be
cooked to-
gether

specie was decided on for two reasons. First it has the largest quantity and second, it has a size midway between the ~~two~~ extremes. Its diameter varies from 2 to 2.5 inches. The botanical name of this particular specie was not known although in Chinese it is called (淡竹). With the help of experts in the Arnold Arboretum, it is thought probable that the specie is *Phallistachys puberula*. However, its identity is not certain because there are no leaves or roots attached to the sample to make sure the identification. As learned from communications from China, this specie is common and it grows mostly in the Chekiang Province.

Specie
chosen is
probably
Phallista-
chys puber-
ula

The Bamboo came in short portions cut from the whole culm. With this particular specie, the surface of the stem or culm is yellowish in color without the marked coating of silicious surface cells which is common in other species/ The low ash content as found by the subsequent analyses proves the point.

(b) Crushing of Bamboo.

Owing to the close compact composition of cells, bamboo stem is very hard, and hence the problem of crushing is not a very easy one. The different crushing machines in the Mining department of the Institute were all tried but none of them was successful because these machines were not designed for crushing

fibrous materials like bamboo. The best results obtained was by the roller crushers but even these only flattened the bamboo out, and no real crushing was taken place.

Suggestion was then ~~came~~⁺ in trying the dropping ~~of the~~ electric hammer as used in the Institute Foundry laboratory. Trial was immediately made and the results were entirely satisfactory. The constant pounding of the hammer breaks up the fibrous structure of the bamboo very completely, even with the nodes. The crushed bamboo was then cut in convenient lengths by shears, and the bamboo is then ready for digestion.

Electric hammer in Foundry solves the crushing problem.

(c) Setting of Apparatus.

The principal apparatus used in this Theses is the digester, so the setting of apparatus reduces itself to the making of this digester. In designing and making of a digester, four factors necessary came into consideration all of which should be well taken care of. The first is the kind and shape of the vessel. Since the digestion has to be carried out under high pressures, the vessel must be strong enough to withstand that pressure. The second is the method of heating - it may be heated by direct steam, steam jacket or direct fire. Third is the means of stirring. It may be directly stirred by stirring devices, or by circulation of cooking liquors of steam. The fourth

Four factors in digester designing

is the devices for loading of raw material and cooking liquor and for relieving and blowing out of the cooked pulps.

Realizing that the writer is working alone, the construction of a large size digester is not advisable, not only because it takes time to build but also because it is difficult to be handled alone. So a small size digester was built with the necessary factors taken care of as completely as possible.

Built a
small size
digester

Following is an account of the construction of this digester together with the stirring devices used.

With ~~the aid~~ of the accompanying photos, the reader will no doubt be able to understand ^{the construction and operation of} this digester.

A piece of cast iron tube, 26 inches long and $5\frac{1}{2}$ inches inner diameter, was threaded on both ends and a flange was screwed on at each end. A blank flange, after surfaced with the flange on the tube, was placed on the latter and both fastened together by bolts. This was done to both ends. The whole thing represents the body of the digester. On one of the blank flanges, a hole was drilled and a thermometer well fixed in, while on the other, a larger hole was drilled, a pipe screwed in. To this pipe, was attached a pressure gage through a T connecting piece and a gate-valve at the end. Knowing that hot caustic soda will attack brass, all auxiliary equipments

attached to the digester ^{are} made of iron or steel. Thus the thermometer well is nothing but a steel tubing with a blind cap; the valve is of iron and the pressure gage is made ~~for~~ Ammonia and ~~is~~ of steel. The reason of putting the gage directly on the line where hot caustic soda can reach it is simply because that the digester has to be rolled by the rotary device as a source of stirring and hence all auxiliaries to the digester should be attached to it directly, and be rotated with it. It was attempted to put a cork between the pipe and gage so that the gage will not be constantly attacked by hot caustic but no steel cork could be obtained from the maker. However, although the gage is hot during digestion, no ill effect was shown by the gage during the digestions only it was plugged up once. The gate valve is a safety device - it serves to reduce the pressure quickly, should it be too excessive, and also to relieve the pressure at the end. At the beginning of each digestion, the valve is closed, after the steam drives out the air in the digester. This gives a better correspondence of pressure and temperature in-side the digester. The loading and discharging of the digester is done by opening one of the flanges.

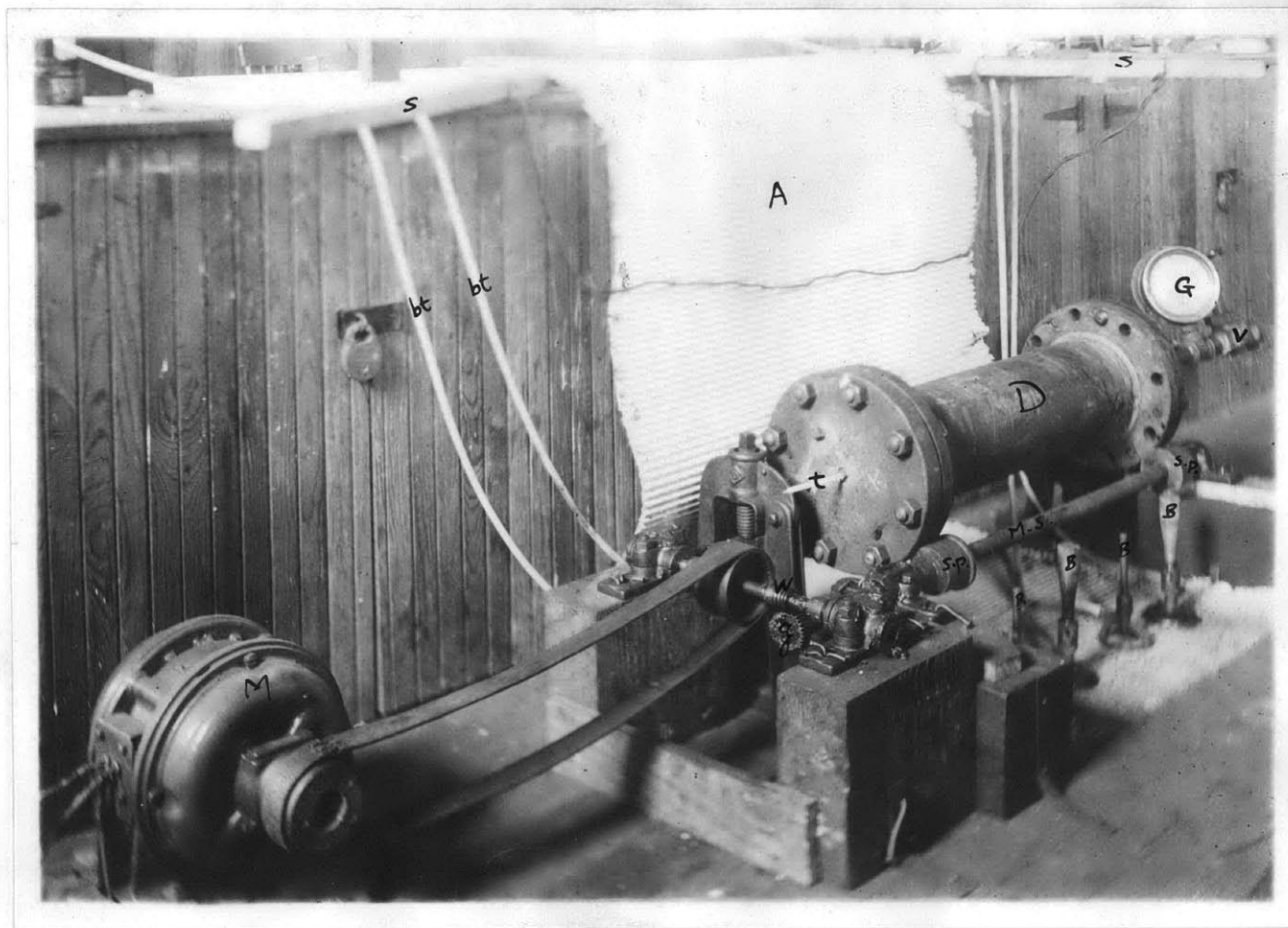
Auxiliaries on digester should be made of iron or steel

The valve serves many purposes

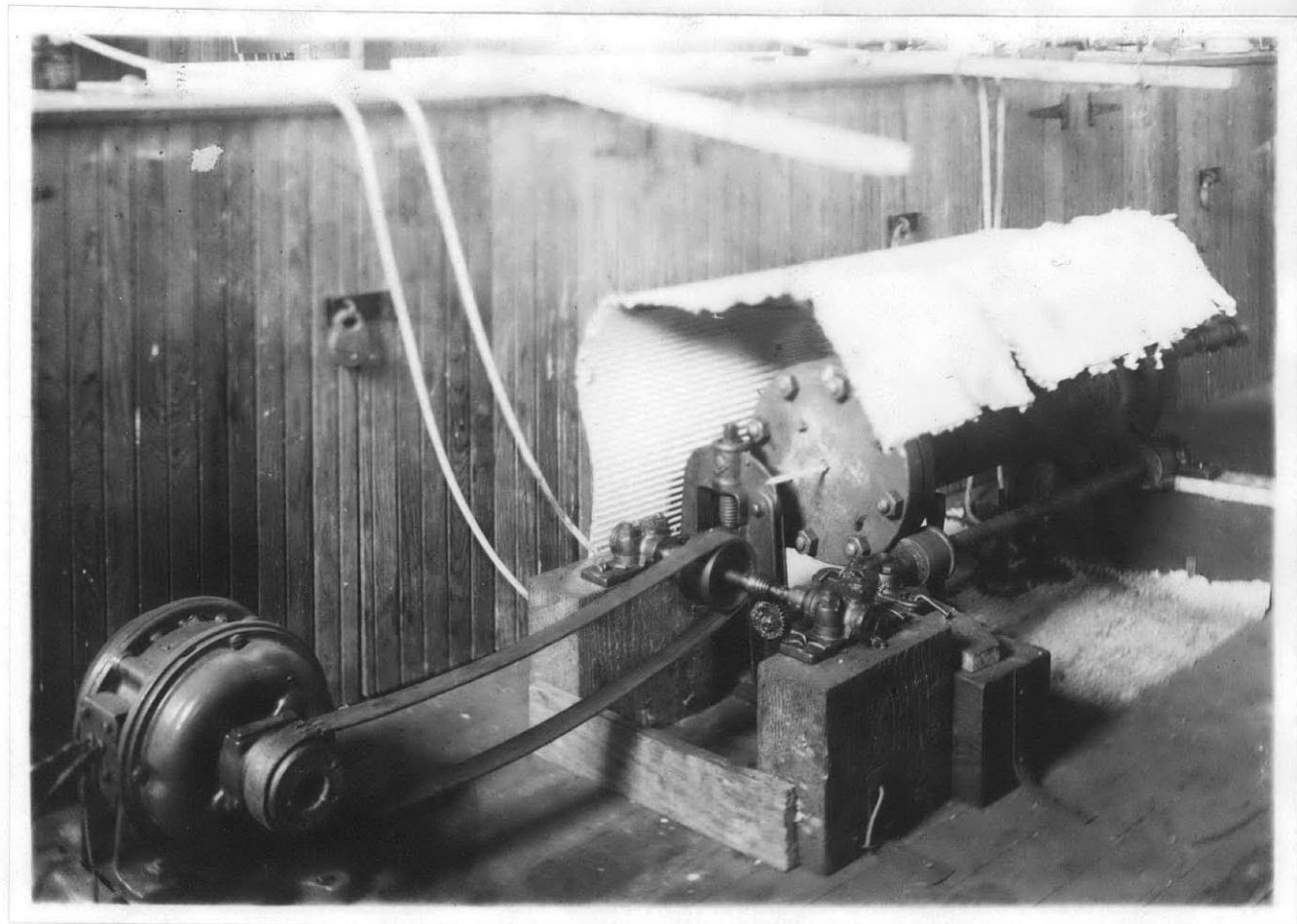
The idea of internal stirring of the charge in the digester was abandoned because of the difficulty of packing the stirring apparatus, which

The stirring device

A = Asbestos sheet
B = Burners
bt = Burner tubing
D = Digester
G = Pressure Gage
g = gear
M = Motor
M.s. = Main Shaft
S = supports for
asbestos sheet
t = thermometer
s.p. = solid pulley
V = Valve
W = Worm.



Photograph of Digester & Stirring device, showing detailed parts.



Photograph of Digester & Stirring Device,
as they appear during Operation.

has to come out of the digester, from steam leaking. Therefore external stirring device was resorted to. The following was devised, constructed and found to run very satisfactory. This consists of two shafts rest in bearings which are supported. Two solid pullys were put on each shaft at distance which will be enough to just offer support for the flanges of the digester. The pulleys have a ~~rain~~ towards center of shaft to prevent the flange of digester from slipping. On the ~~end~~ of one of the shafts which is longer than the other, a gear was set on. This gear was run by a worm fixed on another shaft perpendicular to the first. This third shaft also rests on bearings well supported and it was driven by a motor through a belt and a pulley. Thus, we see, the motor drives the worm shaft which, through the arrangement of the gear, drives the main shaft, which, in turn, ~~totates~~ the digester. The shaft parallel to the main shaft is only a support to the digester and it is turned by the digester, but not connected to the main shaft. It is also through the worm gear arrangement and of the size of solid pulley to digester that the speed is reduced from 1700 r.p.m., the ~~rated~~ speed of the motor, to 14 - 15 r.p.m., the speed of the digester. The gear used has thirty teeth. The motor is a 1 HP three phase induction motor. The whole stirring arrangement works pretty good and no part gets out of order during

the whole series of run. The digester, being heavy, stayed on the shaft very well, and no collars were needed between the bearings and the main & parallel shafts.

The heating system consists of Bunsen Burners burning under-neath the digester. Asbestos board supported by wires were hung over the digester to avoid radiation losses. With four burners to start with, it usually takes only ten minutes to get steaming of the cooking liquor, 35 minutes to reach the maximum temperature of 170°C. At that temperature, two burners are enough to maintain it with only small variations. The reducing of temperature from 170°C to 156°C takes from 10 minutes to 40 minutes depending whether the asbestos sheet is removed and and burner extinguished or not. At 156°C, one burner is enough to maintain it. As the digester is rotated while the burners are stationary, the heating of the former is very uniform and judging from experiences in later works, this heat system is very easy to control.

The heating system

Heating system satisfactory

The digester, and the stirring device, as described above fulfills the requirements of a digester.

The only defects are:

1. The direct attachment of gage is wasteful and may be dangerous
2. The lack of a charging and blowing devices greatly handicap the work as they render the handling of the digester more different. Every

loading and unloading can only be accomplished by taking the digester from the stirring mechanisms, and standing it up; and in case of unloading, time is wasted in waiting the digester to cool down to be comfortably handled. Moreover, as found in the later part of the experiments, the lacking of blowing device causes the fibres of the cooked pulps to bundle together like seeds without being opened up.

Lack of blowing device is defect of digester

However, the digester as it was is not fit to have a blowing device which should attach to the bottom no matter whether the digester is vertical or not. As the present digester is rotating there is no convenient place to attach a blowing pipe.

In general, the digester was satisfactory. Although several leakages has occurred at the threads of the flange joints during the cooking, but no serious defects were observed and these leakages are believed to have caused by the improper threading of digester body.

(d) Analysis of Bamboo.

In order to determine the composition of the bamboo and to find out what amount of NaOH is necessary for bare digestion (following the method of M. Raitt) of this particular specie of bamboo, analysis of bamboo were made. As Raitt claims the existence of marked differences between the composition of nodes

and internodes, analyses of both were made. From analyses of both nodes and internodes, and assuming a ratio of the two as existed in the whole culm, the composition of the whole culm can be calculated.

To get sample for analysis, the node and internodes were filed separately by a wood file and samples taken. This reduces the bamboo to fairly fine particles.

Method of
sampling

The procedure of analyzing the composition of bamboo either at the node or at the internode was adapted from that of Raitt, and the principal constituents to be analyzed are:

Constituents
to be analyzed

1. Moisture
2. Ash
3. Water extracts, consist mainly of starch and starch secondary products.
4. Pectose, including very small amount of Fat and Wax.
5. Lignin
6. Cellulose

Each constituent have to be analyzed separately and several determinations of the same constituents usually have to be made to get check results. The procedure is as follows.

1. To determine for moisture, about .2 - .3 grams samples of bamboo filings were weighed out on the analytical balance, dried to constant weight and

Moisture
determina-
tion

the loss of weight gives the amount of moisture and from this the percentage moisture can be calculated. This determination is usually very accurate, and offers no trouble.

2. To get ash content, sample of bamboo filings carefully weighed out was ignited in porcelain crucibles above the Bunsen flame. Here, care must be taken to heat the crucibles gently avoiding the blowing off of the ash by forcible flames. Ash contents

3. Water extract is determined by boiling weighed sample in test tubes with water for 1 or 2 minutes. The residue filtered, washed and dried to constant weight at 100°C. The loss of weight represents the quantity of water extract and moisture present. This (on the percentage basis) minus the percentage of moisture as previously found gives the % of water extract. Water Extract

4. The determination of Pectose follows the same procedure only this time 1% NaOH soln is used in treating the bamboo filings. The loss of weight due to this treatment gives amount of pectose, water extract and moisture as starches will undoubtedly dissolve by 1% NaOH solution if it is soluble in water. The total loss (on percentage basis) minus the % of moisture and water extract as found gives the % of pectose in Sample. Pectose

5 & 6. The determination of lignin and

cellulose is done in one step. The principle being to isolate the cellulose, and the loss of weight will represent the amount of lignin together with moisture, water extract and pectose. The isolation of cellulose was done by Cross and Bevan's chlorination process.¹⁵

Lignin
and
Cellulose

The weighed sample was first treated with a soln 1% NaOH, boiled for about 2 minutes. It is well washed on a cloth filter, and squeezed to retain not more than its own weight of water. In this condition, it was transferred from the cloth filter to a porcelain crucible and was exposed for one hour to an atmosphere of washed chlorine, the chlorine being prepared by the action of HCl on $KMnO_4$. The fully chlorinated fibres which are yellow in color were then transferred to a test tube, and boiled with a soln of 1% Na_2SO_3 which dissolved the chlorinated products. To complete the isolation of cellulose, the solution was made alkaline with 2% NaOH after which the boiling is continued for 2 or 3 minutes. The fibrous mass which is pure cellulose was then drained through on a gooch filter, fitted with asbestos previously dried and weighed, and thoroughly washed. It is then dried to constant weight. The weight of residue represents the amount of cellulose in the sample.

Chlorination
process used
for deter-
mining cel-
lulose

This method, although simple in principal and considered to be the best, in determining cellulose, is difficult to get accurate results because

the best amount of reagent added is not known and the time of boiling is not specified. The transfer of the moist mass after treatment with NaOH to the porcelain crucible, even carefully done, cannot be exactly quantitative, and a little loss of material is expected. Among the many determinations that were tried on this process, the best checks found were within .6 of 1%. It is believed that with some practise and with determination runs under the same condition every time, accurate checks could be obtained, although the best condition to get accurate determinations have to be secured by experimentation.

There are many other ways of determining cellulose as found in the literature but most of them are long and some determine only part of the cellulose. As the constitution of cellulose is not fully agreed upon it is perhaps not surprising to find such divergence of opinion about its determinations. No doubt, accurate and rapid method of determination will soon be devised after we know more about cellulose.

Ideal method for cellulose determination yet to be devised

Following the above described procedure, determinations of the constituents were undertaken, with one constituent analysed at a time. Runs were made for each determination until results check. The nodes and internodes were both analysed in the same way.

The original data of these determination can be found in the appendix while the merit of the results

of these analyses will be discussed under the next section.

(e) Cooking with excess caustic under atmospheric pressure.

In order to check Raitt method of calculating the % NaOH necessary for bare digestion, it was suggested by Professor Haslam, of the Chemical Engineering Department of Massachusetts Institute of Technology to digest a sample with excess alkali under atmospheric pressure for two or three days until the residual alkali in the liquor is found to be constant by analysis. The mixture being kept boiling all the time. By this treatment, the amount of caustic used up will represent the amount necessary for bare digestion. As the sample was boiled under atmosphere pressure, the hydrolysis of cellulose is not serious. And as the excess alkali was soon removed after the solution of pectose and lignin was complete, no ill effect on the isolated cellulose will be caused by the alkali. The flask in which the crushed bamboo was boiled, was attached with a reflux condenser to keep amount of soln in flask constant. The idea of adding water occasionally to replace the loss due to evaporation was tedious and also there was considerable foaming during the cooking which can only be remedied by attachment of a reflux condenser.

The residue after this treatment was washed and dried and its weight will represent the amount of cellulose present. Although this is a rough way of analysing for cellulose and experiment was not intended for this purpose, yet the result obtained show surprising coincidence with that found by analysis. It is highly desirable to try out this method for cellulose determination.

Method give good indication of the amount of cellulose

2. COOKING WITH NaOH

As discussed previously, there are four factors in the soda process, that effect the yield and quality of the product. These are: Temperature and pressure, duration, concentration of cooking liquor, and amount of caustic soda added per unit of raw material. With the limited time that was assigned to the Thesis and the small amount of bamboo (about 15#) on hand, it was utterly impossible to vary all the four factors in order to get the best conditions for cooking of **this** particular specie of bamboo by soda process.

The best could be done seems to vary one of the factors.

Raitt claims he has found the best time and temperature for cooking bamboo and they do not vary much with the different species he worked on, so it was decided to keep these two factors constant and follow his findings. The concentration of the initial soln,

Decided to vary only one factor

Time and temperature kept constant

effects the cooking not so much as the other factors and its value differs with individual factory practice and the kind of wood used. Of course, the more concentrated the soln is, the more complete and severe will the reaction be. The limiting factor be the amount of soln necessary to insure complete immersion of the charge. For this particular case, it was arbitrarily decided to keep the concentration constant at 50 grams per liter. This is quite a good average used in cooking wood. Thus the only factors that have to be varied is the amount of caustic charged per unit of wood and this is undoubtedly the most important factor.

Fixed concentration at 50grams/liter

(a) The Constant Factors .

The best time and temperature for cooking bamboo as found by Raitt were to raise the pressure to 120 lbs and maintain it for an hour; then lower the pressure to 80 lbs. and maintain that for 4 hours. As the species of bamboo in India are very much larger in size, it was thought to use the maximum pressure of 110 lbs. in this case, while keeping the rest of the schedule as it was. The gage used was very roughly graduated and not very accurate, so the readings were depended on the temperature. Readings of both instruments were taken at convenient intervals during the whole run and plotted as graphic logs which can be found elsewhere in the appendix. As mentioned before, the temperature can be kept reasonably constant by the burners.

The variations of temperature and pressure were kept the same during all six runs but small divergions are apt to occur as found in the graphic logs.

However, a serious mistake was made in interpreting Raitt figures on pressure as expressed in lbs per sq. inch absolute while they were really just gage pressures. So the pressures employed were about 15 lbs. lower than Raitt's best conditions. Of course, this affects the results and as will be shown later, it decreases the percentage efficiency of caustic consumption. But owing to the fact that they were kept the same through the whole series of runs, the effect of varying the amount of NaOH charged on the yield and its quantities can still be studied without any hindrance.

Mistook
pressure
as lbs/sq.
in. absolute

Except for the first cooking, the concentrations of initial solution of the subsequent cookings were all kept constant at 50 grams/liter. The stock soln of NaOH was made from the "technical granules" and its composition (grams of NaOH/cc) was determined by titrating its diluted solution against standard acid. In each run, calculated amount of NaOH was taken from the stock and diluted to the desired concentration.

A stock
soln of NaOH
was made
for all
runs

(b) The varying factor.

Having found the percentage caustic necessary for bare digestion of bamboo by the method*

*The new method gives better results than that calculated by Raitt's method. Two results do not check.

described under section II, part e, it was thought well to start with this amount and increase it by steps of 2% for the subsequent cooks. But moments' thought led to the belief that since this amount is the minimum required, the concentration of the caustic solution towards the later part of the digestion will be so low that its penetrating force will not be enough to complete the solution of pectose and lignin unless excessive time be allowed and pressure increased. Both of these, of course, will affect the yield,. So it was decided to use caustic in excess of the minimum required to afford the necessary driving force at the end. So 20% alkali was used for the first cook. In the subsequent cooks, the amount of NaOH added was increased by steps of 2%. All these percentages being on this basis of normal air dry bamboo taken for digestion.

Used caustic in excess of minimum to start first cook

(c) Subsequent treatment of cooked pulp.

The cooked pulp, after being taken out from the digester was diluted with water and filtered through a thin cloth filter. The filtrate is the diluted black liquor. Liquid is then squeezed out of the pulp by hands and then again washed until the of the filtration came out to be one. The filtration were weighed with sp.gr. taken and sample analyzed for residue NaOH. In this analysis, $BaCl_2$ was added to the sample to precipitate the carbonates that might be present and the coloring matter, and the solution with

Analysed black liquor and washing water for residue NaOH

the ppt. was ~~titrated~~ against standard acid using phenolphthalein as an outside indicator. Results were fairly satisfactory.

Sample of the washed crude pulp was then dried and the total crude yield calculated by proportion.

It should be stated that during cooking, some pieces escaped complete cooking because of being thrown to the space at the ends of the digester and caught there. These uncooked pieces were usually picked out and its amount weighed (in the dry form) and % calculated. Another cause of these uncooked pieces is due to these pieces which are not thoroughly crushed.

Uncooked
pieces
also
determined

The washing on cloth filter is good enough when handling small quantities of pulp. For large quantities, wire screen is more preferable.

It was found in the course of experiments that washing with hot water is more efficient than that of cold. Injecting steam into a mixture of pulp and water, also help somewhat to open up the seed like formation of the cooked chips caused by lack of "blowing" at the end of the cook.

Washing
with hot
water is
more effi-
cient

(3) Bleaching of the Pulp

(a) Beating of the unbleached pulp.

As mentioned in previous occasion, the unbleached pulp obtained in the cooks were seed-like, that is, fibres cling in bundles. This is probably due to the fact that the lack of blowing at the end of

the cook prevent the fibrous bundles from being opened up. But in the later cooks, the seed-like formation seemed to decrease in size. As the later cooks were conducted with increasing percentage of caustic the severity of treatment may cause this change. Also, as mentioned previously the injection of steam into pulp and water mixture in washing may also help in opening up of these bundles.

No matter what is the cause of these seed-like formations, their appearance renders bleaching difficult because it is apparant that these bleaching solutions can only react with the outer side of the bundle without penetrating into the inner fibres. So Professor Haslam suggested that these pulps be beaten up in the beating machine. The idea was followed, and all the pulps were successively beaten in a small beater in Dr. A. D. Little's Research Laboratory. For about an hour, the most seedy pulps were all opened up. The beating was prevented from being excessive because the formation of hydro cellulose in beating may effect the subsequent bleaching.

Beating helps to open up seed-like bundles of fibre.

b) The bleaching conditions .

In order to have any comparison of the bleach consumption among the pulps from the different cooks, it is apparant that each pulp should be bleached to a standard white. But owing to the small quantity of pulp available, this is not practicable. So one

bleach run was made for each pulp with all conditions kept constant and consumption in each case was calculated. These values will be lower than these that could be obtained were the pulp all bleached to a standard white, but this does affect the relationship between the properties of the pulp and its bleach consumption. Discussion on that item in next section will make this more clear.

The bleaching conditions that were kept constant, except in the bleaching of the pulp from the first cook, were:

The constant bleaching conditions

- 1) Amount of unbleached pulp taken.
- 2) " " bleach added
- 3) " " total solution
- 4) Temperature of thermostat
- 5) Duration of bleach

For exact values of these conditions, kindly refer to the appendix.

The pulp from the first cook was left in the digester for two nights due to accident and consequently the color was much darker due to this long contamination with the black liquor. Knowing this pulp will take more bleach so more bleach was added than the other runs. This may not be so fair, because with any pulp the more bleach you add, the more the consumption will be. But as this pulp is apparently undercooked than the other cooks, it will take more bleach even if the accident were not happened.

However, this does effect any accurate quantitative determination of the relation of bleach consumption among the different cooks.

The bleaching was carried out in a glass jar fitted with a stirring paddle operated by a little motor. The bleaching soln was taken from a stock soln and it was analyzed before use. After the operation is through, the residue solution was analyzed for residual bleach while the pulp is washed and dried to determine loss on bleaching.

Pulp from cook #6 bleached to brilliant white although it appears with a tinge of yellow when it is made into a sheet by a hand mould. Samples of unbleached and bleached pulps can be found elsewhere in the appendix.

Pulp from cook #5 and 6 bleached to dull white while that from #3 to light yellow and that from #1 to dirty yellow. Pulp from #3 were also contaminated with black liquor for one night due to unavoidable reason.

4. MICROSCOPIC EXAM. OF FIBRES

With other means of testing the quantity of the fibre not at immediate disposal and also owing to shortness of time, microscopic examinations of fibres were undertaken as a rough means of measuring the quality of the fibre.

Microscopic exam. tells quality of fibre

Dimensions of the bamboo fibre were measured under the microscope and compared with the size of wood fibre from soda pulp.

A microphoto of the bamboo fibres was taken to show the characteristics of the bamboo fibres.

The results will be found in the next section of the report.

III. Discussion of Results.

TABLE I. ^(a) Results of Analyses.
ANALYSIS OF BAMBOO (*Phallostachys Puberula*)

	Nodes	Internodes	Whole Culm*
Moisture	5.46	5.63	5.61
Water Extract	7.58	9.16	9.00
Pectose	16.94	15.43	15.58
Lignin	24.10	22.26	22.45
Cellulose	44.88	46.39	46.24
Ash	1.04	1.13	1.12

* Calculated on the basis of 10% nodes to the whole culm.

The above table shows the percentage composition of the different constituents as found by analysis both in nodes and internodes of this particular specie of bamboo; the third column represents the average composition of the whole culm calculated on the assumption that this bamboo has 10% nodes by weight.

A Comparison of the first two columns will reveal the fact that the composition of both nodes and internodes is nearly identical with but small variations. but little

Composition
of nodes
and inter-
nodes vary

This does not agree to the results of Raitt who claims that there are marked differences between the composition of nodes and internodes. His experimental data goes to show that nodes contain higher percentage of water extract. Pectose and lignin with much lower cellulose contents. While in the present case, the water extract in the nodes is less than that of the internodes, while the difference between the pectose, lignin and cellulose of nodes and internodes appears to be very slight. Of course, whether this is only characteristic to this particular specie or is common among all bamboos in China, the writer does not feel justified to conclude as he has not had the opportunity of analyzing the other species. However, the result of the present case is beyond doubt because all analyses were very carefully carried out.

Does not agree with Raitt's results

Another apparent conclusion that could be drawn from the results of analyses is the low percentage cellulose content of this specie of bamboo. All Indian bamboos seem to have a cellulose content of more than 50%. Here again, the writer does not feel free to say whether the present case is a representation of the Chinese bamboos or not. However, the result, of ^{was checked by the experiment} cooking bamboo with excessive alkali under atmospheric pressure. checked by other means. In the latter case, the % cellulose found is 46% while that from analysis is 46.24%.

Percentage cellulose content is low

Results checked by other means

Now, from the result of analysis, it was planned

to calculate the % of NaOH necessary for bare digestion using Raitt's values of the percentages of NaOH necessary to react with 1% of water extract, pectose and lignin (as found in the analysis) respectively.

Thus we have:-

	Water Extract	Pectose	Lignin
% wet basis	9.00	15.58	22.45
% dry basis	9.54	16.50	23.80
% NaOH required for 1% (dry basis)	.22	.32	.66
% NaOH required for the percentage found in analysis	2.07	5.28	15.70

The total amount of NaOH necessary equals to 23.05% on bone dry bamboo or 21.75% on normal air dry bamboo. This is certainly too high, for results of caustic consumption from the different cooks show that the highest amount is only 18.80%. The same item as found by digestion of bamboo with excess alkali under atmospheric pressure is only 14.05%, which appears more reasonable.

% NaOH for
bare diges-
tion calcu-
lated is
high

b) Effect of varying the amount of NaOH added

RUN No.	1	3	4	5	6
% Total Crude Yield	50.8	44.5	40.0	38.7	32.9
% NaOH Added	19.85	22.15	24.00	26.00	28.00
% NaOH Consumption	15.25	15.60	17.35	16.40	18.80
% Efficiency of NaOH Consumption	76.81	70.44	72.28	63.06	67.14

Above is a table of cooking results. A glance

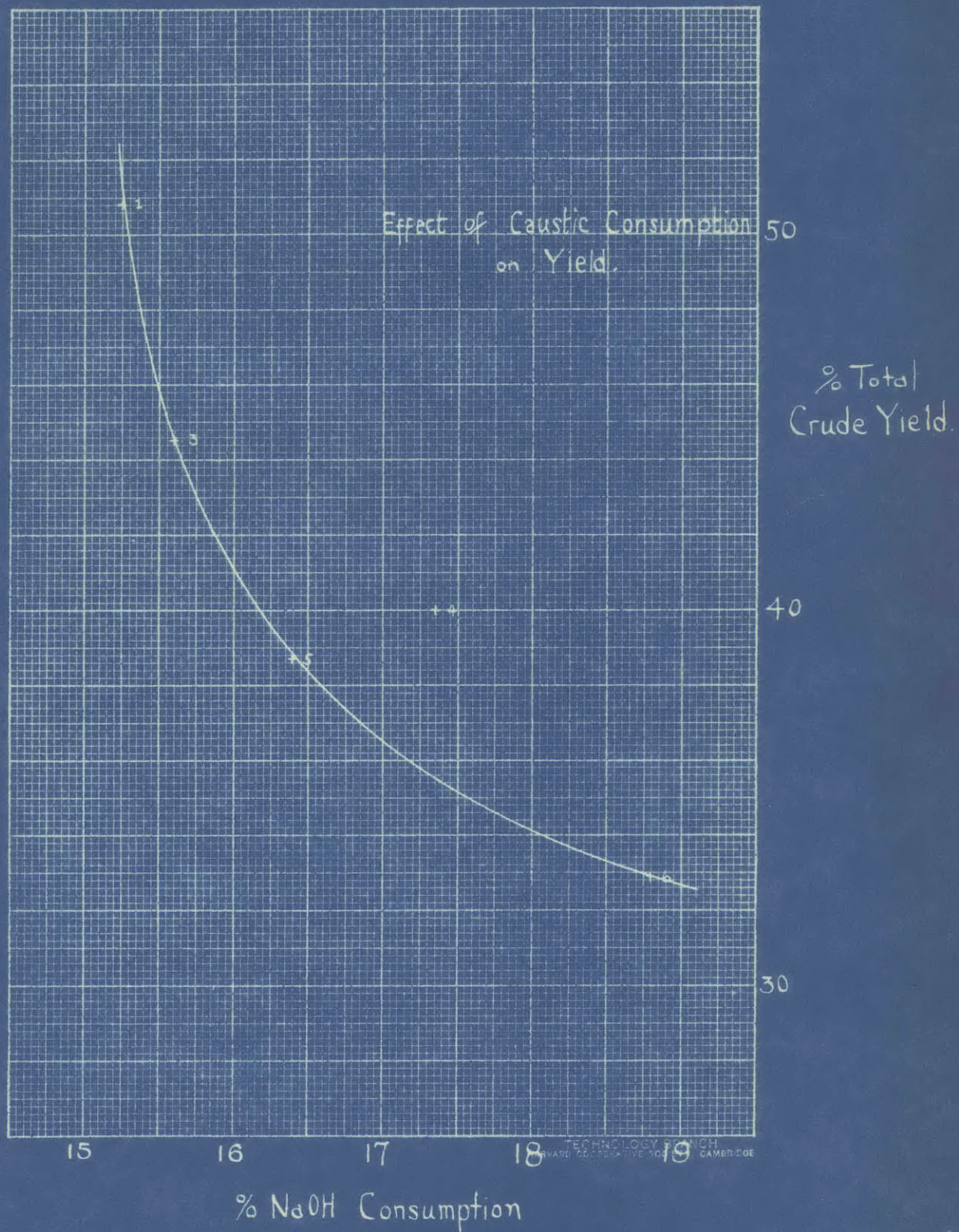


Fig 1.

will show ^{that} the increase of amount of NaOH added decreases the yield. This is due to more severity of cooking caused by the excess NaOH which acts as a driving force towards the end of the digestion. However, not all caustic added is consumed although it is true that consumption increases when more caustic^s added. On Fig. 1 % NaOH consumption is plotted against % total crude yield. From the shape of the curve, it is apparent that yield decreases with increase of NaOH consumption. Point 4 does not fit into the curve because during this cook, more NaOH is consumed than that of 5; this will naturally mean more complete reaction in cook 4, and this is shown to be correct by the ^{un-}usual small % of uncooked chips in this cook as compared with the others, (for complete data, see under original data in the appendix.)

Fig. 2 is a plot of % efficiency of NaOH consumption against % NaOH added. The slope of the curve shows that decrease of efficiency is caused by increase of % NaOH added. This can be explained in this way.

When NaOH reacts with bamboo it first attacks the pectose and lignin. After this reaction is complete, it begins to attack cellulose. It is quite probable that the reaction between NaOH and the former group is quite different from that between NaOH and cellulose. So, when small percentage of NaOH is present, it tends to dissolve all the pectose and lignin,

Increase
NaOH added
decreases
the yield

Increase of
NaOH added
decrease ef-
ficiency of
NaOH consump-
tion

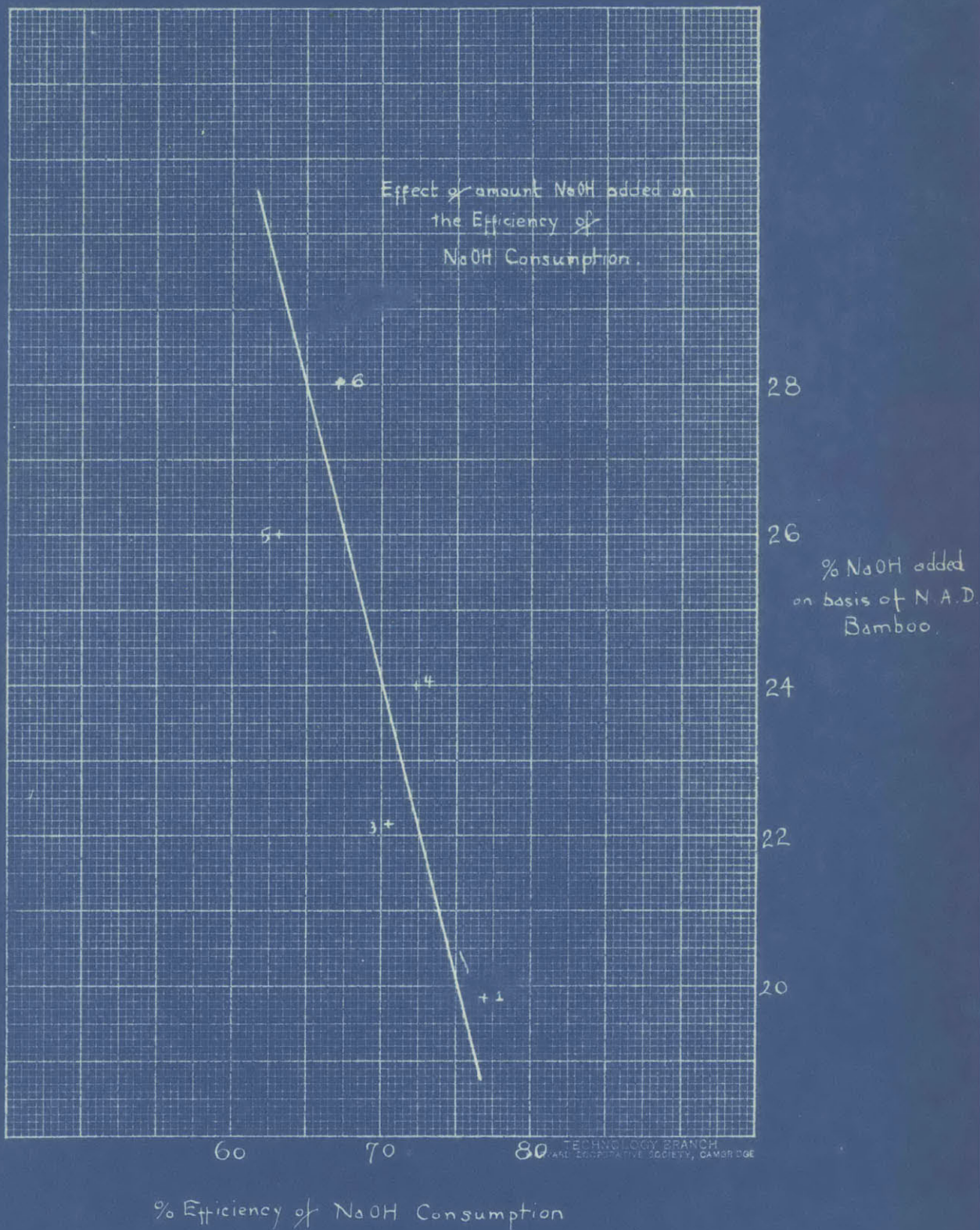


Fig. 2.

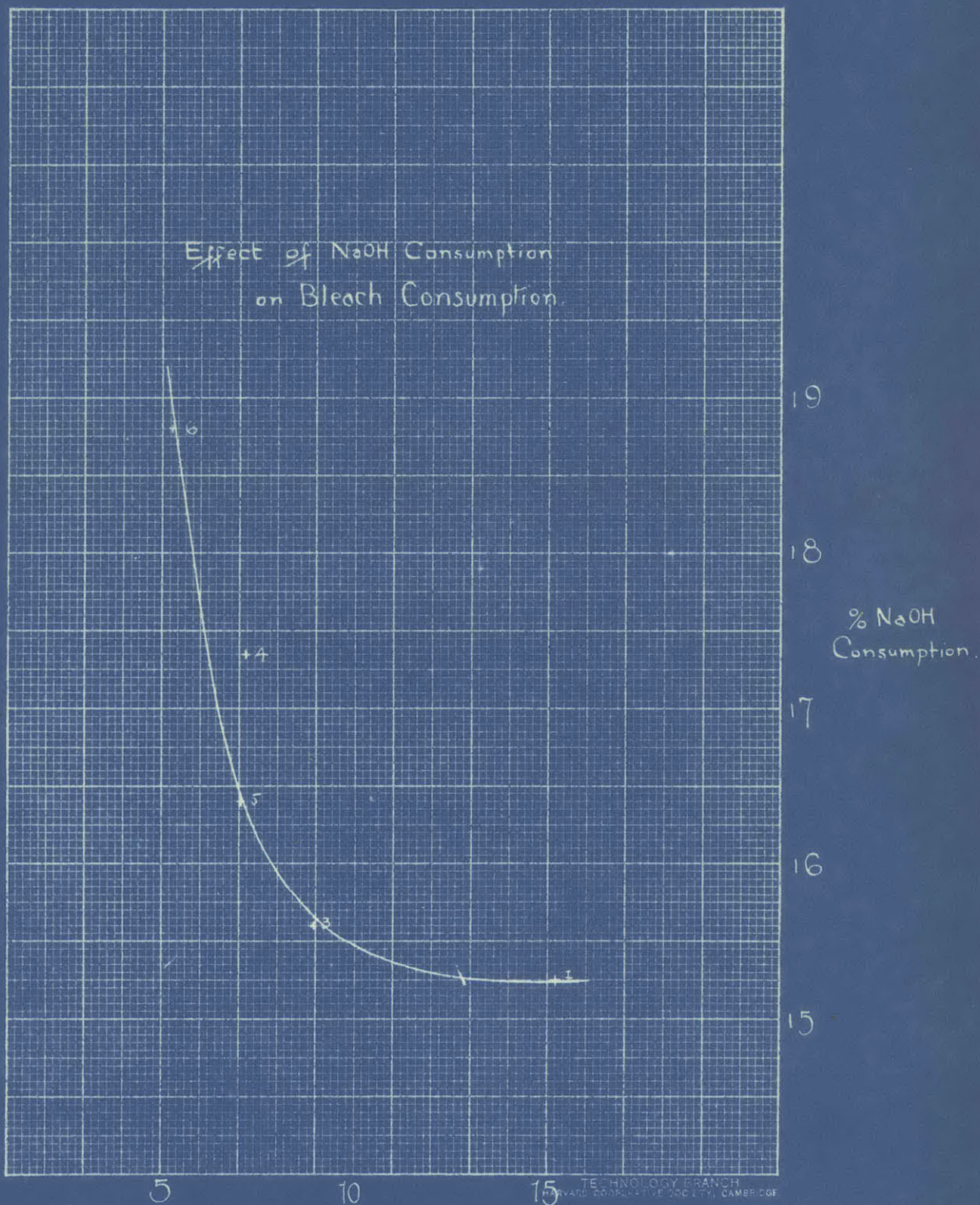
hence efficiency consumption is high. When more NaOH is present, there is an amount in excess of the required to dissolve the pectose and lignin, and this excess reacts with the cellulose. But due to the apparent smaller % of NaOH necessary to react with cellulose, the consumption is small and therefore the percentage efficiency is also small.

It must be stated here that from the data it can be seen that the general efficiency of caustic consumption in the series of runs is low, the higher being only 76.81%. In factory practise this is uneconomical because the alkali has to be recovered and it is wasteful to have great amount of NaOH going through the process unused as in case of low efficiency consumption. However, this efficiency can be increased by varying the ^{other} cooking condition; namely that of temperature and pressure, duration of cooking and concentration of initial soln. It is very probable that the temperature and duration employed were both the best conditions and the mistake of interpreting Raitt's best pressure for cooking bamboo as ^{on} a basis of absolute pressure may have caused this low efficiency.

General efficiency of whole series of runs is low

Efficiency can be increased by varying the other cooking conditions.

c) Effect of pulps from different cooks on Bleach Consumption.



% Bleach Consumption of 35% Bleaching Powder on basis of Raw Material.

Fig. 3.

TABLE III. Bleaching results.

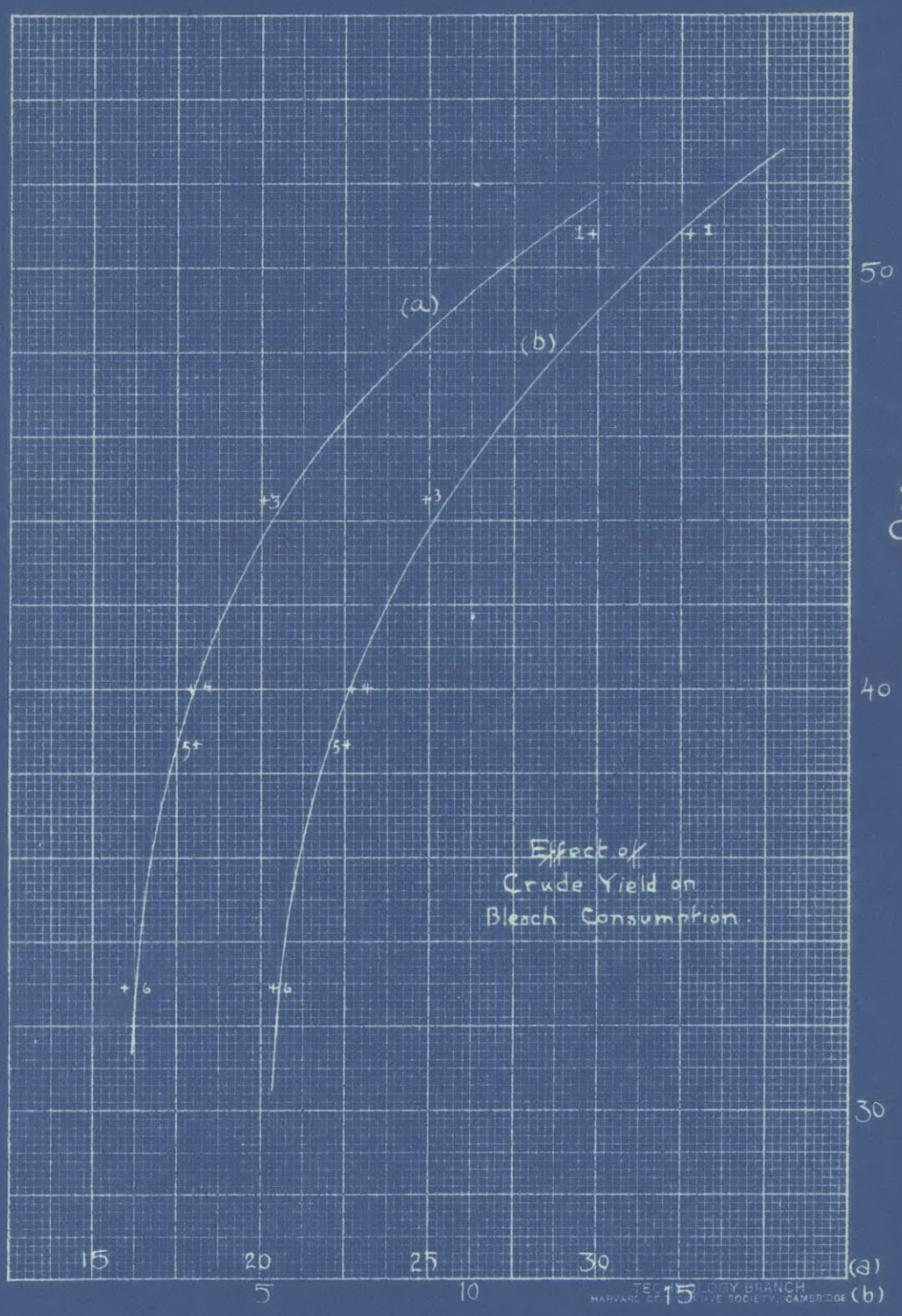
RUN NO.	1	3	4	5	6
% B. Powder Consumption					
a. on Basis of Raw Mat.	15.2	8.95	7.15	7.02	5.25
b. " " " Unb. Pulp	29.9	20.1	17.85	18.12	15.95
% Bleached Yield	46.1	39.9	36.4	37.5	31.4
% Loss due to Bleach	4.7	4.6	3.6	1.2	1.5

In general the bleaching results show that well-cooked pulps require less bleach than those undercooked ones. Good cooks are produced by large caustic consumption, high pressure and long duration of cooking. Of course, it is necessary to have in mind that excess of these conditions will produce over cooked pulps where loss of fibre and NaOH consumption cannot be compensated by the low bleach consumption.

Figure 3 is a plot of % bleach consumption against % NaOH consumption. As high percentage caustic consumption means severity of cooking or completeness of reaction of the pulp produced with naturally required less bleach, so with decrease of % caustic consumption, we have increase bleach consumption. On this plot, point 4 is again out of the curve. As cook 4 took more NaOH than #5, it is expected that its bleach consumption be lower than that of 5, although when figured on basis of raw material, it appears a trifle greater.

Pulp with high % NaOH consumption has low bleach consumption

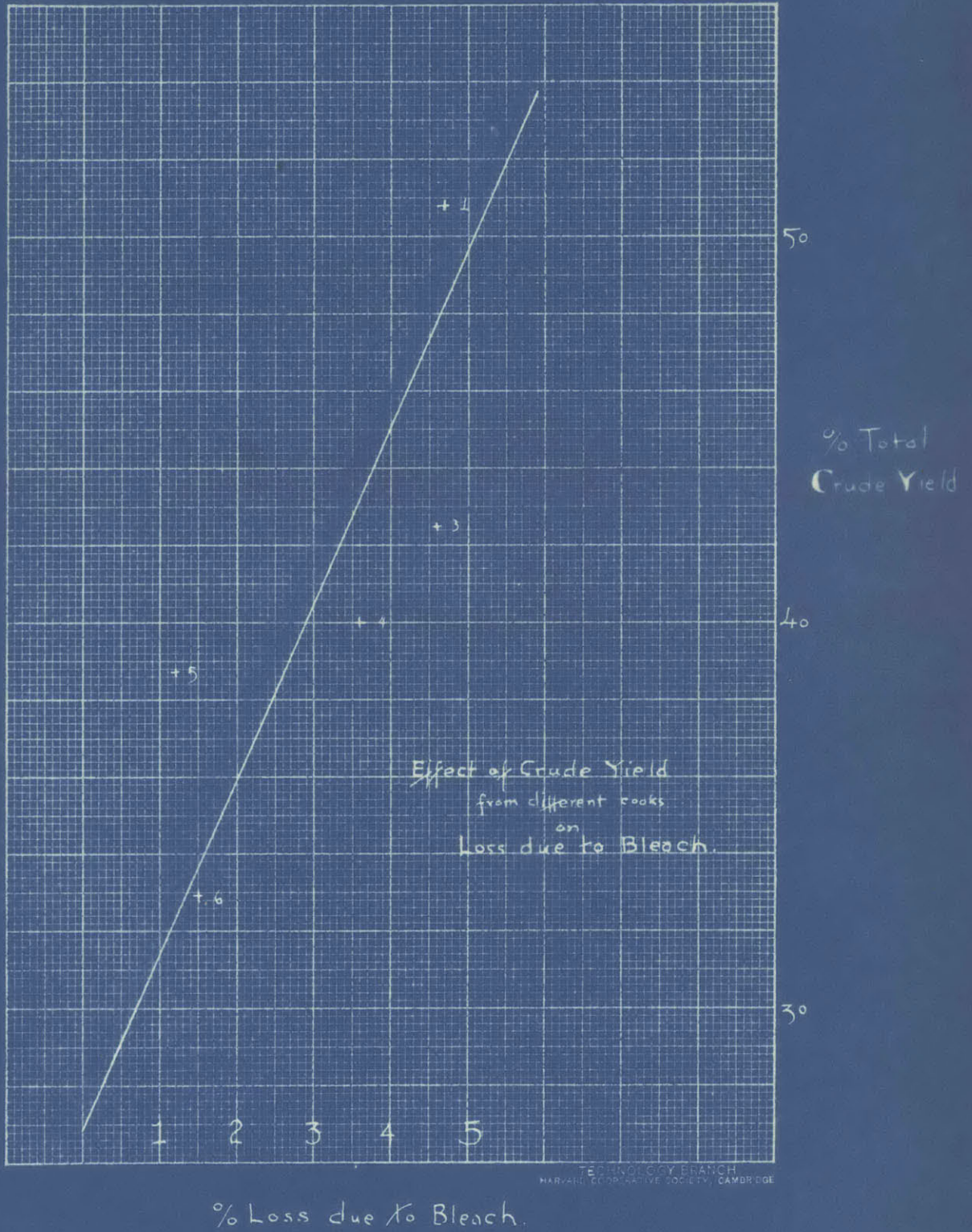
On Fig. 4, the relation of % Crude yield to % bleach consumption on two basis is shown. In both



Effect of
Crude Yield on
Bleach Consumption.

% Bleach Consumption of 35% Bleaching Powder on basis of
a) Unbleached Pulp
b) Raw Material

Fig. 4.



% Loss due to Bleach.

Fig. 5.

cases, large yield requires more bleach, because in large yield, the pulp is generally not well cooked.

Fig. 5 perhaps offers the most interesting result. It is a plot of % loss due to bleach against % total crush yield, and, it shows that loss due to bleach increases as yield increases. This is rather apparent; for as said before, large yields are produced from under cooking and they generally contain some residual lignin. Now, bleach acts on lignin, and so the great loss with large yield is the loss of lignin due to bleach, while with good or overcooked pulps, the loss is that of cellulose fibres. It must be pointed out here that small samples were used in bleaching and hence the determination of loss due to bleach by the difference of weight of the original unbleached pulp and the dried bleached pulp is not accurate. But they show up the relation pretty well.

Loss due to bleach increases with yield

In general, the bleach consumption is comparatively low. While run 1 was abnormal, all rest of the four pulps did not consume more than 10% bleach on basis of raw material, while with pulp #6, which is considered to be a well cooked pulp, it consumed only 5.25%. With Indian bamboo, Raitt finds the best bleach consumption for soda pulp is about 10%.

Bleach consumption is low

d) Microscopic examination of fibres. Measuring the length and width of the fibre of bamboo under the microscope reveals the fact that Bamboo fibres are

Bamboo fibres are longer than wood fibres from soda process

are much larger than the ordinary wood fibres from the soda process, although the width is little less. A glance of the following table will make this clear.

TABLE IV - Comparison of the dimension of fibres from Soda Process.

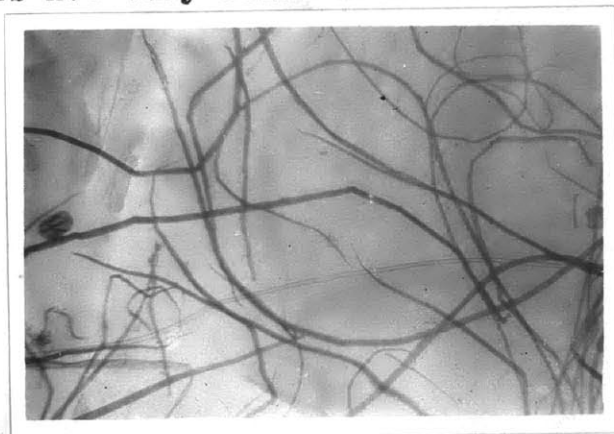
	Length	Width
Bamboo	1.5 - 2.8 mm	.013 - .017
Beach	1.7 - 1.72	.015 - .029
Poplar	.71 - 1.62	.020 - .044
Aspen	.78 - 1.68	.020 - .046
Birch	.78 - 1.63	.014 - .042

The long fibres helps to make a strong sheet of paper as the fibres have more chance to ~~cling~~ ^{Advantage of long fibres} together.

Usually wood fibres from Soda process are shorter comparing ^{with} that from sulphite or sulphate processes.

Raitt claims that the length of ultimate fibres of bamboo is from 2.2mm to 2.6mm according to variety and the diameters are from .018 to .027 mm. This checks pretty well with the present results.

The following is a microphoto of the bamboo fibres. It is not very well taken but it shows that bamboo



Bamboo fibres,
magnified 65
diameters

bamboo fibres are spindle shaped and the ends pointed.

A few cells can also be seen in the photograph.

e) Summary of Conclusions

Viewing from the above results and discussions, the writer feels confident to draw the following conclusions.

1. The composition of the nodes and internodes of the specie of bamboo under investigation does not vary much.

2. The cellulose content of this specie is rather low, about 46%.

3. Cooking bamboo with excess caustic under atmospheric pressure gives good indication of cellulose content and percentage caustic necessary for ~~bare~~ digestion.

4. The effects of increasing the amount of caustic added while keeping the outer factors constant are:

a) Decrease of yield

b) Decrease of Efficiency^{of} NaOH consumption.

5. The conditions that were kept constant during the series of runs are not the best conditions.

6. Bleach consumption decreases with decreasing yield or increasing NaOH consumption.

7. Loss due to bleach decreases with decrease of yield or increase of NaOH consumption

8. The specie is not best fitted for paper

making because of the low cellulose content although it cooks well with 18 - 19 % of NaOH and bleached white with a slight tinge of yellow with about 7% bleaching powder.

IV. RECOMMENDATIONS

The present investigation covered very little ground and many things that ought to be done are not accomplished due to shortness of time. For the benefit of ^{future} investigators, the writer wishes to make the following recommendations:

1. Analyses of the composition of bamboo is not absolute necessary before carrying on any digestion, although the determination of cellulose content is very desirable. To start a thorough investigation, all the common species of bamboo in any country, for instance China, should be analyzed for cellulose content and the specie with the highest value should be selected as the raw material. In this connection the Cross and Bevan's chlorination method should be standardized in order to insure uniform results or better to check the results by some other methods.

Select new material by its cellulose content

2. In studying a particular process for paper making, all factors affect the cook must be varied one after another in order to get the best condition? For example, with the soda process, the

All factors must be varied to get good conditions

best temperature and pressure should be found first, then the best duration, then the best amount of caustic added and finally the best initial concentration. To determine the best amount of caustic added, Raitt method of comparison of the unit cost is recommended to be tried out.

3. It is highly desirable to make similar investigation with the sulphate process which is not only considered as the best process for bamboo but it is well known for producing stronger fibres.

Highly desirable to investigate the sulphate process

4. In determining the bleach consumption of the different cooked pulps, the best bleaching condition should be investigated. Then every pulp should be bleached to standard white by series of runs.

5. The pulp from the different cooks should be investigated regarding to quality. This can be accomplished by

Quality of pulp should be investigated

- a) Comparison of color
- b) " " amount of shives present
- c) " " ash content
- d) " " strength
- e) " " bleach consumption
- f) " " loss on bleaching
- g) Microscopic examinations.

6. The digester employed should have good heating system, good stirring device and devices for loading and blowing. Vertical and stationary digesters are more preferable.

A P P E N D I X

I. Description of Bamboo

The arborescent grasses known as bamboos all belong to one tribe of the family Graminae, but are referred to 30 genera and comprise of 550 different species. The hardness of the stem of bamboo is due to the close compact deposition especially in surface cells, of particles of silicious matter. The hollow stem of the bamboo plant is interrupted at intervals by solid nodes, the portion of hollow stem between two nodes is called the internode. Being an endogenous plant, bamboo stem consists of fibres and fibrous tissue alone, combined with mucilaginous gummo-resinous and other extractive matters common to all vegetable growth.¹

Bamboo has many species

Nodes and Internodes

A few species of bamboo extend to the temperate regions, but the great majority are natives of humid regions of equatorial and subtropical latitudes. They grow in light rich soil with phenomenal rapidity. Most of them are perennial and have more or less branching rhizomes, from which rise the erect culm or stem.

I. The distribution of Bamboo of different species
are as follows:²

Distribution
of Bamboo

1. Monson region of Asia	320	species
2. India(15 genera	136	"
3. South America (8 genera)	179	"
4. Japan	49	"
5. Philippines	30	"
6. New Guinea	8	"
7. Queensland	3	"
8. New Caledonia	4	"
9. Pacific Islands	2	"
10. Africa	24	"
11. Madagascar	5	"

Bamboo usually flowers at long intervals - thirty
or forty years - all the representatives of any one
species in the district come into blossom simultaneously.

Flowers at
long
intervals

After flowering, the culms die, and sometimes the
rhizomes die too. Even if the rhizomes are killed by
flowering, new ones will be produced from self sown
seed in 5 or 6 days, and culm suitable for extraction
will again be available in 8 or 10 years after the date
of flowering.

2. Composition of Bamboo. According to Pearsons,³
only five species of bamboo in India are suitable for
paper making, viz:-

Bambusa Tulda
B. Arundinacea
B. Polymorpha
Cephalostachym Pergracile
Melcanna Bambusoids

A Analysis of three of the above mentioned, bone dry Indian Bamboos are:⁴

	B.Polymorpha %	B.Arundinacea %	C.Pergracile %
Cellulose	54.71	50.32	52.73
Fat & Wax	1.05	1.17	.92
Water extract	8.95	8.48	7.96
Pectose	19.55	24.39	23.09
Lignin	15.74	15.64	15.30
	<u>100.00</u>	<u>100.00</u>	<u>100.00</u>
Ash	3.97	1.60	2.57

Characteristic analyses of Philippine bamboos are given by Richmond as follows:⁵

	Strudtural Bamboo %	Dwarf Bamboo %
Cellulose	53.94	55.75
Fat & Wax	.96	1.03
Water Extract	4.98	4.69
Lignin	24.25	21.27
Water	12.40	11.20
Ash	3.47	6.03

3. Microscopical appearance of Bamboo. The chief feature of the structure of bamboo are the cappillary tubes or sap canals, arranged in groups of four. Among the capillary tubes are arranged the most useful paper fibres. According to Dr. Hanansek,⁶ noted botanist in Vienna, the bamboo fibres are spindle-shaped and smooth, the ends pointed and not furcated. The walls of the short fibres are highly thickened and therefore the lumen is narrow. The fibres also have a special sheath and also knots and displacements. According to Raitt,⁶ the individual fibres resemble

Microscopi-
cal appear-
ance of
Bamboo

straw in appearance and color reaction but are twice as long. The average length of the ultimate fibre is from 2.2 mm to 2.6 mm according to the variety, and the diameters are from .018 to .027mm.⁴

Fibres resemble straw and twice as long

4. Physical Characteristics and how they effect subsequent treatment in pulping:-⁶ Bamboo is porous, thereby differing from coniferous wood; its pores run vertically in close, straight, and regular series through out the culm. They form cappillary tubes, open throughout their whole length, which in green culm are filled with sap, and in dry with air. Dry bambóo is therefore impregnated with air in the state of cappilarity, and this condition renders its expulsion differ-ent. It has been found that chips of bamboo still holds air after two and half hours' digestion. The cappillary air also prevents the complete penetration of the chemical solvent into the tissues. The resistance of bamboo to the action of the digesting agents is increased by its contents of lignin and pectoses. The effect of the solvent action on both is to produce a partially dissolved waterproof colloid film which prevents it from the further attack of the solvent. The nodes in Bamboo are large and hard and strongly lignified, the larger portion of both pectose and lignin form more greater and persistant colloidal resistance to digestion. Bamboo, unlike wood, has very little mass resistance towards the action of the solvents.

Physical character

Capillary air difficult to remove

Colloidal film form pectose and lignin

5. Mechanical treatment of Bamboo.-⁶ If the bamboo is crushed instead of chipped, its specific difficulty disappear, not only with internodes, but with nodes. The crushing must be very thoroughly done. Raitt found the best result by passing the culms split in halves through the heavy cane crushing rollers of a modern sugar plant. By crushing, the air resistance is totally destroyed; the crushed mass sinks at once, the lightest floats for ten minutes only; the nodes difficulty is entirely removed, their additional pectous and lignifous contents sinks into insignificance. The only disadvantage resulting from crushing is the increase of space the mass occupies and the consequent increase of digesting liquor, the latter however, is compensated by the use of weaker liquors. A ton of bamboo chips occupies ninety cubic feet of space and requires 336 gallons of liquor to cover it after it ceases to float. A ton of crushed bamboo requires twice of these amounts. To summarize, the advantages of crushing over chipping are as follows:

- a. It economises the nodes and saves the cost of cutting them out.
- b. It saves two hours in the period of digestion.
- c. It permits a weak liquor to be used, and thus reducing the danger of fibre loss by hydrolysis.
- d. It gives a more evenly digested product.

Crushing solves many difficulties

Crushing increases volume of the mass.

Advantages of crushing

II. PAST INVESTIGATIONS ON THE PULPING OF BAMBOO

The first scientific investigation on the Chemical treatment and commercial possibilities of bamboo as a paper making material was undertaken by Mr. Thomas Routledge, a paper manufacturer in Sunderland, who found that bamboo yields a fibrous paper stock which make a quality of paper superior to Esparto and at considerably less cost. His work was published in two pamphlets under the titles "Bamboo considered as a paper making material, with remarks on its cultivation and treatment." (1875); "Bamboo and its treatment." (1879). Some years later, Mr. R. W. Sindall made an investigation for the Indian Government. His works are found in two pamphlets under the titles: "Report on the Manufacturing of Paper and Paper pulp in Burma" (1906) and "Bamboo for ^{Paper} making." (1909). Then there is Mr. Pearson who made similar investigation for the Government of India and his works are found in different periodicals. Perhaps the most important piece of work that has been done in this field is that of Mr. Wm. Raitt. Mr. Raitt was also in service with the Indian government, and he studied on that subject for about thirty years. His methods are systematic and results satisfactory. He contributed numerous articles to the paper periodicals and the most complete account of his work is found in the "Indian Forest Records" vol. 3, part 3 (1912). There

Thomas
Routledge
first
investiga-
tion

Raitt
studied on
the subject
for 30
years

There are other workers in this field including G. F. Richmond, Jardine and Nelson, McRae and Malcolmson. Six English Patents on this topic can also be found.

There are four chemical processes which are used in making of paper pulp, viz: the sulphite, soda, sulphate and lime processes. All of those processes have been tried in treating bamboo and the following is an account of the results that were obtained.

Four processes for making paper pulp.

1. SULPHITE PROCESS.

In this process, the liquid employed consists of an aqueous solution of bisulphite of calcium or magnesium, and free sulphurous acid, and is usually prepared by passing sulphurous acid gas (produced by burning sulphur or pyrites) up towers packed with lumps of limestone or dolomite, through which water is trickling. The digestors used are usually of steel, lined with lead or acid-resisting brick. The sulphite process is now the most important of the chemical processes used in the manufacture of wood pulp.

Raitt⁴ claims that sulphite process is unsuitable in tropical climates, for the absorption of SO_2 in water is effected by temperature higher than those normal.

Raitt claims it is unsuit-

able

to northern latitudes. As the liquor is corrosive, earth ware jars fitted inside the digester should be used. It was found that bamboo is very sensitive to charring in liquors of greater density than 7° Tw or at temperature above 150° C or in liquor containing an insufficient proportion of SO₂; but in a comparatively weak liquor of the following composition, it digested perfectly in ten hours, of which four were occupied in slowly raising the temperature to 145°C, it being then maintained at that point for further six hours.

Total SO ₂	3.60%
Combined ² SO ₂	1.46%
Free SO ₂	2.14%
Combine Lime	1.28%
Density of soln = 5° Tw.	

The resultant pulp amounted to 49% on the original weight and was of a yellow straw color. Bleaching reduced it to 46.3% and it requires 40% bleaching powder (on weight of unbleached pulp) to bring it up to standard white. Such a bleaching cost is not economically permissible and it seems clear that the sulphite process, even if workable in the tropics, has no advantage for the production of bamboo pulp.

Bleaching
consumption
not economi-
cally per-
missable

The production of the yellow color in the bamboo pulp when treated by the sulphite process is found to be due to the presence of the excess of free sulphurous acid in the liquor in the digester, which is said to char the fibre. JARDINE AND NELSON² (Eng. Patent 18371),

Jardine
and
Nelson
Patent

1913) claims to have overcome this difficulty. In their process, calcium bisulphate is not used, as an excess of sulphurous acid is required to keep the calcium salt in solution. The base selected must be therefore one which yields a soluble sulphite, such as Mg and Na. The bamboo, previously crushed, is digested with a solution of Mg or Na bisulphite which may initially contain an excess of sulphurous acid. During the heating up of the charge, the SO_2 is allowed to escape freely, and during digestion, the accumulation of SO_2 is prevented by a free exhaust being kept open during the whole of the digestion. It is claimed that by this treatment a yield of grayish-white pulp is obtained amounting to about 50% of the original weight of bamboo. The pulp can be easily bleached to a full white.

Base should
yield a
soluble
sulphite

In a later patent specification by Jardine and Nelson (1909/1915), a modification of this process is described, and directions are given to prepare a Mg bisulphite solution containing the requisite proportions of dissolved base and sulphurous acid. By this means the process can be so controlled by adjusting the temperature that the required proportion of base is present without an excess of sulphurous acid, and thus scarcely any sulphur dioxide will be volatilized, and during digestion no arrangement need be made for escape of the gas. The specification describes the application of the method to B. Arundinacea.

Modification
gets rid of
excess sul-
phurous
acid

Contrary to previous investigators, SCHMIDT⁷ finds the sulphite process suitable for the reduction of bamboo. By cooking 14 hours at 3 to $3\frac{1}{2}$ atm with $\text{Ca}(\text{HSO}_3)_2$ soln (density 2.8° Be), a yield of 15% soft, dark pulp was obtained, bleachable with 12% bleaching powder. The paper prepared from this pulp was found to be slightly self sized.

Schmidt finds this process is suitable

MERUSAWA⁸ (Japanese patent 31,675/1917) claims that in making pulp from bamboo, a cooking liquor containing NH_4HSO_3 together with SO_2 or any other soluble sulphite can be used. The acidity of the solution is maintained at .4 to .8 normal and the cooking is carried out in two stages. The NH_4HSO_3 soln is prepared by conducting SO_2 into the soln of $(\text{NH}_4)_2\text{CO}_3$ and $(\text{NH}_4)_2\text{SO}_4$.

Merusawa suggested using NH_4HSO_3

2. Soda Process.

In this process, the liquor used for digestion is an aqueous solution of caustic soda. The caustic soda is recovered by evaporating the liquor and re-causticising the residue with lime.

Sindall experienced unit Soda process

In his book "Bamboo for paper making" (1909),⁹ SINDALL stated that in 1908, experiments were made on B. Polymorpha by Messrs. Thomas and Green, SOHO mills, Woburn, Berks, who found that the material yielded readily to treatment when boiled for 7 hrs at 60 lbs. pressure with caustic soda having a density of 15 TW.

The boiled material was bleached with bleaching powder equivalent to 6% of bleaching powder calc. on dry bamboo pulp. The material worked exceedingly well in the paper machine and produced a good sheet of paper.

Further experiments were made with a more severe treatment and the use of 8 to 10% of B.P., the paper obtained being a much brighter color. Some of the bleached bamboo pulp was sent to the Northern Ireland Paper Mill Co., which firm made further experiments for the manufacture of a thin sheet of paper suitable for lithographic printing and found it very successful.

Raitt⁴ found that the digestion of bamboo by the process depends upon the following factors: Temperature, strength of NaOH soln, and time of digestion. The time of digestion is limited by the usual factory practice of 8 hr. day. To secure complete but bare digestion of crushed B. Polymorpha the minimum alkali being 15.79%, temperature 150°C and time 3½ to 6½ hours. The range of temperature is also limited, because the destruction of fibre at higher temperature, though small between 162°C and 170°C, is serious at 177°C, and loss is extravagant at 183°C. But high temperatures helps to overcome penetration resistance and improve color, and it was found that by conducting digestion at 177°C for not more than 1 hour and then reduce it to 162°C for the remainder of the time, the loss of fibre

Three fac-
tions effec-
ting Soda
digestion

Best Temp-
erature and
time found

the loss of fibre did not occur, and $1\frac{1}{2}$ hours are saved (digestions being managed in 5 hours) Having fixed the temperature and time, Raitt digested the bamboo with more severe treatment by increasing the alkali strength, he found that he could increase bleaching economy with sacrifice of yield. The economical balance can be found by comparison of their unit cost.

$$\text{Unit cost} = \frac{+(\% \text{ Bamboo } \times A) + (\% \text{ NaOH } \times B) + (\% \text{ B.P } \times C)}{\% \text{ Bleached yield}}$$

where A, B, and C are cost per unit of bamboo, NaOH and B. P. respectively. Percent of bamboo is taken as 100. Thus, in a certain case, while 16% NaOH is the amount necessary for bare digestion, it was found that 20% NaOH gives the minimum unit cost. For data of Raitt's work, see else where in this report.

The bleaching Powder consumption of bamboo pulp prepared by the soda process, even though in the best conditions, is undoubtedly high although its cost is more than compensated for by the cheapness of the raw material.

Bleaching
consumption
still high

RAITT² has also devised a "Fractional Digestion" process (Eng. Patent 15,779/1912) for treating of bamboo in successive stages. The raw material is first treated with hot water to remove starch and similar soluble carbohydrates. It is then digested with caustic soda of a strength and at a temperature sufficient to effect the breaking down of the pectose, but to

Fractional
digestion

leave the lignins unaffected. Subsequently a stronger soln of caustic soda is employed under temperature and pressure sufficient to effect the solution of the lignins. The final liquor may be used as a preliminary soln, in the treatment of fresh material.

According to the modification of this method (Eng. Patent 16,488/1915) the preliminary treatment with hot water is omitted, and the bamboo is digested directly with a soln of caustic soda, which is dilute enough so that the starch is dissolved without the formation of the dark-colored substances. The bamboo is subsequently treated with caustic soda of greater strength as in the original process. It is claimed that by this "Fractional Digestion" process, the cost of manipulation is reduced and bleaching facilitated.

Reduces
manipulation
and
facilitation
bleach

PEARSON⁷ stated that *B. Polymorpha* is the species best suited for the paper pulp. By cooking with 18% of NaOH for 1 hour at 120# and 5 hours at 82#, a yield of 46% pulp was obtained, the bleach consumption of which was 10%. *B. Pergracile* requires 23% NaOH and a longer cooking period, While *B. Arundinacea* is still more difficult to reduce, *Meloanna B.* yields an unbleachable pulp.

Pearson
found
behaviors
of different
species

RICHMOND¹⁰ working on Philippine bamboo, found that the soda process is very successful. He got

Richmond
found soda
process is
very success-
ful

about 43 to 45% yield of air dry unbleached fibre under the following conditions.

1. Upright cylindrical stationary digesters.
2. Direct steam heat.
3. 15 to 20% of 76% caustic soda, calculated on the air dry weight of the raw material.
4. a duration of cooking of 4 to 6 hours.
5. Maximum temperature 160°C corresponding to steam pressure of 90 lbs to the square inch.

Fibre, thus prepared, bleached to a splendid white with 12 to 15% of bleaching powder.

RAYNER¹¹ stated that the starchy materials, pectose and lignin exhibit varying solubilities in alkalines, and are chemically effected by the boiling lyes of the concentration necessary to the disintegration of the lignin, turning brown and fouling the material. A fractional treatment is provided, first with hot water at 100°C, next with 7% NaOH during 1 hour and finally with 18% NaOH for four hours and temperature of 162°C and under pressure of 79# (as against 177°C, 55Kg and 6 hours, the usual practise.)

Rayner
favors
fractional
treatment

GROUD¹² cooked bamboo for 3 - 4 hours under a pressure of 66# with 8 - 10% NaOH, washed, defibred and bleached, and got a yield of 45%. He claimed that bleaching with Cl does not yield as white a fibre as

Groud
favors elec-
trolytic
bleach

electrolytic bleach or KMnO_4 .

3. SULPHATE PROCESS

The liquor used in this case consists initially of a soln of sodium sulphate and caustic soda. After digestion with bamboo, the liquor is evaporated and the residue incinerated. In the course of digestion, however, the sulphate becomes reduced to sulphide by the organic matter dissolved from the bamboo. The resulting ash is dissolved in water, rendered causticly by being boiled with lime and used again, the loss of strength being made up by the additional of sodium sulphate.

RAITT⁴ claims that in the sulphate process, the actual digestion is done wholly by the $\text{NaOH}, \text{Na}_2\text{S}$ having but light saponifying effect on lignin. Its chief effect is to reduce the coloration products formed to compounds of a light color, and hold them more perfectly in soln, so that they can be easily washed out without ppt on the pulp. It therefore exercises a strong bleaching effect and the pulp produced is consequently lighter in color and more easily bleached by bleaching powder. It has also a retarding

Na_2S has strong bleaching effect

influence upon hydrolysis of fibre by NaOH, and to a slight extent, increases the yield. As the loss of soda is made up by sulphate of soda, which is a cheaper article than the carbonate, the cost of chemicals is slightly less than that for the soda process. Na_2S can only to a small extent be substituted for the NaOH necessary for bare digestion; but its bleaching effect is so much greater than that of excess NaOH that is desirable to use as much of it as circumstances will permit, provided the proportion of NaOH is not thereby reduced below the necessary amount, and the factor which chiefly controls the respective proportions is the efficiency of the soda recovery plant. With a recovery of 80 to 85%, these will usually average a ratio of three to one. The unbleached pulp from the sulphate process is a light brown in color, and there is also an improvement in softness and strength. Mr. Raitt's experimental data on the digestion of bamboo by the sulphate process can be found elsewhere in the report. The superiority of the sulphate process in treating bamboo is attested by its high degree of purity of the product as shown in the following analysis in the unbleached condition.

Sulphate
digestion
increases
yield

Ratio of
NaOH to
 Na_2S is
usually
3 to 1

Mineral Matter	1.68
Hydrocellulose (Soluble in alkali)	4.37
Cellulose	91.64
Lignin (by difference)	<u>2.31</u>
	100.00

In unbleached sulphite wood pulp, cellulose rarely reaches 89% and it frequently as low as 80%. The chief objection to the sulphate process is the odour of sulphuretted hydrogen given off when discharging digesters and the short life of its digesters which last about 10 years.

STEVENS ¹³ also mentioned the fact that bamboo, pulped by the sulphate process produces a fibre of highest quality, using 20% NaOH, and 7% Na₂S, cooking for six hours, at a pressure of 90# with 8 lbs of liquor to 1 lb. of bamboo. Unbleached and bleached yields were obtained of 50% and 44%. The bleached bamboo pulp is particularly adopted for the manufacture of book paper that requires a soft, well closed sheet, which is extraordinary opaque which bulks well to a high finish/

4. LIME PROCESS

This process, in which the raw material is digested with milk of lime, as applied to straw, especially in Holland, for the manufacture of a half-stuff,

suitable for the manufacture of straw boards and coarse packing paper.

A process of this kind as applied to bamboo is the subject of a patent (No. 14,871/1911) of McRae and Malcolmson.² In this method, the bamboo, having been previously prepared by cutting and crushing, is digested with water, containing lime in quantity varying between 5% and 25% of the dry weight of the raw material, for a period of 4-6 hours, depending on the steam pressure, the grade of material and other factors. The pressure employed may vary from ordinary atm pressure and 100 lbs. After the boiling operation, the product may be allowed to mellow by being left in contact with liquor for some days, and is then washed and disintegrated in some form of breaking machine. This process may be very inexpensive, but the product would consist of a brown pulp which would not be in condition for bleaching.

McRae and
Malcolmson's
patent

BLEACHING BAMBOO

In the patent granted to Turner and Maxwell,¹⁴ (Eng. Patent 6,277/1908) the method of preparing bamboo and the like for bleaching is modified to consist in steeping bamboo pulp in sea water or other suitable salt soln containing oxygen in soln, sulphuric acid being added. After washing, the pulp is again steeped in a weak alkali soln. The Patentees claim

Patented
process for
bleaching
bamboo

that what probably forms, when H_2SO_4 and other acids are added to soln of chlorides containing oxygen in soln, is an oxy-acid of chlorine which again liberates in contact with the fibre, and combines with the colored film or particle that is sought to be removed without free chlorine being produced. Pulp, after this treatment, can be bleached in a 2% soln of ordinary bleaching powder which causes no injury to the fibre.

III. Experimental Data from Mr. Mw. Raitt's Work⁴

(a) ANALYSIS OF BAMBOO

I. Analysis of Internodes and Nodes:- The proportional weight of nodes and internodes are first ascertained and the analysis sample made up of internodes and nodes in according therewith, the internodes being reduced to fine shavings and nodes to coarse filings.

% of whole	B. Polymorpha			C. Pergracile		
	I.N. 91	N. 9	Av. Whole	I.N. 94	N. 6	Av. Whole
Water Extract	8.70	9.83	8.80	7.96	9.07	8.03
Fat & Wax	1.04	1.40	1.07	.90	1.24	.92
Pectose	19.15	25.04	19.68	22.14	26.27	22.39
Lignin	15.29	17.60	15.50	15.10	16.72	15.20
Cellulose	<u>55.82</u> <u>100.00</u>	<u>46.13</u> <u>100.00</u>	<u>54.95</u> <u>100.00</u>	<u>53.90</u> <u>100.00</u>	<u>46.50</u> <u>100.00</u>	<u>53.46</u> <u>100.00</u>
Ash	3.87	4.50	3.92	2.50	4.20	2.60

Remark: The maximum yield of cellulose can only be obtained from bamboo which is not merely dry but is also seasoned. By seasoning, the starch which is in the secondary form, is oxidized by air and dispersed in the atmosphere. This reduces the water extract and consequently increases the percentage of cellulose.

II. Analysis of absolutely dry seasoned bamboo representing whole culm.

	W.E.	F.&W.	P.	L.	C.	Ash
Young culm, $\frac{3}{4}$ grown	13.10(a)	.75	24.48	5.62	56.05	2.61
B.Tulda, 1 yr	11.40	.90	22.09	13.33	52.28	2.72
Ditto 2 yr	7.86	1.01	24.33	13.80	53.00	2.56
B.Polymorpha, 1 yr	11.60	1.07	17.95	15.66	53.72	4.73
Ditto, 2 yrs	8.95	1.05	19.55	15.74	54.71	3.97
Ditto, 3 yrs	6.90	.95	21.57	15.96	54.62	2.18
B.Arundinacea, 1 yr	12.42	1.15	21.95	15.46	49.02	2.56
Ditto, 2 yrs	8.48	1.17	24.39	15.64	50.32	1.60
C.Pergracile, 1 yr	11.57	1.04	20.74	14.96	51.69	3.37
Ditto, 2 yrs	7.96	.92	23.09	15.30	52.73	2.57
Melocanna B., 1 yr	15.66	1.77	10.54	21.43	50.60	3.05
Ditto, 2 yrs	13.57	1.70	12.77	21.73	51.23	2.35
Bhabur Grass	9.08(b)	2.64	35.57	3.40	49.31	6.20
Wheat straw	7.79(c)	1.24	29.06	10.23	51.68	2.70
Spruce Wood	6.73(d)	3.02(e)	.00	35.76	54.49	.05

Note: (a) No solid starch
 (b) Colouring matter, soluble salts and traces of starch.
 (c) Colouring matter, soluble salts and no starch
 (d) Gum and Mucilage, no starch
 (e) Resin, no fat and wax.

- Remarks: 1. Lignification is practically complete within one year.
2. With increase of age, we have
- a. Reduction of water solubles
 - b. Increase of Pectous matter.
3. Crushing enables the use of mixture of bamboo of all ages.
4. Preliminary boiling in water before digestion removes all the starchy matter.

III. Apparent amount of NaOH required to effect the resolution of Fat and Wax, Pectose, and Lignin. Starch being previously extracted.⁴

	% of Pectose, F&W at .32 NaOH for each 1%	% lignin at .66 NaOH for each 1%
B. Tulda	25.34-8.11	13.89-9.11
B. Arundinacea	25.56-8.18	15.64-10.32
B. Polymorpha	20.60-6.59	15.74-10.39
C. Pergracile	24.01-7.68	15.30-10.10
Melocanna B.	14.47-4.63	21.73-13.34
	% NaOH on dry bamboo	% NaOH on N.A.D. bamboo plus .5 excess
B. Tulda	17.22	16.00
B. Arundinacea	18.50	17.15
B. Polymorpha	16.98	15.79
C. Pergracile	17.78	16.51
Melocanna B.	18.97	17.58

(b) RESULTS ON EXPERIMENTS OF DIGESTION OF BAMBOOBY SODA PROCESS

	B.Tulda	B.Arundin- acea	B.Poly- morpha	C.Pergra- cile	Meloca- nna B.
NaOH % TW	16 9.75	17.5 10.5	16 9.75	16.5 10	18 10.75
Initial Temp	170	177	177	177	177
Press.	100	120	120	120	120
Time(hr)	1	1	1	1	1
Subsequent Temp	162	162	162	162	162
Press	80	80	80	80	80
Time	4	4	4	4	4
Unbleached Yield	48.7	46.5	50.0	48.7	47
Bleaching Poder: a. % on Unb. Pulp	32	37	36	35	42
b. % on raw material	15.6	16.2	18	17	19.7
Bleached yield	43.5	42	44.5	43.5	42.5
UNIT COST	7.12	7.91	7.41	7.45	8.33

$$\text{Note: Tw} = \frac{\text{Sp.Gr} - 1000}{5}$$

Taking cost of Bamboo as 1, NaOH(n) will be 5 and B.P.(b) 8.33, and calling y as % bleached yield, we have

$$\text{Unit cost} = \frac{(B)(1) + (n)(5) + (b)(8.33)}{y}$$

Taking first data for illustration, we have

$$\text{Unit cost} = \frac{(100)(1) + (16)(5) + (15.6)(8.33)}{43.5} = 7.12$$

It has been found that by using excess NaOH in digestion, one will get better results at start, but a point will be soon reached when increase of NaOH will decrease the bleached yield considerably to upset the saving of bleaching cost. In order to find at what point one can get the best desirable results, the unit costs offer a good comparison and the soln for problem.

The following table illustrates this point.

% NaOH	Unb. Yield	B.P. (on raw Mat.)	B. Yield	Unit Cost
16	49	17.6	43.2	7.56
18	47	13.6	43	7.05
20	44	10.1	41.5	6.84
22	41	8.2	39	7.14

Thus, it is clearly shown from the comparison of the unit costs, the using of 20% NaOH in this particular is the most economical.

Best Digestion results with NaOH in excess of amount required for resolution of non-cellulose of crushed starch free bamboo. (temp. press. and time being the same as in previous cases)

NaOH %	B.T.	B.A.	B.P.	C.P.	M.B.
	19.5	21	19.5	20	21.5
Tw	11.5	12.5	11.5	12	13
Unb. Y.	44	43	45.5	44.5	43
B. P. a	23	25	25	23	27
b	10.1	10.8	11.4	10.2	11.6
B. Y.	42	40.8	43	42.3	41
Unit Cost	6.94	7.23	6.82	6.73	7.42

(c) DIGESTION RESULTS ON TREATMENT OF BAMBOO
WITH SULPHATE LIQUOR

Species of Bamboo:	B.T.	B.A.	B.P.	C.P.	M.B.
NaOH & Na ₂ S calc % as NaOH Tw	20.5 12	22 12.75	20.5 12	21 12.5	22.5 13.5
Unb. Yield	45	44	46	45	44
B.P. a.	16	17	17	15.5	18
b.	7.20	7.48	7.82	6.97	7.92
B. Yield	43	42	44	43	42
Unit Cost	5.88	6.22	5.85	5.87	6.36

B.P. a. = % on Unb. pulp
b. = % on raw material

The sulphate liquor consists of NaOH and Na₂S in proportion of 3 to 1. Temperature, pressure and time are same in previous cases.

Unit cost of NaOH plus Na₂S calc, as NaOH = 4.5

The superiority of Sulphate process is shown in this table where the unit costs are much lower than that resulted from the Soda treatment in the previous table.

IV. ORIGINAL DATE

I. Analysis of Bamboo.

1. Composition of the internodes.

No. of Runs	1	2	3	4	Av.
Moisture (a)	5.63*	5.56	5.63*	5.41	5.63
Water Extract(b) plus (a)	14.78*	14.81*			14.79
Pectose (c) plus (a) & (b)	30.92	30.31*	20.12*		30.22
Lignin, plus (a) (b) (c)	44.82*	53.36*	52.11*	55.36*	53.61
Cellulose	55.18	46.64*	47.89*	44.64*	46.39
Ash	1.15*	1.12*			1.13

2. Composition of the Nodes.

No. of Runs	1	2	3	4	Av.
Moisture (a)	6.16	5.86*	5.21*	5.32*	5.46
Water Extract(b) Plus (a)	13.16*	13.00*	15.16	13.01*	13.04
Pectose (c) Plus (a) & (b)	27.55	29.97*	29.98*		29.98
Lignin, plus (a), (b) & (c)	30.99	54.90*	56.17*	54.30*	55.12
Cellulose	69.01	45.10*	45.83*	45.70*	44.88
Ash	.90	1.04*	1.05*	.95	1.04

* Valuse from which the averages are taken.

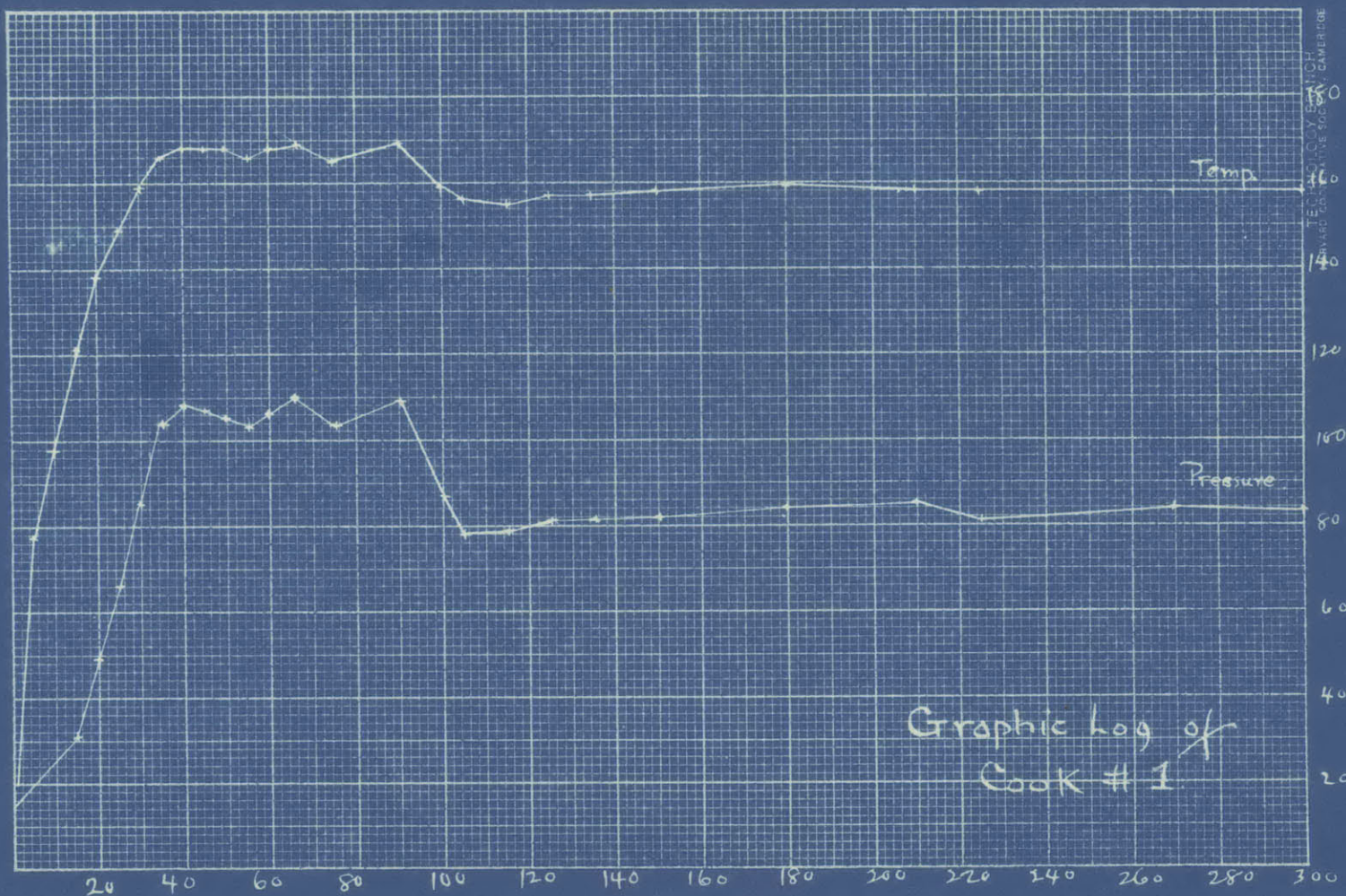
2 COOKING CONDITIONS AND RESULTS

No.Run	1	2*	3	4	5	6
N.A.D.Bamboo taken(gms)	670	470	343	350	350	319
NaOH added a.gms,	153	101	76	84	91	89.2
b.%	19.85	21.4	22.15	24	26	28
Conc.of Soln. gms/liter	38	50	50	50	50	50
Liters of soln taken	3.5	2	1.5	1.68	1.82	1.79
Time to reach max.Temp.&P. in Minutes	35	65	40	30	40	30
Maintenance at Max.Temp& P.	55	55	50	60	40	50
Time to reduce pressure	15		8	10	40	30
Maintenance at subsequent P.	195		187	200	150	170
Total time of Cooking in Min.	300	120	285	300	290	280
Max.Temp.°C	168	170	171	170	169	171
Max.Pressure lbs/sq in abs	108	109	110	109	108	110
Subsequent Temp.	158		156	155	156	156
Subsequent Pres.	82		77	75	76	78
% Total unbleached Yield	50.8	46.6	44.5	40.0	38.7	32.9
% Uncooked Chips	3.18		7.88	2.28	9.76	7.50
Residue NaOH in Gms.	30.79	24.20	22.37	23.30	33.60	29.22
% NaOH Consumption	15.25	16.10	15.60	17.35	16.40	18.80

* Cook not completed due to defect of apparatus.

3. BLEACHING CONDITIONS AND RESULTS

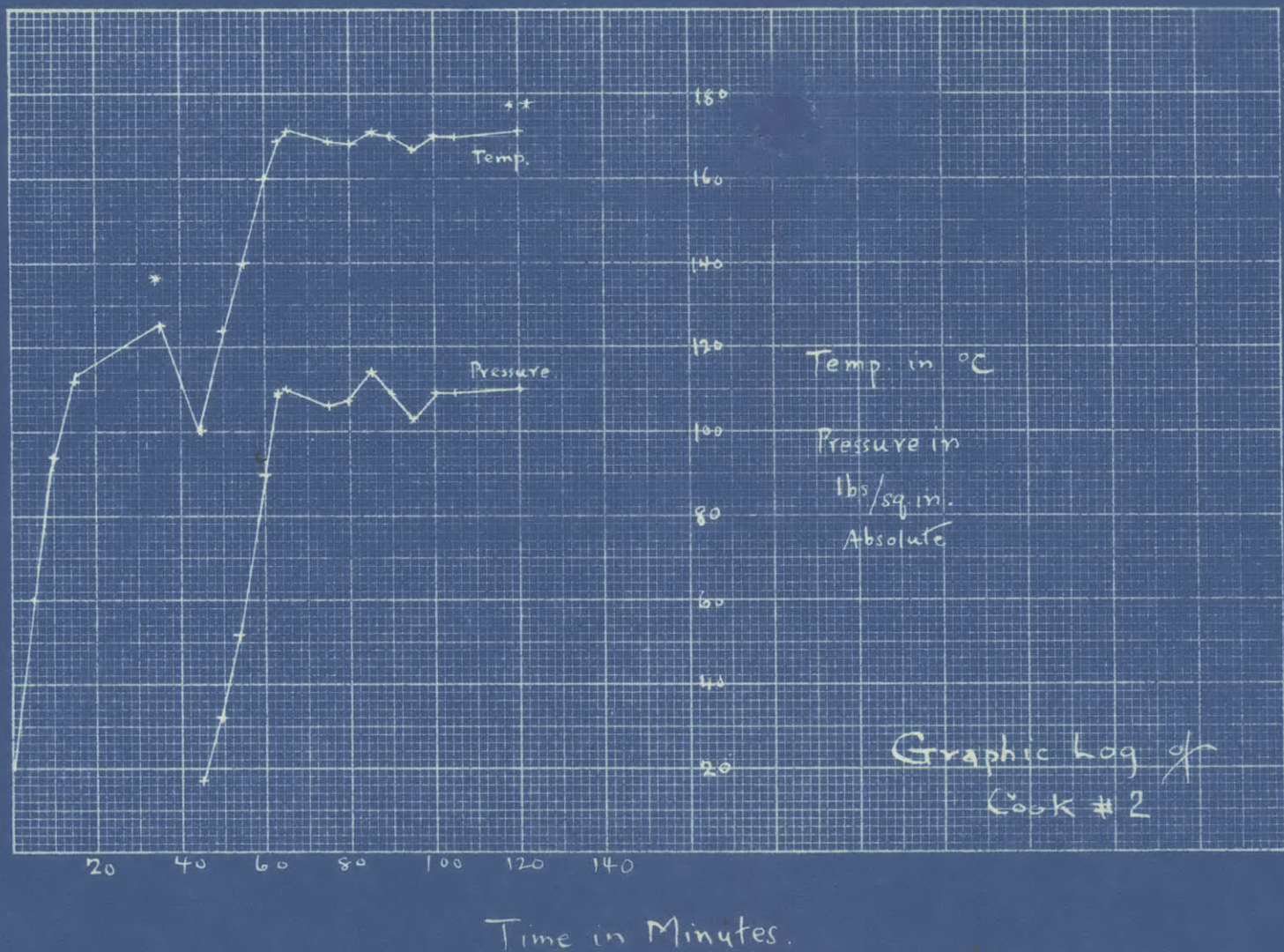
No. of Runs.	1	3	4	5	6
B.D.unbleached pulp taken	26	19.6	20	20	20
Gms Available Chlorine added	2.97	1.41	1.41	1.41	1.41
Volume of Total Soln.in liters	1	1	1	1	1
Temp.of Bath °C	35	35	35	35	35
Time of Bleaching in hrs,	1	1	1	1	1
Residue bleach in gms of available chlorine	.25	.03	.16	.14	.29
Bleach Consumption % of 35% B.powder on a.Unb.Pulp	29.90	20.10	17.85	18.12	15.95
b.Raw material	15.20	8.95	7.15	7.02	5.25
% Bleached yield on basis of raw material	46.10	39.90	36.40	37.50	31.40
% Loss due to Bleach on basis of raw material	4.7	4.6	3.6	1.2	1.5



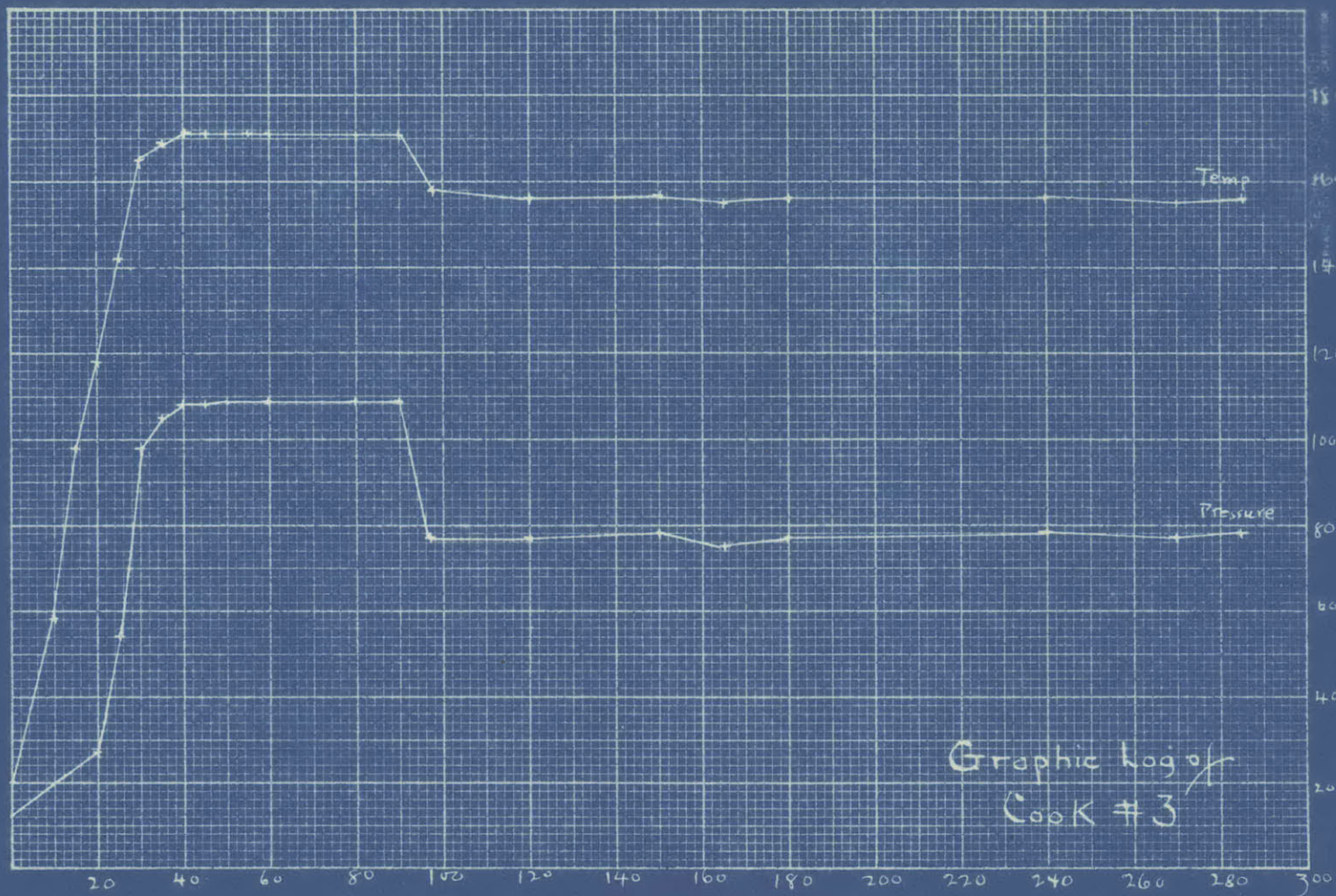
Time in Minutes.

Graphic Log of
Cook #1.

Temp. in °C
Pressure in
lbs/sq.in.
Absolute.

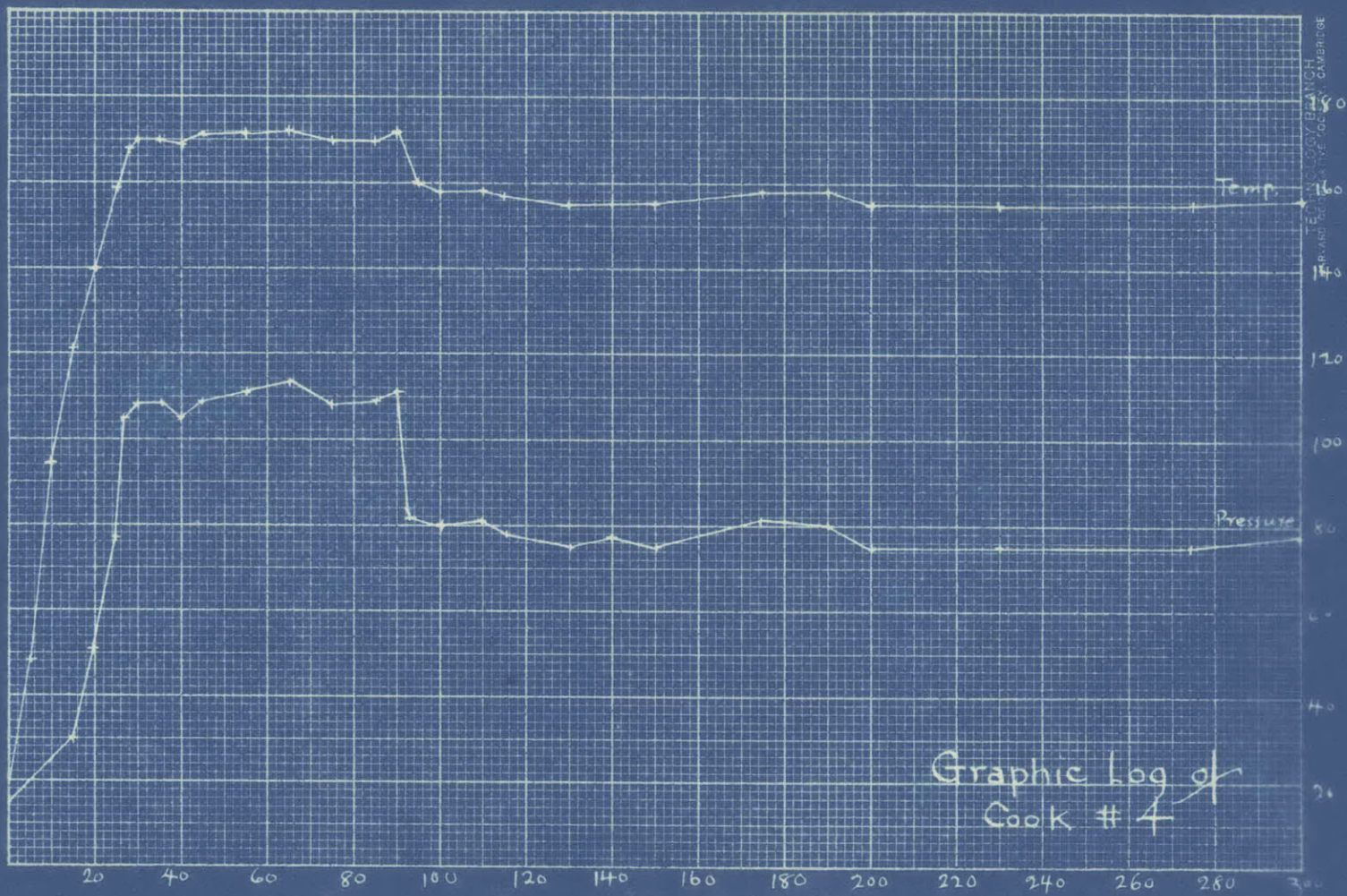


* Run interrupted to repair Gage.
 ** Run stopped due to leakage.

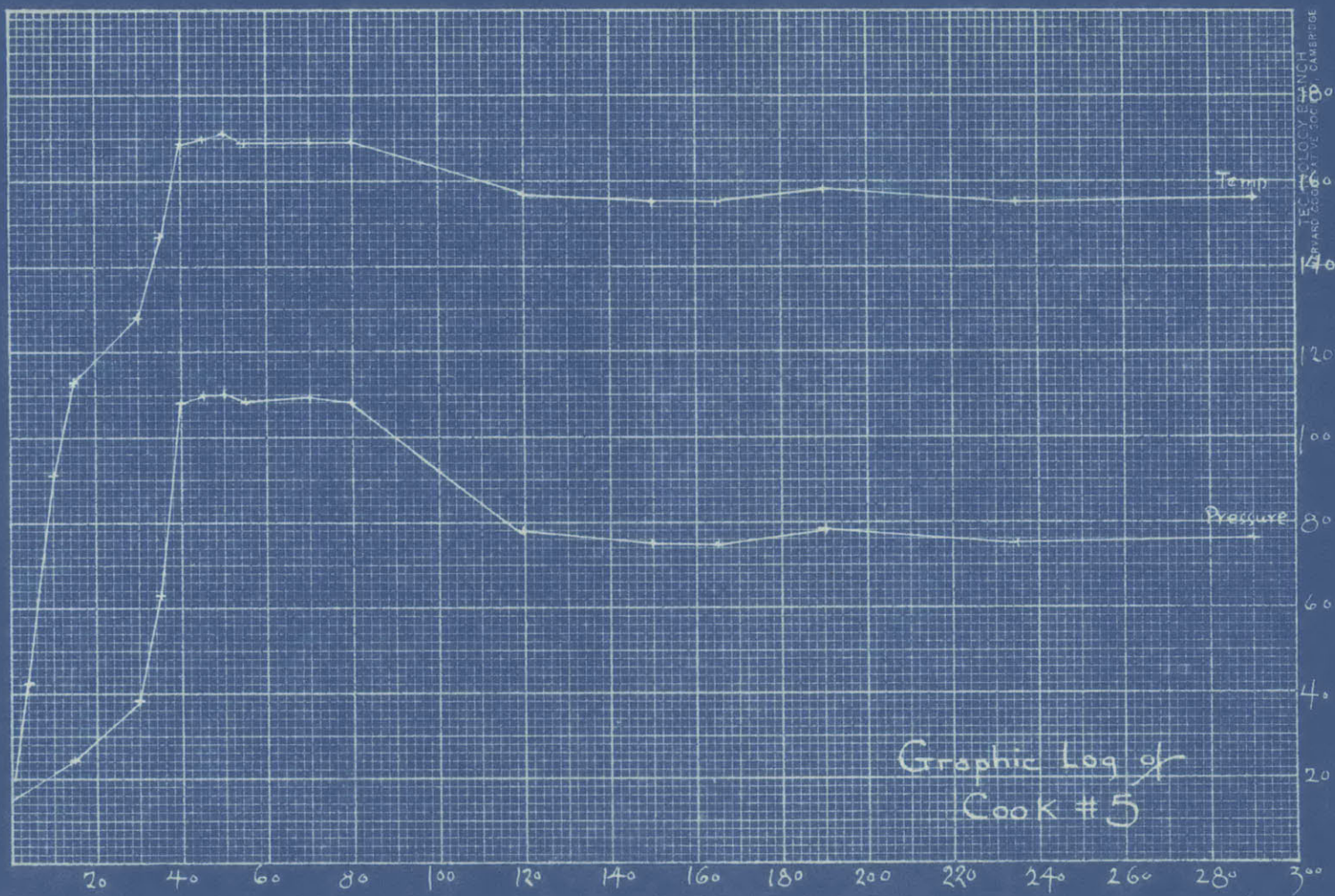


Temp. in °C
Pressure in
lbs/sq. in.
Absolute.

Time in Minutes.



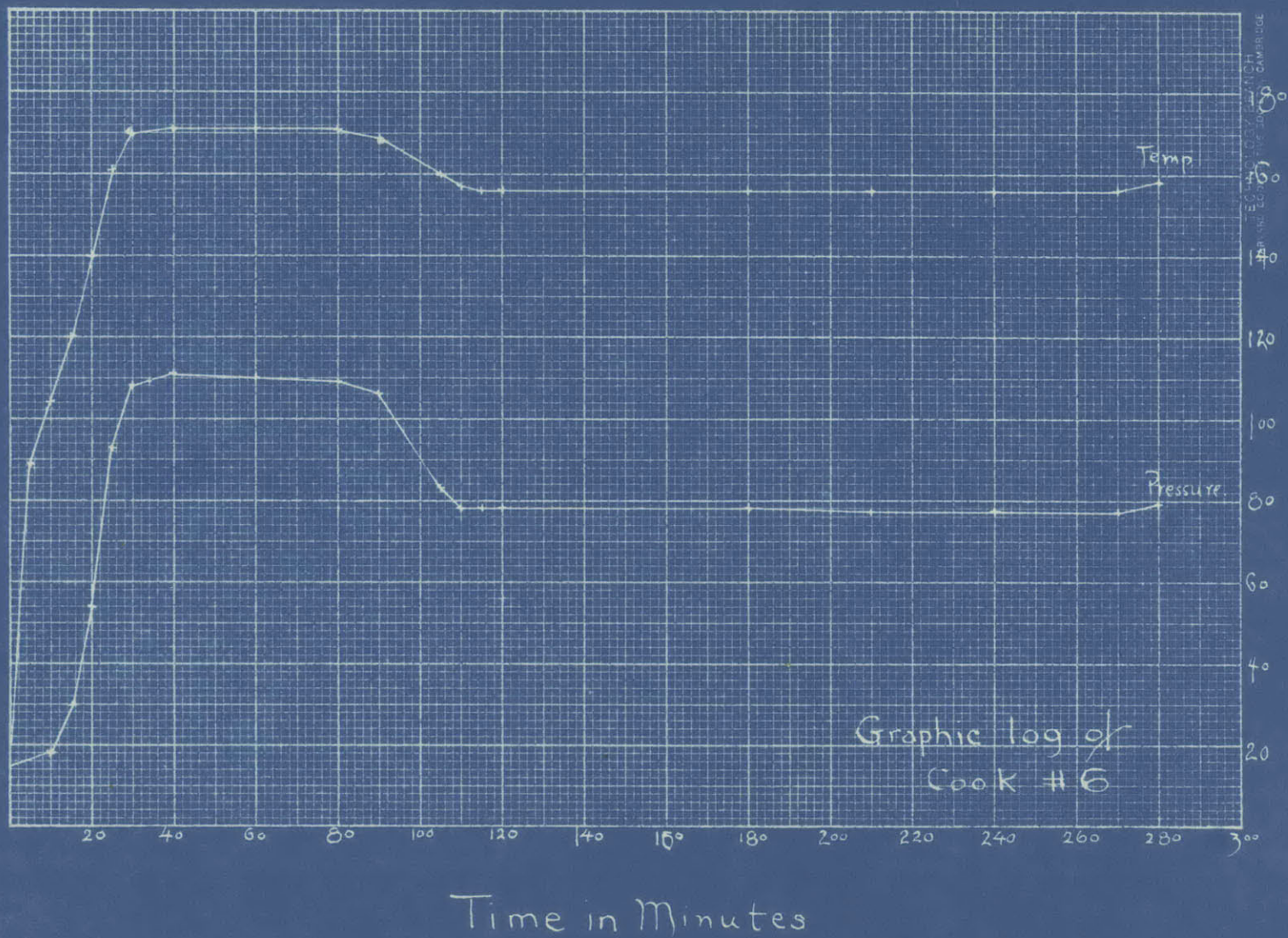
Temp. in °C
 Pressure in
 lbs/sq.in.
 Absolute.



Graphic Log of
 Cook #5

Time in Minutes.

Note: Cook interrupted during early part of Digestion because of leakage.





Sample Unbleached Pulp.



Sample Bleached Pulp.

Note: Both samples are from Cook #6. Some Shives are present in the samples because the pulp was not screened before the sheet was made.

V. Methods of Procedure.

a. Preparation of Standard solutions and reagents.

The standard hydrochloric acid solution was prepared by diluting a certain amount of concentrated acid with water, and the normality calculated from the ratio with the standard alkali solution which was standardized against potassium tetraoxalate. Acid Solution

The stock bleach solution was prepared by dissolving commercial bleaching powder in water, well stirred, and the clear solution decanted after settling. The bleach solution was kept in a glass bottle away from sunlight. The strength of this bleach solution was determined by analysis before use. bleach soln.

The Iodine solution was made by dissolving iodine and potassium iodide in water. The two chemicals were taken in rough proportion of their atomic and molecular weights in sufficient quantity to make the desired amount of solution and diluted with water to the approximate desired normality. The solution is standardized against pure As_2O_3 . Iodine soln.

The Arsenite solution was made by dissolving sodium arsenite in water with Na_2CO_3 added to make the solution neutral. The normality of this solution is calculated from its ratio with the standard Iodine solution. Arsenite Soln.

The stock Caustic Soda solution was made by dissolving the "Technical Granules" NaOH in water in ratio of about 1:1. The strength of this solution was analysed by titrating against a standard acid using phenolphthalein as an indicator. Stock NaOH Solution.

b. Method of Analyses.

To analyse for black liquor, 10 cc. sample was taken and diluted to 400 cc. 20 cc. of saturated $BaCl_2$ solution was then added, and the solution titrated against standard Black liquor

acid with constant stirring using a diluted solution of phenolphthalein on a spot plate as an indicator. The end is taken ^{point} when no pink color appears after a few drops of solution ~~are~~ mixed with the indicator for one minute. With diluted black liquor where the coloring matter is not so intense, the indicator can be added to the solution directly and fairly good end point can be obtained.

Analysis of available chlorine in the bleach solution was done by taking a 10 cc. sample from the stock solution, run in a large excess of standard sodium arsenite solution and titrate back with Iodine solution using starch solution as an indicator. The end point is taken when the blue coloration still exists after twenty seconds. As the end point in titrating Arsenite solution against Iodine is usually rather inaccurate, this arbitrary procedure insures uniform results. In analysing for residual bleach, 100 cc. was taken from the used bleach solution.

Available
Chlorine.

VI. Methods of Calculation.

In all analyses and cooks, normal air dry bamboo was used. So percentages are all figured on this basis unless otherwise stated.

In the analyses of bamboo, determinations other than moisture and ash are collective, that is, when we determine pectose, the loss of weight on treating with 1% NaOH represents not only pectose, but also water extracts and moisture. So, in order to get the percentage pectose, the percentage moisture and water extract as previously determined should be subtracted. It is plain that if a mistake be made with the determination of water extract, the accuracy of the Pectose determination will be thrown off. In getting the final data for analysis, results from different determinations of each constituent were averaged and other constituents subtracted in case they are included in the determination. To get the composition of the whole culm from the composition of the nodes and internodes by assuming 10% nodes to the whole culm, the calculation is simple. To illustrate, the pectose content in nodes is 16.94%, and in internodes, 15.43%. Therefore the pectose content of the whole culm is

$$(16.94)(.1) + (15.43)(.9) = 15.58\%$$

In obtaining the percentage crude yield, a small quantity of unbleached pulp is dried to constant weight. By this bone-dry factor, the dry weight of the entire material is calculated.

The percentage bleach consumption is calculated both on the basis of unbleached yield and raw material expressing the bleach as weight as weight of 35% bleaching Powder. From the grams of available Chlorine used, the percentage bleach consumption on the former basis is calculated by the following expression.

$$\frac{(\text{Gms of Chlorine}) (100)}{(\text{Gms of Unb. Pulp Taken}) (.35)} = \text{Percentage bleach consumption on Unb. yield.}$$

Calling this percentage \mathcal{Q} , the percentage on the basis of raw material is:

$$\frac{(\text{A}) (\% \text{ Crude Yield})}{(100)} = \% \text{ Bleach Consumption on raw material}$$

The percentage bleached yield is calculated as follows:

$$\frac{(\text{Wt. of Bleached Pulp}) (\% \text{ Crude Yield})}{\text{Wt. of Unbleached pulp taken}} = \% \text{ Bleached Yield.}$$

The loss due to bleach is the percentage crude yield minus the bleached yield.

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