various tests employed in pulp & paper industry

A. B. GOPAL*

The economic stability and success of any industry not to speak of Paper Industry, depends mostly on the control that is brought to bear on the process or processes employed. This is all the more important in the case of Chemical & Allied industries, and so the knowledge of the methods employed for a quick evaluation of the intermediate and end products and their proper appreciation is of vital importance for the proper understanding of an Industrial process.

So far as the Indian Paper Industry is concerned, the methods of tests employed can broadly & classed into two. They are (1) those approved and followed as standard procedures by the Technical Association of the Pulp & Paper Industry (Tappi method) and (2) those laid down by the Indian Standards Institution, i.e. the I.S.I. method. For most of the methods that are at present followed for routine control of process, the Indian Paper Industry owes its allegiance to its counterpart in the western countries. Now that the Indian Pulp & Paper Technical Association (IPPTA) has come into being, we may now look forward to have integrated standard methods laid down by this organisation in collaboration with I.S.I., who will take into consideration the nature of our raw material, i.e., bamboo, bagasse etc. and other local conditions prevailing in India.

Let us deal with the various tests that are usually carried out to process bamboo pulp and paper.

I. Chipper House :

Bamboo & its Chipping :

Moisture : Bamboo, as it reaches the site, is sometimes in the semi-green, green or dry state. For purposes of payment, it is necessary to estimate the amount of moisture and volatile matter contained in it. This is usually done by taking a representative sample or samples from the arrivals and record its bonedry weight. This is done by drying the bamboo pieces in an electric oven at $105 \pm 2°C$ to constant weight,

Later, moisture in chipped bamboo as well as in the bamboo dust are also determined in the same manner. These are necessary for two purposes viz. (1) to calculate exactly the amount of chips in the digester charge after allowing for moisture and (2) to evaluate the losses in chipping by way of dust which may amount to 2-3% or more.

Digester House :

1. *Fresh Liquor Analysis* : From the point of view of digestion, the active pulping agents of the cooking liquor (usually called white liquor or fresh liquor) are sodium hydroxide and Sodium Sulfide, collectively called the Active Alkali, as the Sodium Carbonate, the remaining major component is known to be of little value for pulping. Hence, it is of vital importance to know the exact concentration of the above chemicals in the cooking liquor as well as in the Black Liquor that is usually added to the digester charge for make-up volume and for predigestion in the case of fractional digestions. For the same reason, the chemical ratio in the digestor is calculated on the basis of Active Alkali (Sodium hydroxide plus Sodium Sulfide).

Among the chemicals known to be specific towards their reaction with Lignin, the combination of Sodium hydroxide and Sodium Sulfide ranks second, after the Chlorine. Concentration of Sodium Sulfide in the cooking liquor exerts a beneficial effect in the process of pulping and has a direct bearing on the extent of delignification. In fact, the sodium sulphide is thought to act as a buffer by its slow hydrolysis into Sodium Hydroxide and Sodium Hydrosulfide, as the concentration of the original sodium hydroxide present decreases. The term "SULFIDITY" is defined as the ratio of concen-

*Chemist, J. K. Paper Mills, Jaykaypur.

tration of Sodium Sulfide to the concentration of the Active Alkali (Sodium hydoxide plus Sodium Sulfide) expressed on a percentage basis as Na_2O. Concentration of the various chemicals are expressed in terms of Na_2O equivalence in order that they may be compared easily. A knowledge of the Active Alkali concentration and the sulfidity of the cooking liquor is extremely essential before a digestion is commenced. Alkali variables in cooking liquor are determined analytically before the start of every digestion.

2. *Free Alkali in Black Liquor* :—Any free Alkali remaining in Black Liquor after cooking gives scope for its further utilisation either for makeup volume or for pre-digestion. It also improves washing & recovery operations. Cooking should proceed to an extent that there is some free Alkali left ; but the permissible limits of the amount of free Alkali in black liquor are governed by the considerations of Alkali losses, washing equipment and strength properties of pulp. Amount of Free Alkali varies from 3-4 g/c to 15-18 g/l. as NaOH in the Black Liquor from Mill to Mill and process to process.

(3) Having completed the digestion, it is necessary to assess the degree of cooking. This is ascertained by determining Permanganate Number of the pulp. This is a test, in which residual lignin in pulp is allowed to be oxidized by Pottassium Permanganate Solution of known strength under certain specified conditions. There are other methods like Kappa Number, Roe Number etc., based more or less on the same principle. But the Permanganate Number test being simple and easy is most widely carried out.

The Permanganate Number test is carried out usually at two stages, one just after the digestion in order to know the degree of pulping (delignification) achieved and again after the pulp has been washed and screened.

Brown Stock Washers : The concentration of solids in the Black Liquor is checked at regular intervals at each stage of brown stock washing to ensure proper washing efficiency. The Alkali lost in the pulp after final washing is also determined from time to time.

To achieve a satisfactory degree of washing by a counter-current washing system the black liquor that comes along with the pulp from the Digesters undergoes dilution usually to an extent that the solid content is decreased to nearly two-thirds of its original VALUE. The dilution factor is usually chosen by the dictates of the demand at Evaporators. An ideal figure quoted for sulfate pulp is 3.2. Too high a dilution factor is not desirable from the point of view of Alkali losses. The conditions chosen are intermediate between the two and dictated by practical considerations.

(4) *Screens* :—Efficiency of screening mostly depends upon the consistency of stock. These operational variables are checked from time to time to ensure that a minimum fibre loss takes place as rejects from the Secondary or Tertiary, as the case may be. It is necessary to keep a check on screening losses by ascertaining the fibre losses in Rejects. Sometimes long fibre passes along with the rejects in the centrifugal screens due to faulty consistency.

BLEACHING PLANT :

(a) *Chlorination* :

About 40 to 60% of the total Chlorine demand of the pulp is dosed as gaseous chlorine. To achieve maximum efficiency in this reaction, it is absolutely essential to maintain pH as low as possible preferably below 2. Chlorine in presence of water forms Hypochlorous Acid above a pH of 4 and the bleaching action of Hypochlorous acid is highly oxidative. There are chances of fibre degradation. The only check probably possible from practical and economic considerations is to guard against high pH and temperature of pulp in the chlorination tower.

(b) *Caustic Extraction* : Next stage in the system of Multistage bleaching usually caustic extraction. The chloro-lignins formed during the chlorination reaction are not soluble in water, but they easily dissolve in alkali. If they are allowed to be retained in the pulp, they render the pulp coloured and cause higher bleach consumption at later stage. Amount of caustic soda used for such extraction varies from 1—2% on the weight of pulp.

(c) *Hypo-Chlorite Bleaching* : Bleach liquor is prepared by passing Chlorine into a slurry of milk

of Lime (Calcium hydroxide) of about 20 g/c. available lime concentration till almost all of the milk of lime is converted to Hypo-Chlorite of Lime, the end point being usually determined by the flash test with phenolpthalein. A slight excess of lime is usually left in the Bleach Liquor to render it stable. Reaction that takes place may be stated as follows :—

(1) $2\ Ca(oH)_2 + 2\ Cl_2 —\ Ca(od)_2 + Cad_2 + 2H_2O$
(2) $Ca(od)_2 + Ca\ Cl_2 —\ 2Ca\ Cl\ o\ Cl$

The Bleach Liquor so prepared has a pH of about 11. As pH goes down and temperature increases, its stability decreases resulting in decomposition of Hypochlorite into Hypochlorous acid. The effect of pH during Hypochlorite bleahcing has been very extensively studied by various investigators. The results show that the rate of bleaching is low at higher pH range, although there is little degradation of pulp at higher pH ranges compared to neutral range. In fact, Hypochlorite bleaches in neutral range vary rapidly, but it degrades strength properties of pulp. Viscosity measurements being directly related to the Degree of Polymerisation, viscosity measurements are made to judge the extent of degradation of pulp. The most effective pH range suggested is 9%, whereas there is little improvement in strength properties above a pH of 9. pH during Hypochlorite bleaching is usually maintained by addition of sodium hydroxide to the extent of 0.25 to 1.0% according to the nature of pulp. pH decreases as the bleaching reaction proceeds, but the washing of pulp at pH of 8.5 to 9.0 is desirable.

PULP EVALUATION :

Various methods have been suggested and practised for the evaluation of pulp strength and quality. One of the common methods followed for routine control purposes is to beat the pulp in a standard equipment for beating (Jokro Mill, Lampen Mill or Valley Beater) at a known consistency for a fixed time and determine the freeness (rather slowness) achieved by the pulp by means of a freeness tester.

Another method is to beat the pulp at a known fixed *consistency* till a fixed freeness, say 40 to 45, is achieved, the time taken being an indication of the hardness or softness of pulp. The strength of pulp sheets made from the above pulp may further be determined as compared with the standard figures.

Another suggested practice is to determine the viscosity of pulp (either by the Cuprammonium method or the Cuprethylene Dianine) and correlate the viscosity to the degree of degradation of the pulp has undergone during bleaching.

Viscosity measurements are usually carried out on Rayon grade pulp, in which case the viscosity or the degree of polymerisation of the High L-Cellulose content pulp can be directly related to strength properties.

Yet another method of gaining popularity in the continental countries is to evaluate the water Retention value of a pulp. Water Retention value may be stated to be the water retaining capacity of the pulp by physico-chemical combination, which, in turn, depends on the extent of swelling of fibres and average fibre length of pulp. One of the fundamental requisites for a good quality paper pulp is that the fibres should have been properly swollen so that it easily acquires hydrating characteristics, that are so much necessary for the proper strength development. Also important is the necessity to retain the average fibre length of the pulp to the maximum extent during the digestion and bleaching operations. These properties are evaluated by determining the water-retention value of the Pulp. This is done by removing the surface moisture in a water soaked pulp suspension by means of a high speed centrifuge and determining the amount of water held by physico-chemical combination in the fibres after weighing the pulp before and after drying. The ratio of the amount of water held by such physico-chemical combination to the weight of dry pulp expressed as a percentage is the water retention value of the pulp. The above test, though simple, is an easy but accurate guide in assessing the strength properties of the pulp.

OVER BLEACHING OF PULP—METHOD OF ASSESSMENT

In commercial practice of bamboo pulp bleaching, most often it is rather difficult to maintain ideal

conditions of bleaching, as the importance is laid on achieving a higher brightness for the pulp. It is seldom possible to attain as high a *brightness as 80% without unduly degrading the pulp.* The result is that the pulp, though bleached to an 80% brightness range, has poor colour stability and causes the paper made from it to revert in brightness at a faster rate than an otherwise mildly bleached pulp.

This tendency of colour reversion is assessed by Copper Number tests. In this test, a known weight of pulp is allowed to reduce a standard alkaline solution of copper hydroxide for a fixed time and the amount of cuprous oxide formed by reduction is estimated and expressed on a percentage basis of the weight of pulp.

Another test to assess the colour stability of pulp is to evaluate the post colour value from Kubelka & Munk Numbers. Pulp sheets dried in air are kept in over at 105°C for 16 hours, after determining their initial brightness. During this agency test, the pulp undergoes reversion in colour to the extent of the reducing end groups present in the pulp and knowing the brightness before and after ageing, the post colour value is derived. Post colour values in the range of 2 are considered good indicating high colour stability whereas P.C. values in the range of 40 are considered highly undesirable.

STOCK PREPARATION & PAPER MACHINE :

The determination of Consistency (i.e. the amount of bone dry pulp present per 100 units of pulp) is highly important once the pulp reaches the stock preparation. It is on the basis of consistency in chests that the loading and other additives are added. Also equally important is the knowledge of consistency during beating action or refining in beaters. Refining treatment is to bring about gelatinous nature of pulp by hydration and cutting action. The pulp before beating is a free stock, meaning thereby the readiness with which it can be deprived of its water content. During the process of beating/refining, the fibres while being rubbed or brushed, undergoes such changes in characteristics that they are (1) fibrillated as a result of the peeling action and (2) undergoes hydration by virtue of which it acquires more and more affinity towards water, thereby making a 'slow' stock. The freeness or rather the 'slowness' of the pulp is measured by means of simple but ingenious device called the freeness tester, wherein a known weight of pulp in water suspension is allowed to drain through a 60 mesh wire gauze, the rate of drainage of water being measured by collecting the drain water through a fixed spout maintained at a *definite height* in the drainage pipe.

A check on the moisture content of paper web at the different stages of wet and dry ends of a paper machine often helps in tracing out the reasons of frequent breakages at the dry end. Presence of fines in large quantities in pulp often manifests as pick-up troubles. In such case, a check on the pulp quality by fibre classification reveals the trouble.

A proper classification of pulp at different stages of its processing is absolutely essential to assess its behaviour in paper making. It is done by a screen analysis of pulp with the help of a standard fibre classifier. This gives a good idea of the *difference of processing conditions of the pulp.*

Tests on Paper : Most of the tests carried out on paper are for ascertaining its suitability for the end uses for which they are manufactured. Some of the most common tests are as follows :

(1) *Bursting Strength* : Bursting strength of paper measures the degree of interfibre bonding which in turn depends on the degree of beating/refining and average fibre length of the Pulp used. This is measured by allowing the paper to burst under an increasing hydrostatic pressure applied at a fixed rate. The inter fibre bonding in the cross direction of paper being weaker than in the Machine direction, the break occurs along the cross direction at the limiting value which is noted and the Burst Factor for the Paper calculated.

(2) *Breaking Length* : The Breaking *length of paper may be defined as* that length of a paper strip of 15 mm. width which would break by its own weight when suspended freely. The breaking weight is determined by a suitable testing equipment. It, expressed in meters, is calculated as the ratio of the Breaking weight in Kg. to the mass per unit length (metre) of the strip in Kg.

(3) *Double Folds* : Folding endurance of a paper is of special importance especially in case when a paper is required to withstand folding during its handling. This is measured by allowing a 15 mm. strip of paper kept in tact under a definite tension of 1 kg. to be folded both ways and recording the number of double folds it can withstand before it snaps.

(4) *Tear* : Tearing strength of paper measures the amount of *Shearing force* required to separate the inter-fibre bonding and tear a specimen of paper. This, in turn, mostly depends on the average fibre length of pulp used. The tearing strength is measured by means of the Tear tester, in which a small definite size of paper sample is clamped and allowed to be torn by a shearing force, the force being indicated on a quadrant scale attached to the instrument.

(5) *Cobb test* : Water absorption characteristics of paper are compared by the values of 1 minute cobb test, in which a definite volume of water (100 ml. distilled water) is allowed to be in contact with a definite area (100 sq. cm.) of the specimen for 1 minute duration as measured by gentle blotting and the gain in weight of the specimen noted and expressed on a percentage basis.

(6) *Wax Pick* : A series of waxes standardised for their picking qualities and designated with numbers by the Dennison Co. of Canada, are used for assessing the printability of paper or boards. One of the requisites for a good quality printable paper or boards is that the layers should have a good degree of adhesion and should not peel off during printing. The standardised waxes with their specific picking properties are melted and held on the paper sample till it is cool (say 15 minutes), after which they are lifted from the paper surface vertically and the minimum number of the wax from which the pick starts is noted. The result is generally expressed as the maximum number that would give no pick with the paper.

(7) *Sizing* : Sizing materials are added in paper to improve its writing properties, opacity etc. The degree of sizing is generally assessed by noting the time taken for the colour reaction of two reagents, like Ammonium Thiocynate and Ferric Chloride or Potassium Ferrocynide and Ferric Chloride kept in contact with both the surfaces.

(8) *Ash in Paper* : Ash percentage in paper is determined to assess the extent of loading materials given in the stock and retained in the paper.