Theoretical Approaches to Improvement of Pulp Yield by Sulphate Pulping.

ABANISH PANDA

INTRODUCTION

During the recent years the increase in the price of bamboo and other woody and nonwoody fibrous materials as a result of the growth of the paper industry as well as the non-availability of such raw materials has been felt by the paper industry. Although the cost of bamboo or the wood in India is not the dominating production cost, the trend is however upwards everywhere. It is therefore quite natural that both the industrialists and the technologists are getting interested in improving the pulp yield.

The term "pulp yield" may mean or be understood differently by different people. To the mill Management, which looks at pulping and paper-making as one, the pulp yield may mean the amount of finished paper of desired qualities made from every ton of fibrous material. It may also express the overall yield in terms of money gained for every ton of fibrous material. To the pulp technologist, however, the pulp yield is the weight percentage of pulp made from every tonne of chipped and screened fibrous material. Throughout this article the latter type of yield has been kept in mind.

APPROACHES OF INCREASING PULP YIELD

The major chemical components of chips are the cellulose, hemicellulose and the lignin. Lignin binds the fibres and the fibre bundles together. The rigidity of the chip is mainly due to the binding action of lignin. In order to prepare the chips suitable for sheet making the fibre bundles have to be thoroughly separated into relatively smaller bundles. This is done either by modifying the native lignin so that it loosses the adhesiveness or by partially removing the lignin by chemical pulping processes.

Kraft pulping system has a low yield. Many methods have been suggested and tried to improve pulp yield. Yield improvement is made either by limiting the degree of delignification or devising the methods of selective delignification techniques.

By limiting the degree of delignification, one face s the limitations of capacity of screen room and reject handling equipments as well as brightness requirements. For bleached pulp grades the higher demand and cost of bleaching chemicals balances the financial savings in yield.

The sulphate pulping process is a complex of topochemical reactions. The delignification process involves heterogeneous reactions due to the structural and chemical and chemical heterogeneity of the chip itself.

The structural heterogeneity of the chip offers difficulties in obtaining a uniform quality of pulp. Thus a greater amount of uncooked or partially cooked chips are produced if the conditions of the cook are not optimum. This means a partial utilisation of the chips and also a lower yield.

Similarly, when one looks to the chemical aspects of pulping, one notices a large amount of nonlignous matter, namely carbohydrates, being dissolved inadvertantly along with lignin. In case of bamboo, for example, although the total polysaccharides constitute about 60 percent on the weight of chips, one hardly gets unbleached pulp yield anything near that figure. If one assumes that the unbleached pulp yield of a modern pulp mill using bamboo is 45 to 47 percent (of which a part constitutes lignin), one has a material loss of 15 to 18 percent. The economical involvement of this lowering is quite noticeable.

The dissolved carbohydrates originate mostly from the hemicelluloses and partly from the celluloses. Only a small portion of the dissolved material is found as carbohydrates in the cooking liquor. The major portion of it is in form of low molecular acids. About two-third of the given alkali is thus consumed for neutralisation of these acids. If by some means the dissolution of carbohydrates can be minimised or prevented, an improvement of yield of pulp can be achieved.

The methods of improvement of yield can best be grouped into:

A. Choice of variables of conventional pulping method for highest yield.

B. Modification of conventional pulping method which would induce the carbohydrate stabilisation.

and

C. New pulping methods using non-conventional chemical additives.

A. VARIABLES OF CONVENTIONAL SULPHATE PULPING FOR HIGHEST YIELD

The factors affecting pulping are the following:

(1) Chemicals charged.

(2) Time-Temperature relationship of the cooking; and

(3) Chip dimension.

Although these factors have been classified into individual factors and an effort is made to spe-

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Dr. A. Panda, Orient Paper Mills Ltd., Brajrajnagar, Orissa.

Table I. Distribution of Alkali Consumed by Various Reactions in Bamboo Pulping.

	% Na2O on Chips
A. For neutralisation of acetic acid formed	1.0
B. For Hemi-cellulose reactions	8 — 10
C. For lignin dissolution	4 — 5

* 15% active alkali as Na_2O has been used on O. D. Chips

cify the role played by each factor, there exists a strong interrelationship between these factors.

(1) Effect of Chemicals Charged on Pulp Yield.

During the initial part of the cook, a major portion of alkali is consumed for breaking the hemicelluloses and neutralising the saccharinic acid and other acid products formed from the hemicelluloses.

The following table I gives the approximate percentages of alkali consumed by various reactions in bamboo pulping under specified cooking conditions. Thus nearly 70 to 75% of alkali is consumed by the hemicellulose reactions. Reactions with the hemicelluloses occur at about 100 °C or lower, while the delignification of technical significance starts at higher temperature.

In order to study the delignification process during the course of cooking of bamboo, chips were digested with 13% active alkali for different periods in an autoclave, the contents cooled in water and the lignin contents of the digested material determined the degree of delignification for the different periods have been calculated and plotted against the cooking period. It is seen that under the conditions of cooking studied, the delignification starts already at about 100 °C with bamboo (Fig. 1).

It is known that by increasing the chemicals charge of a cook the yield of pulp for a desired degree of delignification is reduced. The relation between Kappa number and the yield of pulp for different charges of alkali is shown in Fig. 2. With increased alkali charges the pentosan content of bamboo pulp decreases at equal degree of delignification. Table II shows a comparison between two cooks with 13% and 18% active alkali. For almost the same degree of delignification the pulp with higher alkali charge (18%) shows a lower yield and higher pulp brightness. The higher brightness is due to the higher concentration of active alkali at the end of the cook. The pentosan content of pulp cooked with higher alkali charge is less. The higher alkali charge removes thus more hemicelluloses during the cook. The yield loss in the cook with higher alkali charge is not only due to the greater removal of hemicellulose. If one calculates the lignin and pentosan-free pulp yield, one finds that cook with higher alkali charge shows a lower value. This is due to the greater degree of alkaline degradation (probably chain scission) of the pure cellulose. It has been shown that the lignin and glucuronoxylan in soft wood is disolved at a more rapid rate than other hemicelluloses and the glucomannan is practically unaffected by an increase in alkali charge (2a).

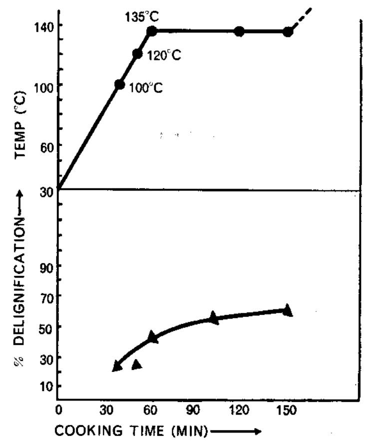

Fig. 1—Progress of delignification during cooking of bamboo with 13% A. A as Na_2O on O.D. chips of mill. The bath ratio was 1:2:5 & Sulphidity 20%

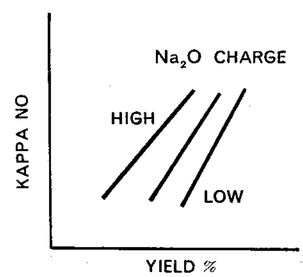

Fig. 2—Relation between pulp yield & Kappa No. of pulp of various alkali charges in wood chips.

Table II. Effect of Alkali Charge on Pulp Yield of Bamboo Chips

Active Alkali on chips, Na2O, %	13	18
Screened unbleached pulp yield%	40.9	38.3
Kappa Number of pulp	39.3	38.6
Ash-free lignin on pulp, %	3.7	3.8
Pentosans in pulp %	14.4	10.8
Pulp brightness, Elrepho, %	17.6	19.8
Lignin free pulp yield %	39.34	36.85
Pentosan-free pulp yield %	35.0	34.15
Lignin and Pentosan-free pulp yield %	33.44	32.71

Table III. Comparison of Cooks with Different Time-Temperature Schedules having same H-Factors*

Active Alkali on chips, Na2O, %	13	13
Chips/Liquor ratio	1:3.5	1:3.5
Top Temperature °C	170	170
Total time, Minutes	270	243
Screened pulp yield on chips, %	38.0	40.9
Rejects on chips %	0.8	0.6
Permanganate No. of pulp (25 ml. Tappi)	19.5	20.0
Ash-free Lignin, on pulp, %	3.3	3.7
Pentosans, % on pulp	13.2	14.4
Pulp Brightness, Elrepho, %	17.5	17.6

* For time-temperature schedule, see Fig. 3.

(2). Time-Temperature Relationship of the Cook and Pulp Yield.

In order to obtain a desired quality of pulp, the maximum temperature and the total cooking time are determined by the chip size, chemical concentration and the digester capacity in relation to the pulp production requirements. Once the maximum temperature and the liquor concentration are fixed, the time-temperature schedule can be varied for a given total cooking time. Naturally, the pulp yield is dependent on the time-temperature schedule. The influence of time and temperature is conveniently expressed in a single variable, which is termed as the "H-factor". The value of this factor is used as a means of predicting the yield and lignin content of the pulp (3). Regardless of the actual combinations of the time and temperature, cycles having equal H-factors are found to produce pulp of similar yield and lignin content.

In order to verify this generalisation with bamboo pulping, two cooks, with different time-temperature schedules but having the same H-factor, have been carried out. Table III shows a comparison of details of the cooking conditions, yields and pulp analyses of the two cooks. Although the pulp properties and compositions are nearly same for the two cooks, the pulp yield of one of the cooks is higher by about 1.9%, which is quite a significant difference. It can be cautioned that although the H-factor can be used as a guide for indication of yield, it should not be a parameter for accurate yield control and comparison. Actual cooking trials can not be dispensed with.

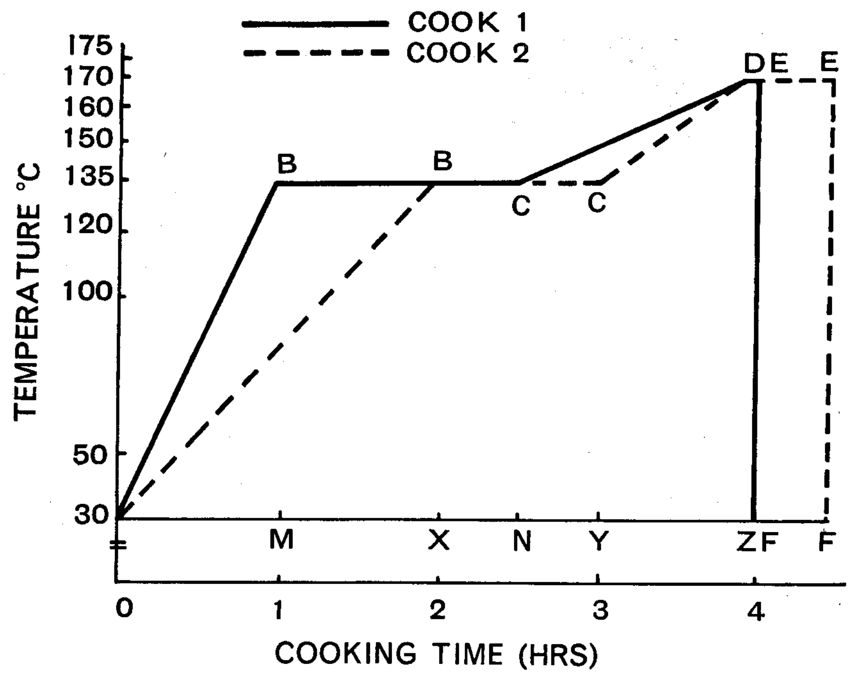

Fig-3-Time-Temperature schedule of two cooks with same H-Factors in bamboo cooking.

Another question which occurs to our mind is whether we should have a "straight-to-top" temperature cook or a two-stage period of temperature raising. Our experiments and experience indicate that the two-stage heating-up cook is preferable to the straight-to-top temperature cook especially for bleachable grade pulp. Pulps obtained by two-stage heating cooks are easy bleaching for the same lignin contents and therefore give higher bleached pulp yield for a given bleaching conditions.

(3) CHIP DIMENSION

Since chemical pulping is a complex of topochemical reactions, the dimensions of the chips have an appreciable influence on the rate and completeness of the pulping reactions. In order to prepare a reject free homogeneous pulp, it is necessary that the chips should be exposed to the chemicals so that delignification can proceed with increasing speed.

Also in kraft cooking the impregnation of liquor into chips is most essential. In chip impregnation two different phenomena play part. One is the penetration (flow of liquor) and the other is the diffusion (movement of chemicals). Penetration is dependent on pressure gradient and is relatively quick, while diffusion is dependent on concentration gradient and is slow. The chip capillarity determine penetration and the chip porosity the diffusion.

TABLE IV. Influence of Chip Thickness on Pulp Yield and Rejects and Screenings in Bamboo Sulphate Pulping, at various chemical charges.

Cook No.	1	2	3	4	5	6	7	8	9
Active Alkali Na_2O %	11	13	15	11	13	15	11	13	15
Chip thickness mm	2	2	2	4	4	4	8	8	8
Screened pulp yield %	51.5	47.9	42.9	46.8	48.4	46.4	29.1	31.1	32.2
Lignin free pulp yield %	44.0	44.6	40.7	42.6	45.3	44.3	26.5	28.6	29.2
Reject %	1.0	Nil	Nil	5.5	0.8	0.3	32.0	18.2	15.0
Pulp brightness, Elrepho, %	8.5	16.0	20.5	13.8	18.5	22.9	13.9	15.2	16.5

NOTES : Chips have been prepared in Laboratory with uniform length of 6 mm. The chip thicknes has been kept almost equal to 2, 4 and 8 mm respectively. Cooking has been carried out with a bath ratio of 1 : 3.5 using white liquor of 20 per cent sulphidity. Total cooking time in all cases was four hours and top temperature 170°C. Temperature raising was as follows: 1 hour to 135°C, 1½ hrs. to 170°C, as soon as temperature reached to 176°C digester was blown.

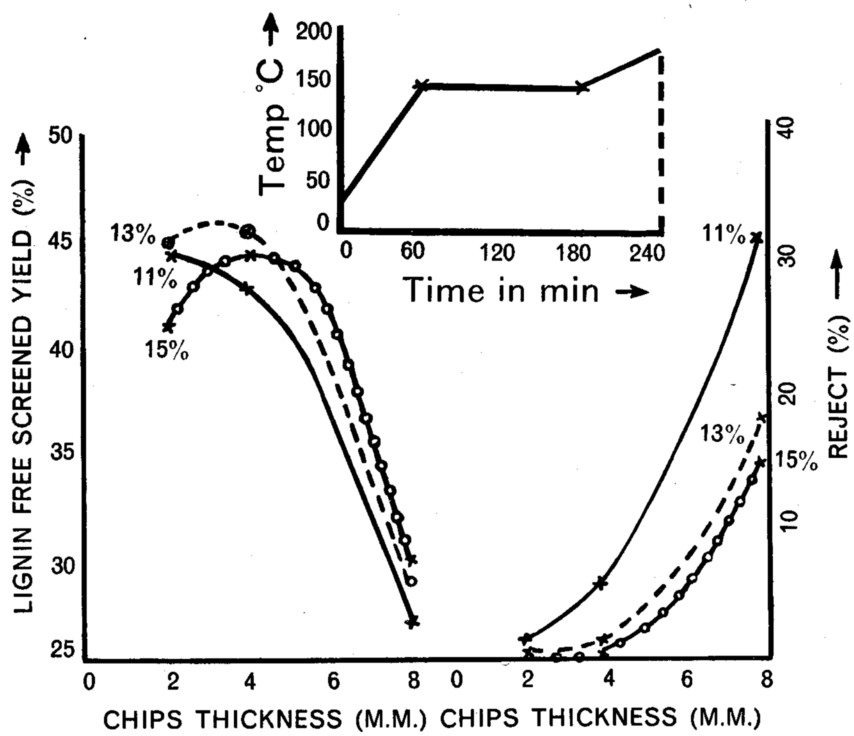

Fig. 4—Effect of chips thickness on the amount of reject and screening and pulp yield of bamboo.

Most of the chemicals transported by penetration are consumed for neutralising the carbohydrate degradation products. This takes place at the beginning of the cook at lower temperature. Chemicals required for delignification are predominantly transported by diffusion.

In short heating-up cook, delignification may start before the whole chip has been impregnated. Delignification would therefore occur in those part of the chip where chemicals are present.

Chemicals can be transported with least resistance through the shortest distance or the smallest dimension of the chip. The smallest dimension of the chip is the thickness of the chip. Hence thickness should determine the concentration gradient and hence the delignification of the chip.

As with wood, the chip thickness in bamboo is the most critical parameter for even delignification and better yield of pulp. Fig. 4 shows the effect of chip thickness on the rejects and screenings and the pulp yield. The yield figures in the graph are the lignin-free screened pulp yield. It is observed that thinner chips give higher yields upto a certain thickness below which the yield decreases again. For bamboo chips thickness of chips below about 3 mm have detrimental effect on the yield.

Further as the chemical charge is lowered, the amount of rejects increases with the thickness. The increases in amount of rejects is more evident as the chip thickneess increases and chemical charges decrease. With 11 % active alkali the rejects amount to as high as 32% of the original chip weight when the chip thickness is 8 mm. Thus by reducing the thickness of the chips, the yield is increased and the reject percentage decrease even at higher lignin content.

How then would one reduce the chip thickness? The conventional chippers are designed on the basis of chip length control. However, fortunately for us, there exists a fairly good relation between chip length and thickness. Shorter the chip, thinner it is. For bleachable grade pulp with conventional sulphate cooking the optimum chip thickness seems to be 3.0 — 3.5 mm. For this thickness the chip length must be 18 — 20 mm.

However, many mills have which produce longer chips. Should they invest on newer chippers, say for example, the parallel chippers for preparing chips? Should they have impregnation system outside the digestors? Or should they shred their normal chips prior to cooking?

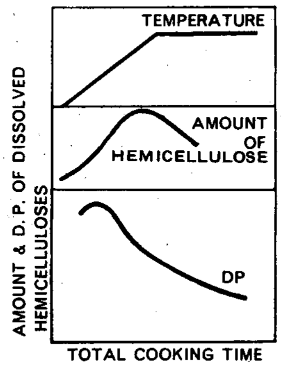

Fig. 5—Dissolution of hemicellulose & change of degree of polymerisation (D.P.) of hemicellulose during cooking of wood.

Let us look at the last alternative as the former two require bigger investments.

Chip Shredding

If the normal chips can be shredded to increase the surface area by longitudinal splitting along the length axis of the fibre without mechanical damage to the fibre so that no loss of strength properties occur, then one can increase the yield. However, it is difficult to have an ideal shredding operation in industrial practice.

In recent years shredding equipments of disc refiner type have been developed for industrial use. One such machine is the "Vertiflex" of Jones division of Beloit Corporation. Here the discs are fitted with concentric rows of triangular pyramid shaped teeth. The teeth are made of high grade hardened alloy steel. The life of such disc segments is 8 to 10 weeks.

It has been found in industrial trials that the so shredded chips can be pulped to a kappa number of 35—40. The yield increase is about one per cent more than with conventional chips with soft wood (4). It may be desirable to try such equipments with bamboo chips.

B. MODIFICATION OR CONVENTIONAL SULPHATE PULPING METHOD

(a) Recirculation Cooking (5)

During conventional kraft cooking a part of the hemicelluloses dissolves in the cooking liquor. The dissolution of hemicelluloses takes place throughout the digestion period at a steady rate. The dissolved hemicelluloses undergo degradation as well. Fig. 5 gives a qualitative picture of the amount of hemicelluloses dissolved in the cooking liquor as well as the degree of polymerisation (DP) of the dissolved hemicelluloses (5). One observes that the curve passes through a maximum followed by a decline. The rising of the curve indicates the increasing amount of hemicelluloses dissolving in the liquor. The decline can perhaps be explained by the fact that reabsorption of hemicelluloses takes place on the fibre wall of pulp when the pH of the cooking liquor decreases towards the end of the cook. (6,7).

The DP-Time curve shown in the same figure has also a maximum and then a decline. The point of maximum both for the amount of the hemicellulose dissolved and the DP depends on the cooking conditions and the raw materials.

If this loss of hemicelluloses into the liquor can be reduced, the pulp yield would naturally increase. How can it be carried out industrially? One method is to draw out the cooking liquor having the maximum hemicellulose concentration and then introduce it into the digester at the end of the cook, so that some of hemicelluloses can be reabsorbed on to the fibres. (8, 9). This would, of course, increase the unbleach-pulp yield, but on beating and especially on bleaching the loosely held hemicelluloses would be again lost, so that the net increase in yield of bleached pulp is negligible.

Another method is the so called "Recirculation cooking", where

Table V: Composition of Poysaoocharides in Bamboo

Sugars	% on Hollocellulose
Glucan	63.9 %
Xylan	23.9 %
Araban	2.0 %
Galactan	1.1 %
Mannan	0.5 %

Table VI: Cooking Conditions of a Normal Sulphate Cook and of a Hydrazine — Sulphate Cook of Bamboo

Hydrazine Hydrate on chips, %	Nil	20
Active alkali on chips, Na2O. %	14	7
Maximum Temperature, °C	165	165
Total time, hours	3½	3½
Screened Yield, %	49.5	51.8
Rejects and screenings, % on chips	2.7	3.6
Permanganate Number 40 ml. Tappi	24.1	25.2
Brightness of pulp, Elrepho, %	20.4	17.4

Other cooking conditions, which were same for both the cooks were:

Bath Ratio	1. : 3.5
Sulphidity of white liquor	20 %
Time to reach 135° C	1 hr.
At 135° C.	1 Hr.
To 165° C.	1 Hr.
At 165° C.	½ hr.

the free liquor, when the dissolved hemi cencentration and DP are high, is drawn off and the rest of the cook is carried out without free liquor. The liquor from the first or former cook is mixed with fresh white liquor and used in another cook and so on. By this technique the regained hemicelluloses are resistant to bleaching and beating and therefore the bleached as well as unbleached pulp yield increases. This technique has only been tried in pilot plant scale.

(b) **Methods Based on Stabilisation of Carbohydrates**

During sulphate kraft cooking alkaline degradation of celluloses and other poly-sachharides takes place. Acidic products are produced which consume alkali, thereby reducing the effectiveness of alkali for the delignification. reactions.

The carbohydrate chemistry has been very well developed and during the last decade our understanding of the mechanism of the alkaline degradation of polysaccharides has been clearer.

When cellulose is cooked with alkali, the former is attacked. This attack depends on the presence of a reducing group at the end of the polysaccharide chain. The reducing end group is transformed and separated from the chain as isosaccharinic acid. This takes place in a stepwise manner and is called the "peeling reaction" as if the chain is peeled off like an onion. This peeling off process goes on until a metsaccharinic acid end group is formed, which is stable against alkali. This reaction is called the "stopping reaction". Fig. 6 shows the peeling and stopping reactions as well as the alkaline hydrolysis of cellulose (10).

At temperature below about 150°C the loss of weight of carbohydrate is due mainly to the peeling reaction. At higher temperature hydrolysis of the glycosidic linkages also occur. This exposes new terminal end groups at which peeling reaction can start (11). If the cleavage takes place at a distance of about 6 anhydroglucose units from the end of the chain, the short oligosaccharide formed can dissolve in liquor directly. Roughly about 65 glucose units go into solution directly for each cleavage of the cellulose chain (12).

Due to economic interest many trials have been made to find out means of stabilising the polysaccharides against the attack of alkali. If the reducing end groups are modified, the peeling reaction would stop and there would be no loss of yield. Although many methods have been developed and tried the two following methods are of industrial importance:

(1) Polysulphide pulping (13, 14) where the reducing end group is selectively oxidised to carboxylic acid without affecting the rest of the molecule;

and

(2) Borohydride pulping (15) where the end aldehyde group is reduced to a primary alcohol. This chemical, due to its high price at the time, excludes its use in technical scale.

From the commercial point of view the polysulphide pulping process is more attractive and hence is dealt with briefly here.

(1) **Polysulphide Pulping .... .Process**

In polysulphide pulping, a liquor containing polysulphide ions is used for cooking. Polysulphide ions are formed when sufficient amount of sulphur is dissolved in a Sodium sulphide solution. Experiments both in pilot plant and industrial scales have shown that the polysulphide pulping method increases the pulp yield by about 8 to 10 per cent in case of spruce and pine and by about 5 to 6 per cent in case of birch wood more than those of the conventional sulphate process. In pine and spruce the yield increase is mainly due to the stabilisation of glucomannan, while in case of birch the yield increase is due mainly to the stabilisation of xylan (16, 17).

Could we expect to have an increase of yield in case of bamboo?

A paper chromatographic study of the bamboo polysaccharides shows that xylan constitutes 24 to 25% of the total sugars (Table V). Although no detailed study has been made on the structure of bamboo xylan, it has been suggested that it may probably exist as 4-0-methyl glucorono-araboxylan (1).

Preliminary experiments with bamboo chips have shown that a yield increase of 3 to 5% on weight of chips can be obtained by the polysulphide method. The polysulphide liquor has been prepared by air oxidation of a mixture of black and white liquor followed by sulphur additions. The method of preparation of the polysulphide liquor has been according to a method developed by the PFI, Oslo (18). The amount of sulphur added was 2.5% to 3% on chip basis.

Besides yield increase polysulphide pulps are easier to bleach and beat. They possess higher breaking length and burst, but lower tear and fold. One advantages of the poly pulps is the beating and refining qualities as comparable as to that of sulphite pulps, but still with strength properties comparable to those of sulphate pulps. The low tear value is shown by pulps of higher yield invariable. This is due to the lesser number of fibres present per unit weight of the pulp of higher yield.

C.—Pulping Methods using CHEMICAL ADDITIVES

There are many other chemicals which increase the pulp yield. Of these are the hydrazine and hydroxylamine most promising. It has been reported that with pine and spruce the yield increase has been upto 10% and with birch 2 to 4%. (19)

With bamboo the yield increased by only 2.5%. Table 6 shows the comparison of cooking conditions of a normal kraft cook and that of a hydrazine added kraft cook. It is remarkable that one could achieve a pulp brightness of 18% with only 7% of active alkali charge. The comparison has been made with a cook using 14% active alkali. It is also noticeable that chemical pulp of Kappa number of 38—39 can be obtained by charging alkali as low as 7%. It has probably been possible because carbohydrates have not consumed much alkali due possiby to the stabilisation of carbohydrates. The hydrazine also imparts some alkalinity.

The hydrazine pulps are slow to beating action and they show low tear as with all other high yield pulps. The hydrazine pulps improve sheet formation.

CONCLUSION

Pulping methods with chemical additives for use in commercial scale are prohibitive in cost at the present moment. They are at present of basic interest only.

Polysulphide pulping method has been accepted in industry both in Scandinavia and North America. The progress of acceptance has been slow mainly due to the high cost of chemicals recovery system and complicated liquor preparation methods. Further, corrosion of recovery equipments has been a set back.

Assuming the cost of bamboo per ton as about Rs. 120/- and an increase of bleached pulp yield of 5% by the polysulphide method over that of the conventional sulphate method (40% bleached yield) the saving comes to about Rs. 35/- per ton of bleached pulp. As the amount of sulphur used is 3% on the weight of chips, the yield increase would just be sufficient to pay for the cost of sulphur at the current price. However, there are other cost saving items such as reduced wood handling, higher digester capacity and lower steam consumption.

Yield improvement by reducing the chip dimension seems to be the most economical means of increasing the pulp yield, although the yield increase is not very spectacular.

Last and the most important but neglected factor is the good house keeping in the pulp mill itself. By proper choice of optimum cooking and bleaching conditions, well planned layout and selection of equipments and carefulness and alertness of personnel working in the plant, loss of pulp either through overflow or drain can be totally eliminated thereby preserving the yield, which means an improvement in itself.

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DISCUSSION

A. Panda

N. R. Jangalgi

What was the amount of total sulphur added?

A. Panda

1.0 to 1.5% sulphur as sulphur on based on O. D. Chips.

N. R. Jangalgi

In my view this appears to be very low.

N. S. Jaspal

Comments:

From the investigations on bamboo carried out by me at C C L, Stockholm, it was found that for a pulp of K. No. 25 the pulp yield improved by the redeposition of hemicellulose was 1% or less. Significant improvement in yield was observed when K. No. is 16 to 18 and not at higher value.

C. T. Dathatreya

Will you please elaborate the method adopted for determining the amount of Na_2O required by different reactions as given in Table I?

A. Panda

Starches of different molecular weights were used for determining the acetic acid and saccharinic acid formed, which give the amount of Na_2O required in these reactions. The difference between the total Na_2O consumption and that determined as above is used for lignin dissolution.