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Sixth Cess Training Programme

on

"Calibration & Testing Methods"

at

CPPRI, Saharanpur

July 22 - 26, 2006

Sponsored by

Development Council For Pulp Paper & Allied Industry

Ministry of commerce & Industry
Govt. of India

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ANSI-RAB QMS ACCREDITED

केन्द्रीय लुग्दी एवं कागज अनुसंधान संस्थान
CENTRAL PULP & PAPER RESEARCH INSTITUTE
AN ISO 9001:2000 INSTITUTION
In pursuit of cleaner production, resource conservation & quality excellence

केन्द्रीय लुग्दी एवं कागज अनुसंधान संस्थान
Central Pulp & Paper Research Institute
AN ISO 9001:2000 INSTITUTION
In pursuit of cleaner production, resource conservation & quality excellence
An autonomous organisation registered under societies Act under the administrative control of the Ministry of Commerce & Industry (Govt. of India)

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Dr. A. G. KULKARNI
Director

PREFACE

Under the present scenario of trade liberalization in the country, Indian paper Industry is required to be globally competitive. There is a need to explore innovative means to reduce costs while improving environmental performance, safety and efficiency of mills and ultimately of quality of products. Today, quality of the end product has become one of the most important factors. Evaluation of quality is closely linked with the proper calibration of the testing equipments/ instruments. In Europe, organizations/ institutes like Finish Pulp & Paper Research Institute, Helsinki & PIRA in UK, etc. have been providing calibration service to the pulp and paper mills in their countries for quite long time. The calibration service helps the pulp and paper mills to check the performance of the testing equipments/ instruments so that the desired quality parameters of the raw materials and end products are ensured. CPPRI has also made concerted efforts in this area and established **Intra Laboratory Quality Assessment Service** between the Institute and Pulp & Paper mills.

Keeping in view the changing requirements of the Indian Paper Industry, **"Calibration & Testing Methods"** has been chosen the topic for the Sixth Cess training programme under the Cess funded scheme "Continuing Education for Technical Personnel of Pulp & Paper Industry". The training programme is scheduled for July 22-26, 2006 and will be organized at CPPRI, Saharanpur. The programme will be attended by 32 participants representing 18 mills / Govt. organizations from all over the country. Eminent faculty from CPPRI will share their wide experience with the participants of the training programme. Participants will also be exposed to practical demonstration of calibration of testing equipments/ instruments & various testing methods in CPPRI's laboratories.

I wish this training programme a success.

(A.G. Kulkarni)

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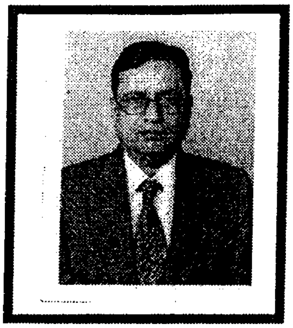
CHARACTERISATION OF CONTAMINANTS IN RECYCLED FIBRE

PRESENT STATUS OF THE PAPER INDUSTRY



Dr. A. G. Kulkarni,
Director, CPPRI

About The Author

Dr. A.G. Kulkarni, Director Central Pulp & Paper Research Institute (CPPRI) Saharanpur, Uttar Pradesh, India has been with the CPPRI since its inception. He holds a Master degree in Chemistry & Doctorate in Black Liquor and Lignin Chemistry.

Dr. Kulkarni has pioneered the research work on Desilication of black liquor with eventual development of mill scale plant, installed at Hindustan Newsprint Ltd., Kerala and High Rate Bio-methanation of black liquor rich effluent and a mill scale unit is successfully operating at Satia Paper Mills is another achievement of Dr. Kulkarni. His contribution in the area of physico chemical & thermal properties of agro - residue non-wood black liquors has now made it possible to process this liquor in chemical recovery boilers. He has published more than 300 scientific papers in Indian and International journals. He is widely traveled in Europe, S. E. Asia, and Australia and has been on several foreign missions as UNDP/UNIDO Consultant.

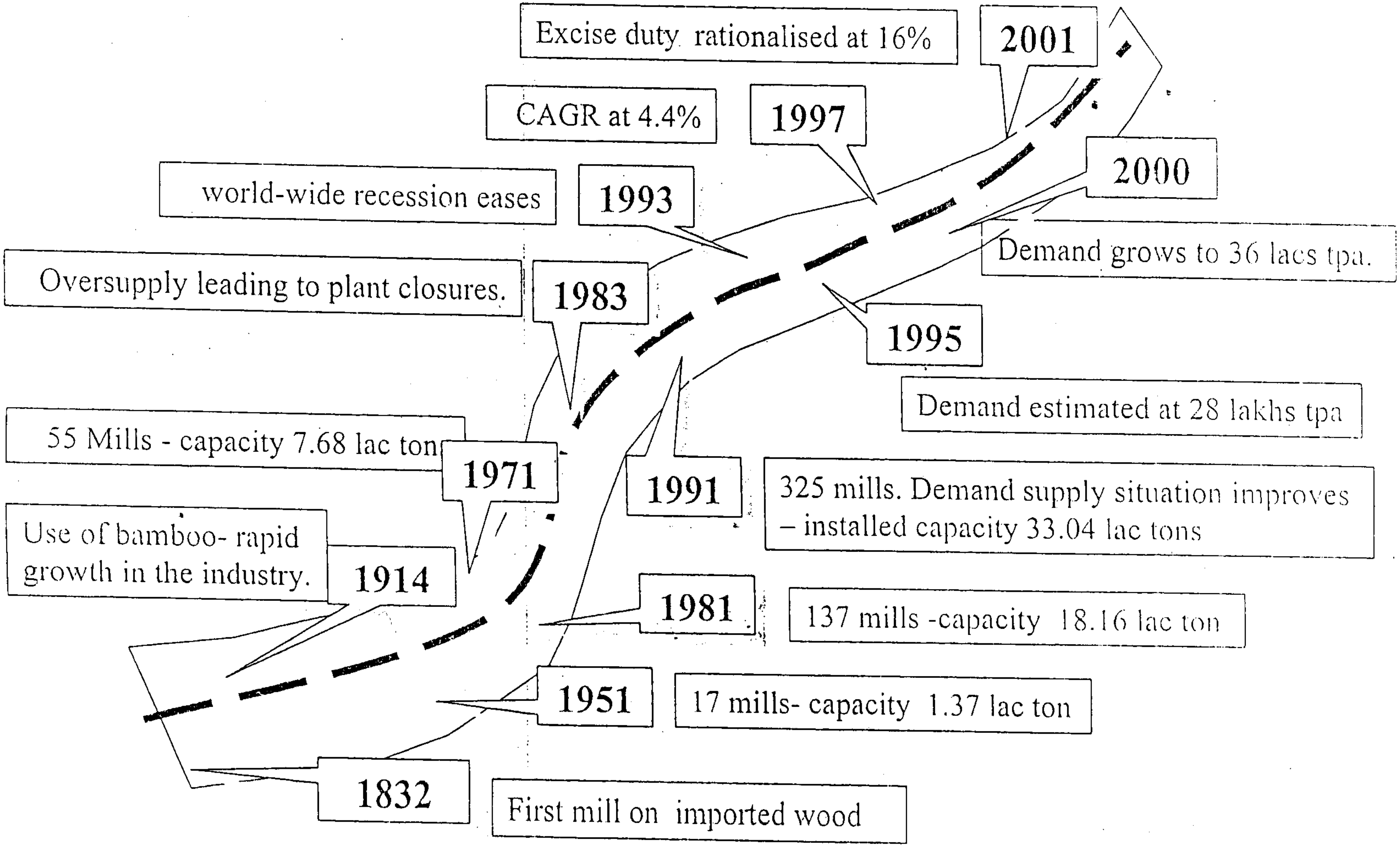
His areas of specialization include pulping and bleaching, black liquor-its chemistry & processing, environment and energy management. Dr. Kulkarni holds several patents-important ones being on desilication of black liquor, thermal treatment of black liquors and Direct Alkali Recovery System etc. He is a member of several National and International Scientific & Technical organizations and also on board of Directors of Paper Mills & Research organizations

PRESENT STATUS OF THE PAPER INDUSTRY

A.G. Kulkarni

The article discusses the chronological developments in Indian Paper Industry in terms of its growth, production level and levels of capacity utilization. The information also provides the technological status of our industry in terms of trends available on raw material and basic input requirement along with the cost involved. The technological development in pulping and bleaching, paper machine, and also focus on the energy and environment in various categories of the mills, has been addressed.

Indian Paper Industry : The Road Map from 1832 to 2001



Excise duty rationalised at 16%

2001

CAGR at 4.4%

1997

2000

world-wide recession eases

1993

Demand grows to 36 lacs tpa.

Oversupply leading to plant closures.

1983

1995

Demand estimated at 28 lakhs tpa

55 Mills - capacity 7.68 lac ton

1971

1991

325 mills. Demand supply situation improves – installed capacity 33.04 lac tons

Use of bamboo- rapid growth in the industry.

1914

1981

137 mills -capacity 18.16 lac ton

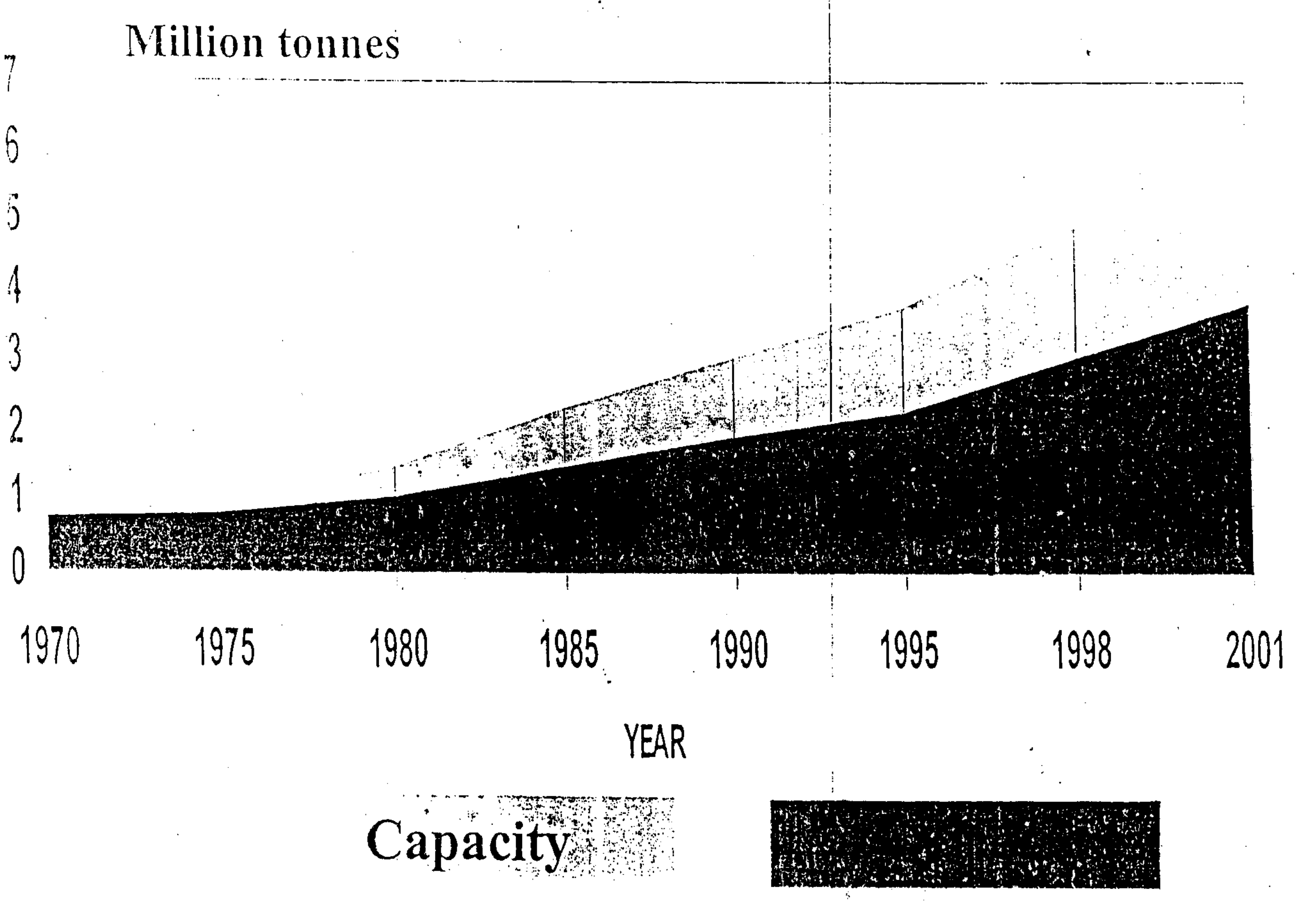
1951

17 mills- capacity 1.37 lac ton

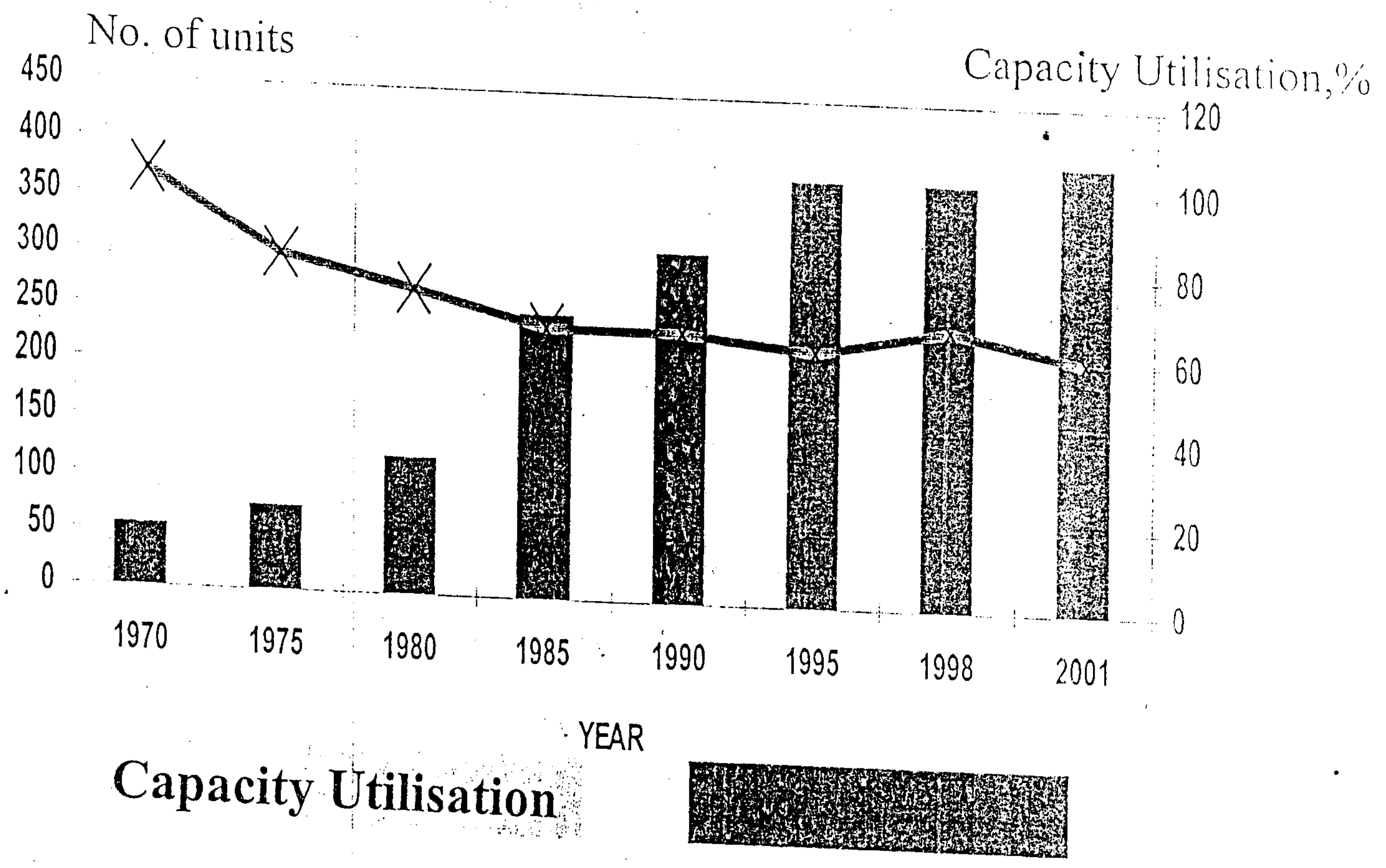
1832

First mill on imported wood

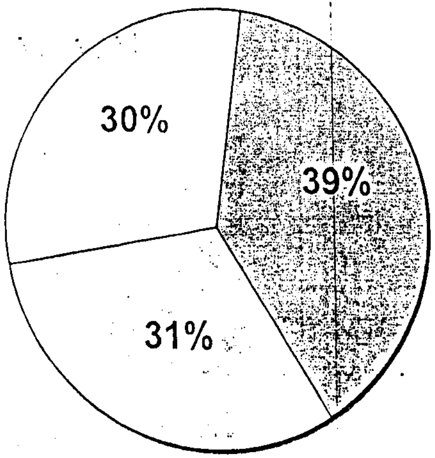
Growth of Indian Paper Industry Installed Capacity & Production



GROWTH OF PAPER INDUSTRY - CAPACITY UTILIZATION & NUMBERS

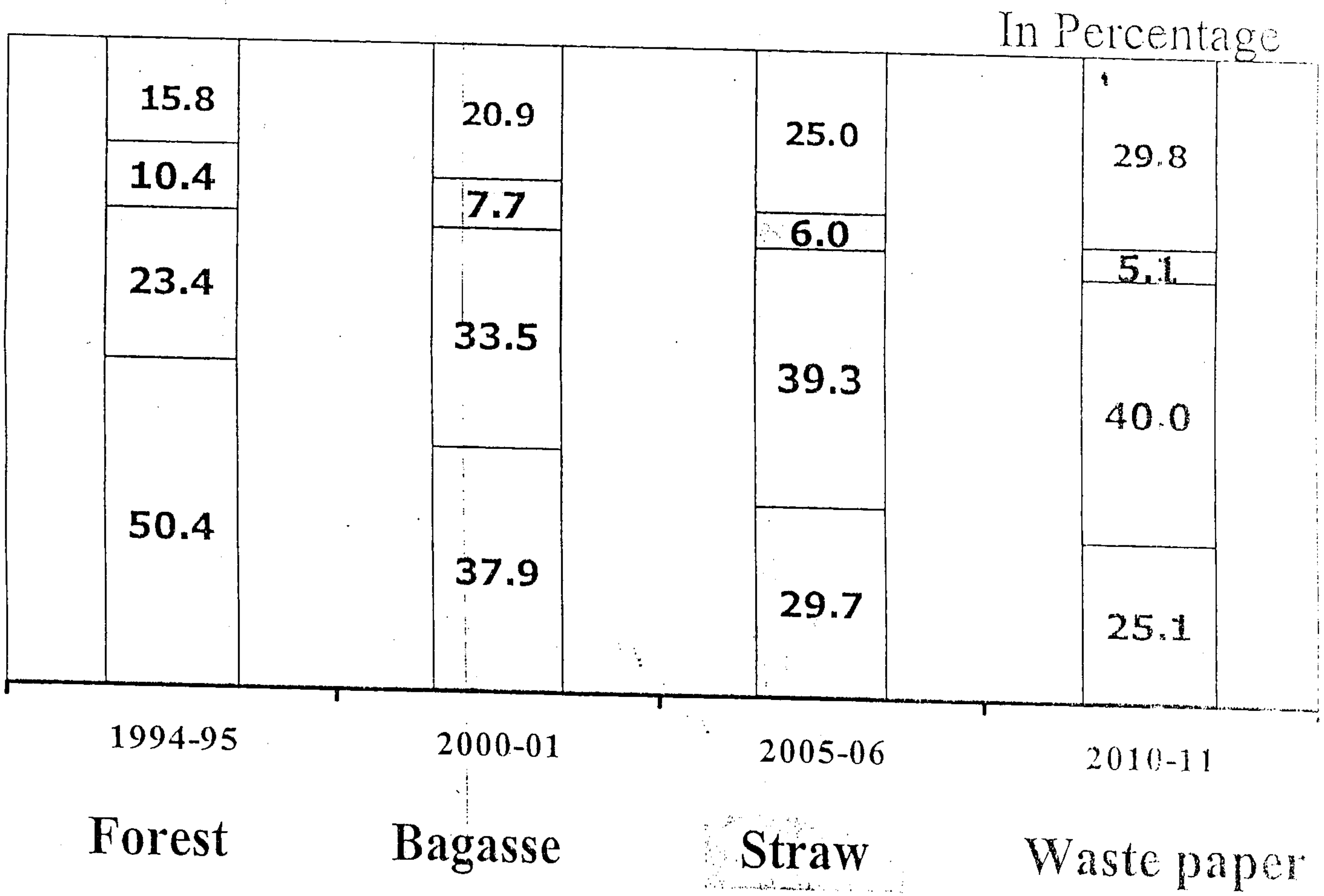


Indian Paper Industry : It's Structure

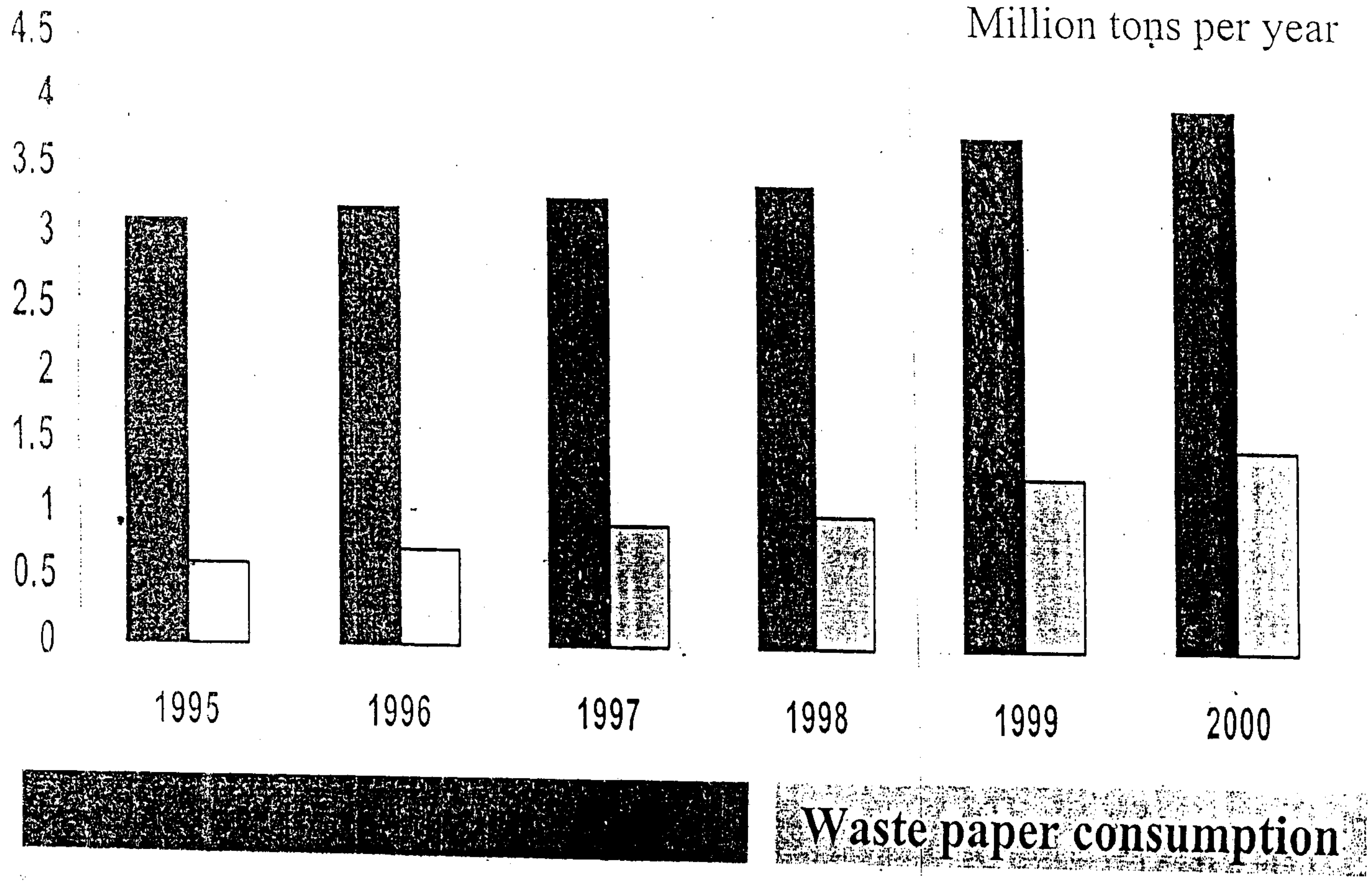


Data	Sector			Total
	Forest based	Agro Based	Recovered Paper	
No.of Mills	28	131	241	400
Installed, Capacity Mn tons/yr.	2.4	2.1	1.9	6.4

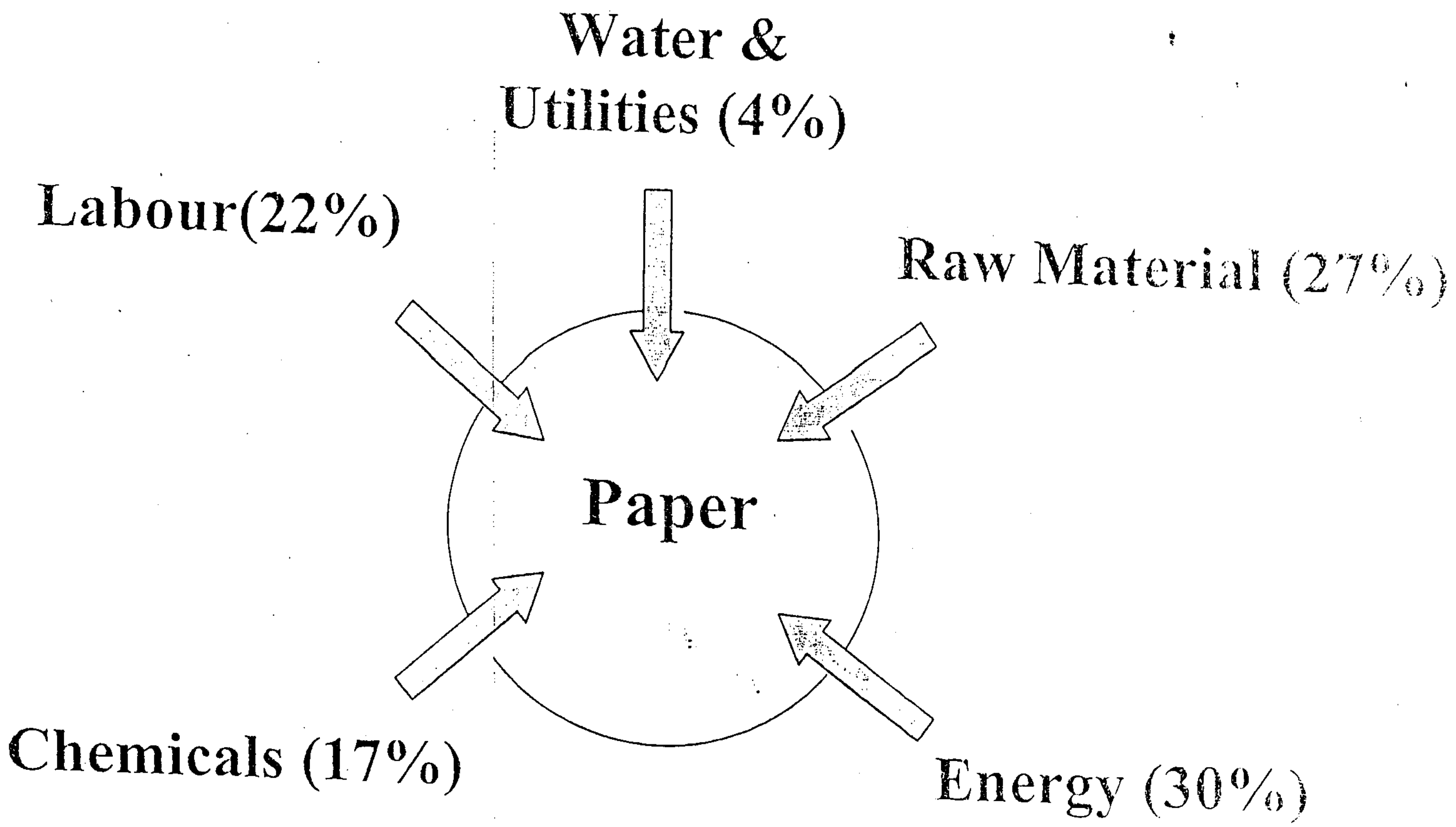
Paper Production from Fibrous Raw Materials



Share of Waste Paper in Total P & B Production (1995 - 2000)



Basic Inputs



Water & Utilities (4%)

Labour(22%)

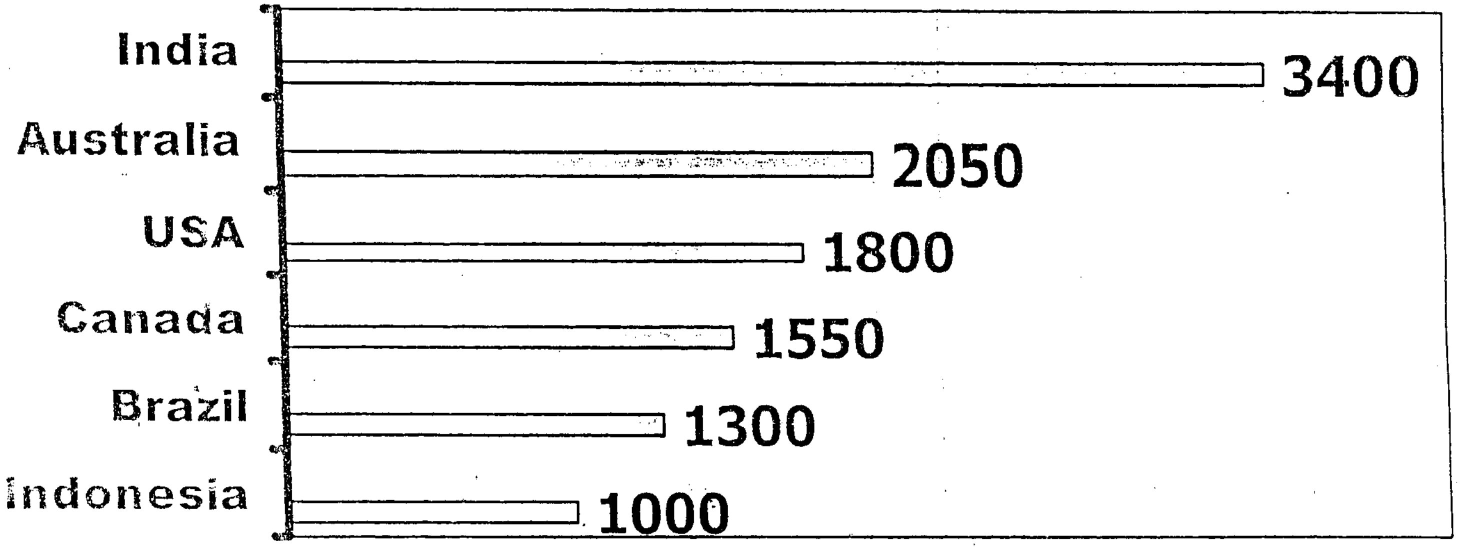
Raw Material (27%)

Paper

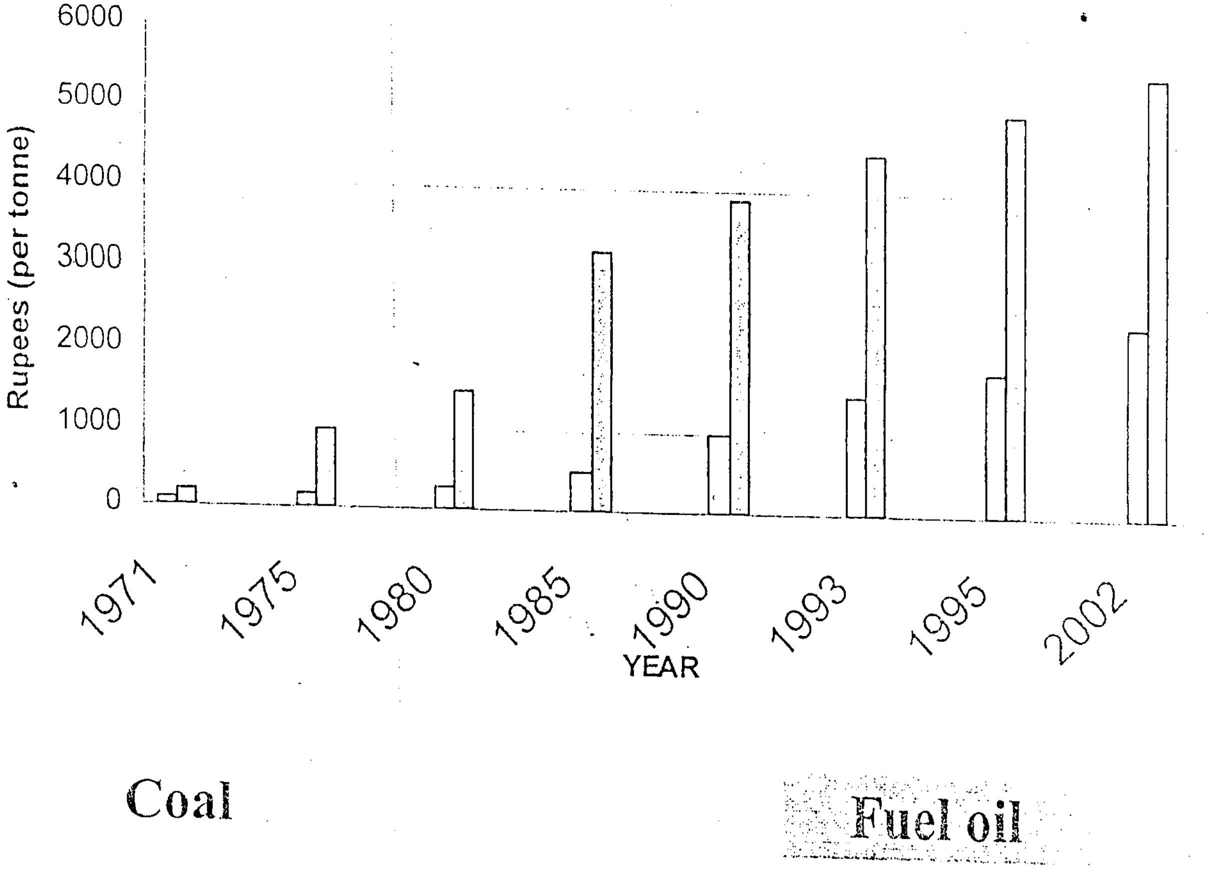
Chemicals (17%)

Energy (30%)

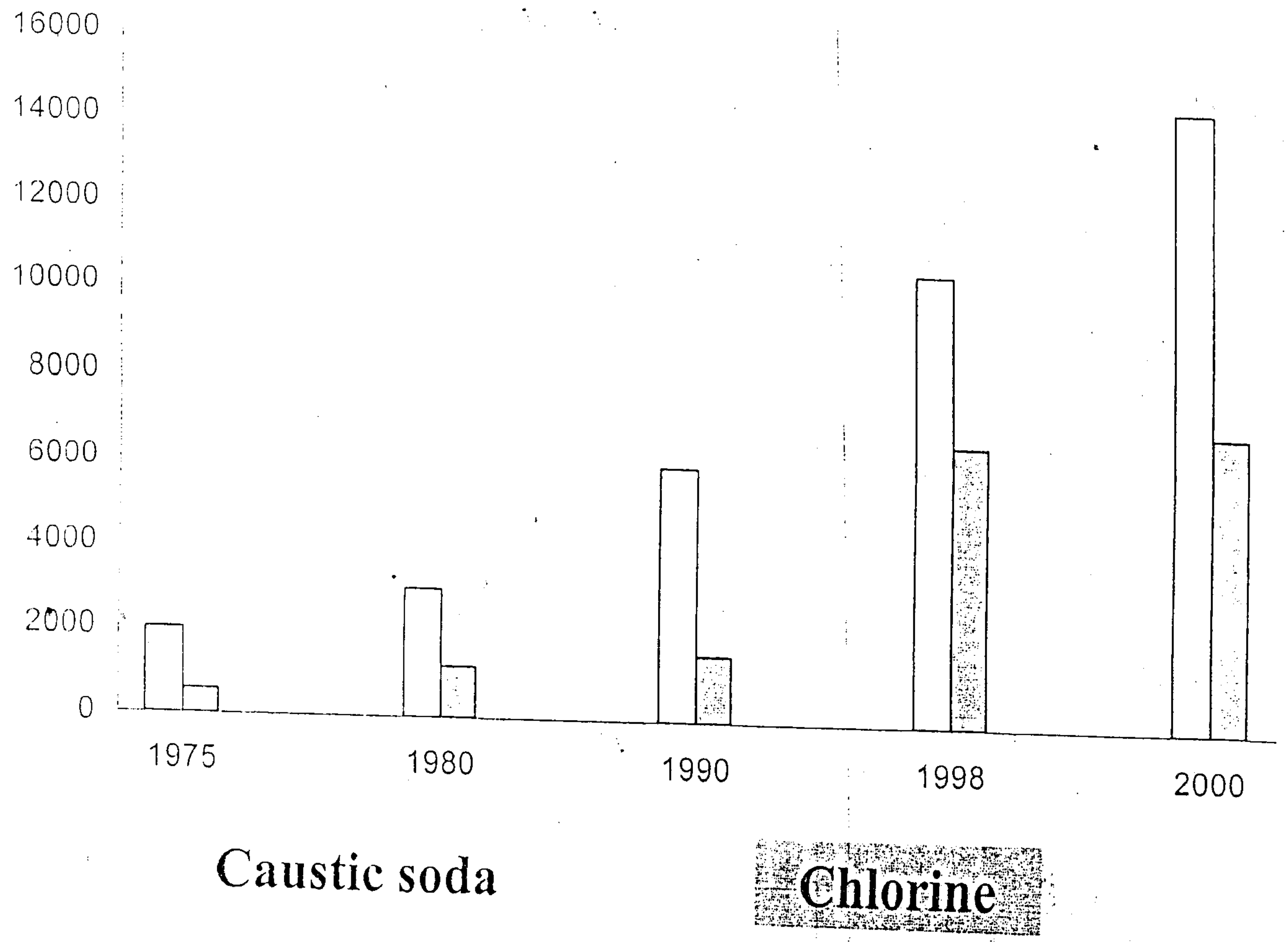
Raw Material Cost (Rs per Ton)



COSTS TRENDS OF COAL & FUEL OIL

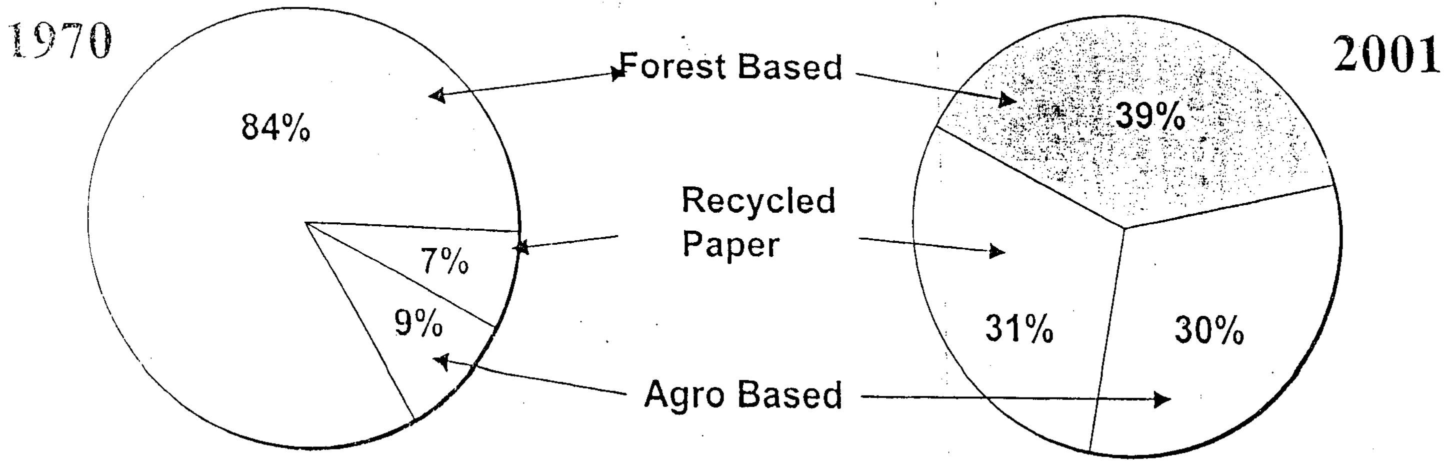


TREND IN RISE IN COST OF CAUSTIC SODA & CHLORINE



- Sustained raw material availability.
- Inadequate infrastructure.
- Uneconomic scale of operation.
- Obsolesence of technology in most cases.
- High cost of basic inputs.
- Eco friendly state of art processing technologies.

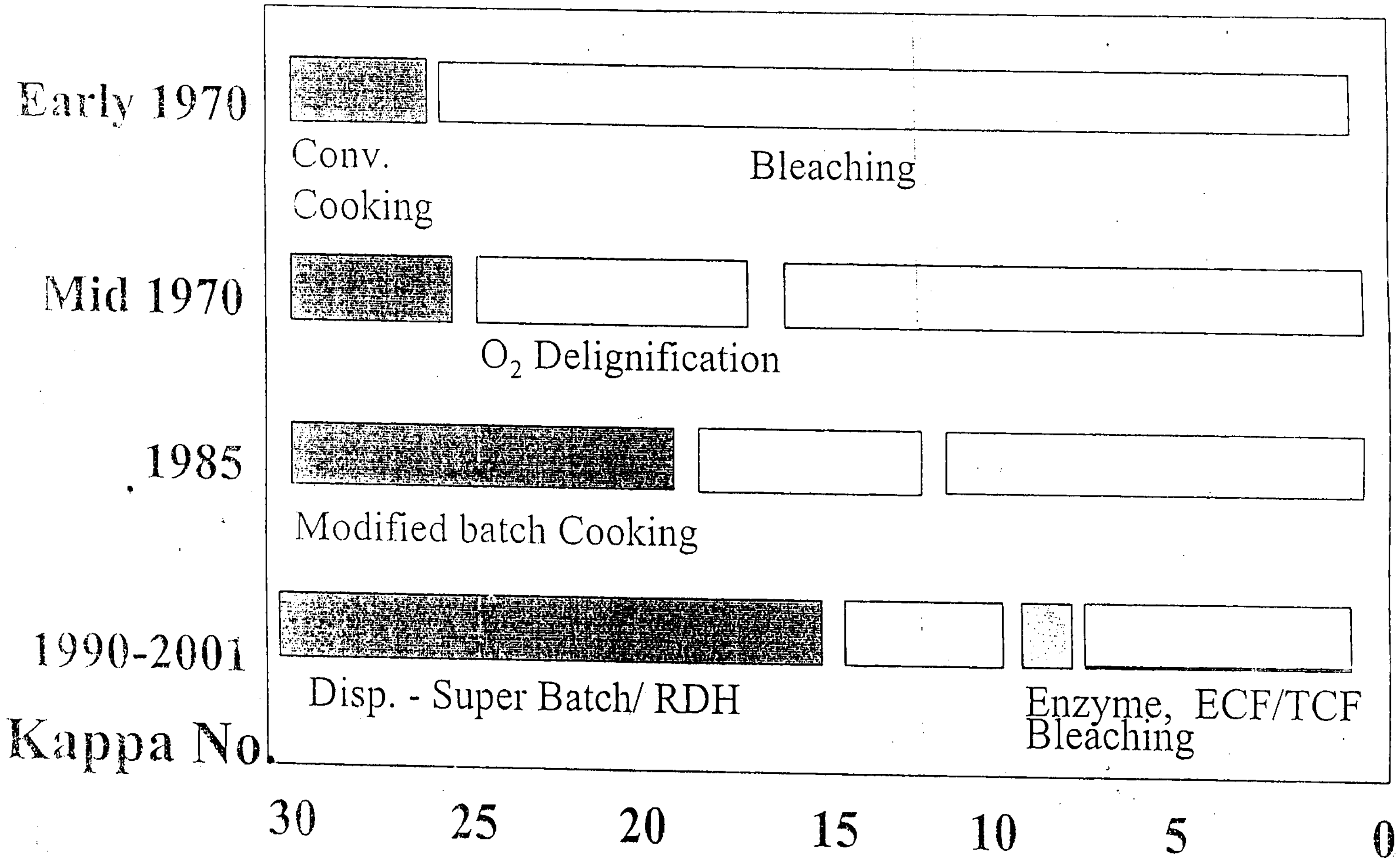
Raw Material : Change in Trends



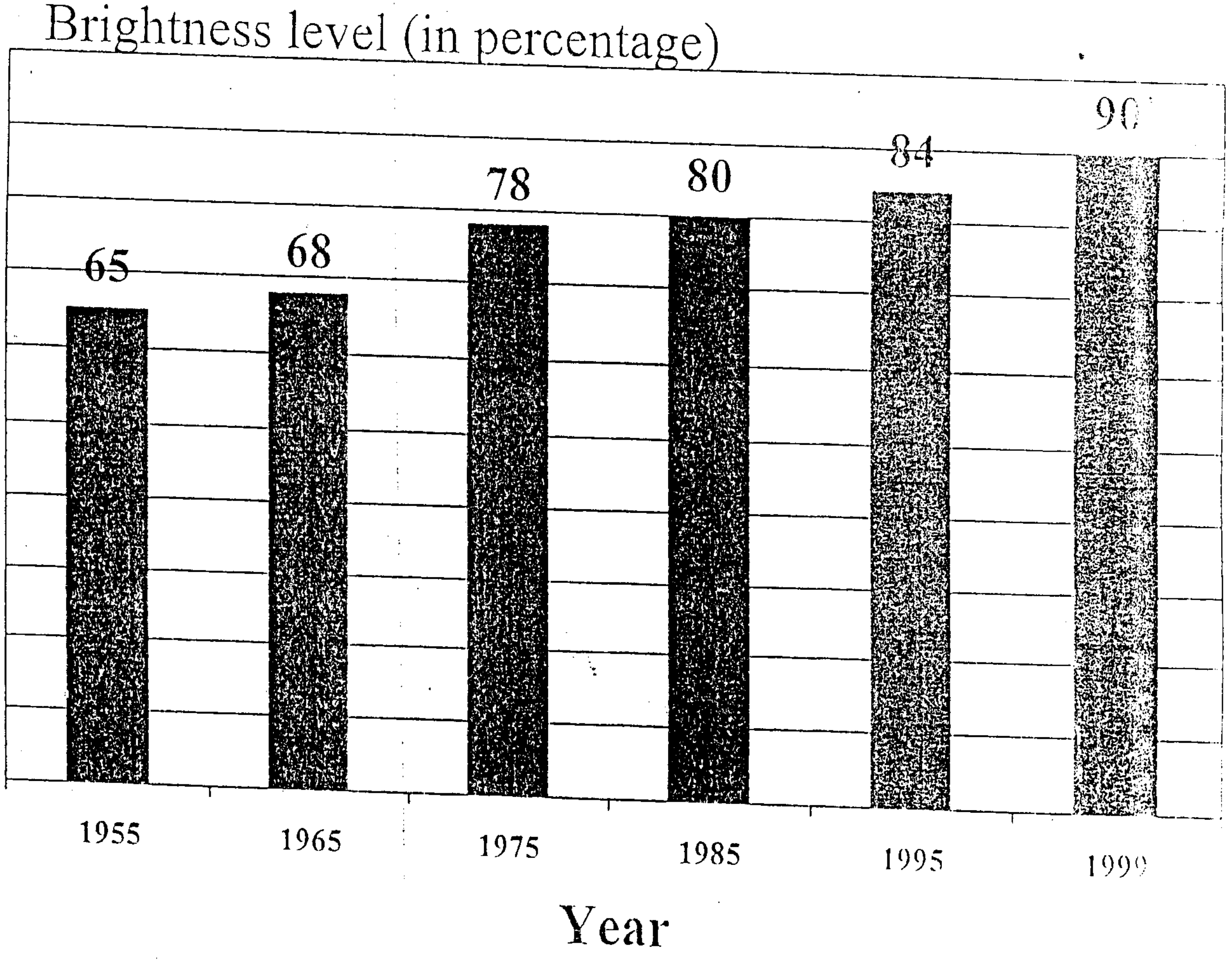
Data	Year	
	1970	2001
Registered Units	57	400
Production, Million tons/yr.	0.77	3.85

- Improved Raw Material Handling & Cleaning.
- Continuous/ Modified Batch Digesters.
- State of Art Washing Systems.
- Elemental Chlorine Free Bleaching.
- Omission of Direct Contact Evaporators.
- Lime Sludge Reburning System.
- Improved Solid- Liquid Separation Techniques.
- Energy efficient Effluent Treatment practices.

Pulping & Bleaching Scenario
(1970-2001)



Brightness Level of Paper



Large Paper Mills

- Higher water usage & energy discharge of colored effluent
- Toxic emmissions of chlorinated organic compound (AOX).
- Solid waste generation – Lime sludge & Fly ash.

Medium & Small Paper Mills

- Higher AOX generation.
- Lack of chemical recovery leading to energy & chemical losses.
- Inefficient equipments leading to high utilities.

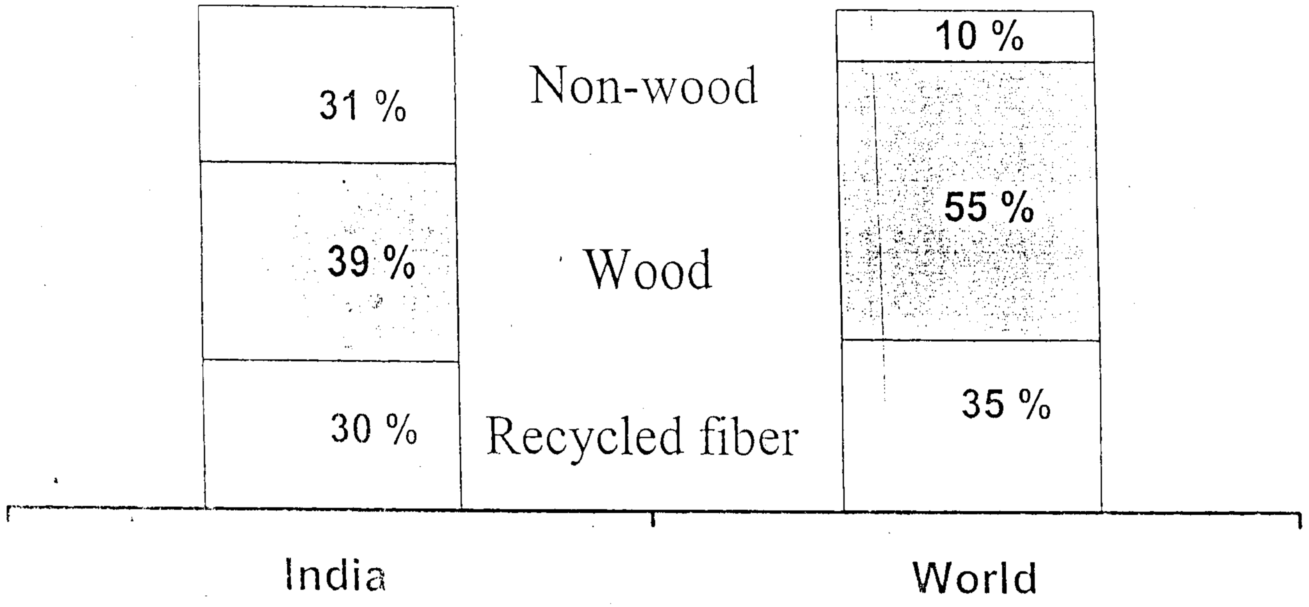
AOX DISCHARGE LEVELS : SEGMENT WISE

TYPE OF MILL	AOX LEVEL kg/T PAPER
RAYON GRADE PULP	<0.5
NEWSPRINT PULP	<0.5
LARGE PAPER MILL	1.0 – 2.5
MEDIUM & SMALL MILLS	4.0 – 6.5

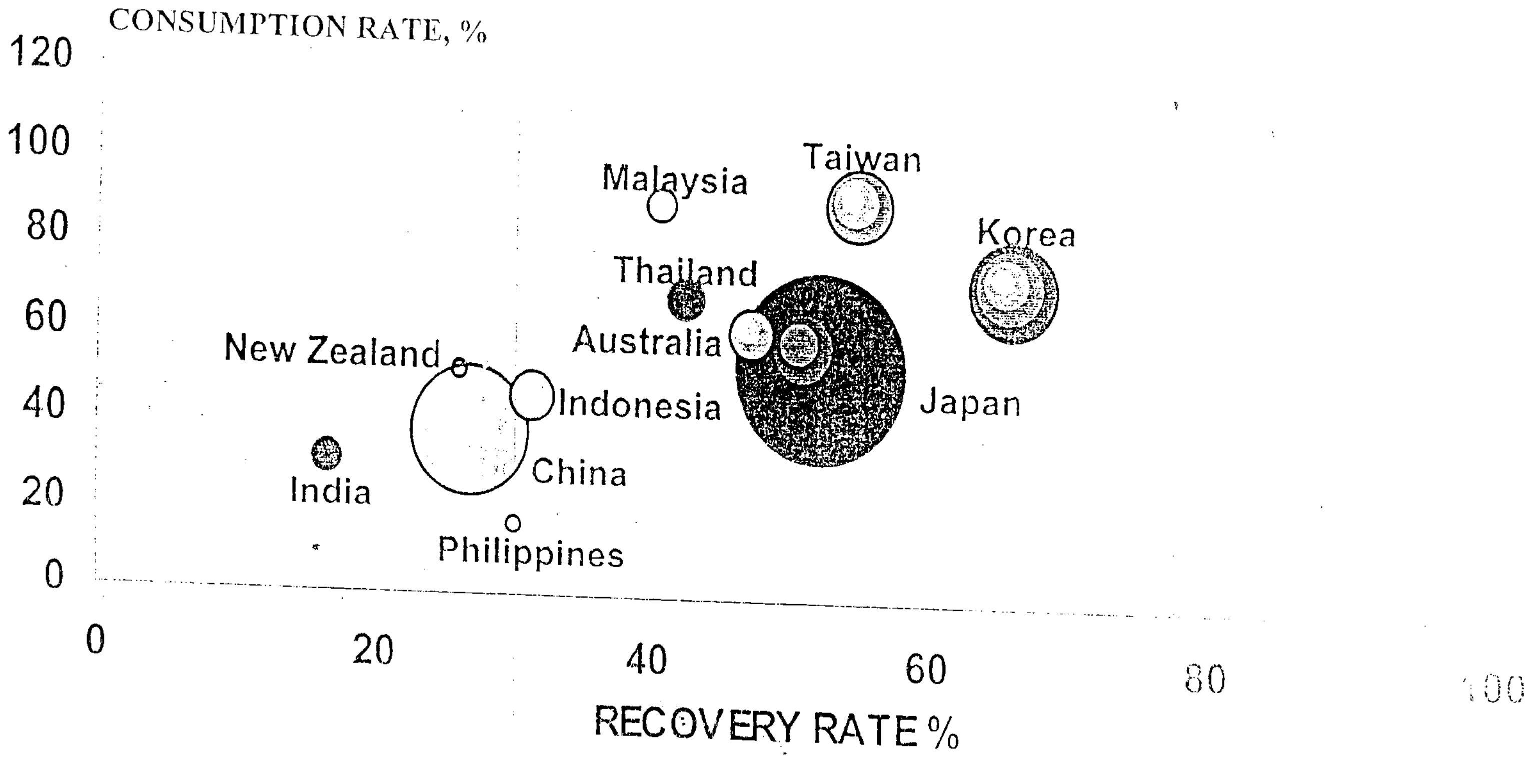
India Vs World : An Overview (2000)

Parameter	India	World
Installed Capacity, Million tons/yr.	5.4 ( )	364
Average size, tons/yr.	13,300	41,000
Production, Million tons/yr.	3.9	323
Production Growth Rate,%	4.6	2.6
Per Capita Consumption, kgs	4.0	54.0
Consumption Growth Rate,%	3.5	2.9

Raw Material



Waste Paper Consumption & Recovery

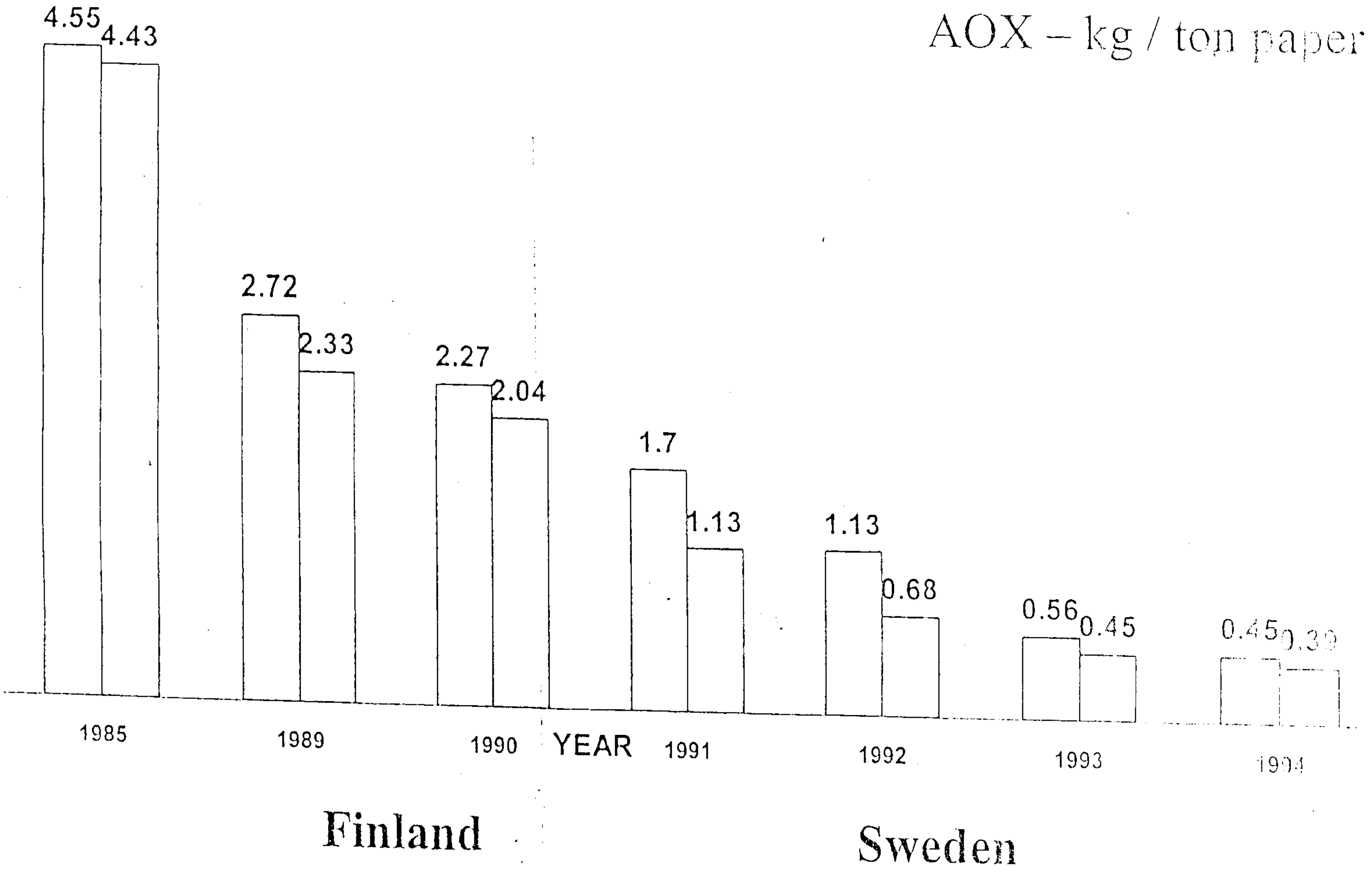


Bubble size is a function of waste paper recovery in tonnes

Consumption of Basic Inputs

Input Norms (per ton basis)	Mills in India	Mills Abroad
Raw material, tons	2.0- 2.4	1.8-2.0
Energy, GJ	23-37	18-22
Water, m^3	150-250	50-100
Chemical Recovery,%	88-94	95-98
Manpower,nos.	14-20	5-7
Technical manpower, no.	2	4

DEVELOPMENT OF AOX DISCHARGES FROM PULP AND PAPER INDUSTRY



Paper mill structure in India 2001

There are over 500 paper mills in India. Most of the mills are small, only 34 mills have a capacity of over 33 000 tons. The Indian paper machines are mostly small units. In an international comparison, even the largest machines are medium-size, as large-scale machines are today in the range of 400 000-600 000 t/a, and have a trim width up to 10 metres.

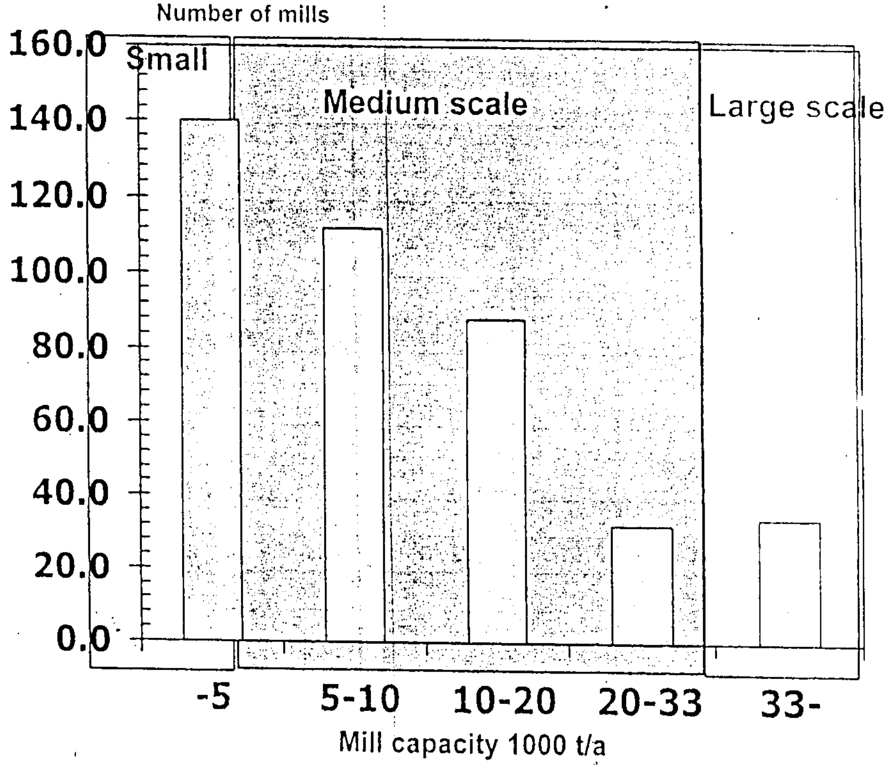
The following parameters are illustrative of the Indian paper industry:

The average capacity of paper machines is about 14 000 t/a

Most of Indian paper machines have a trim width from 1.5 to 3.5 m

There are only 9 paper machines with trim width of 5 m or more

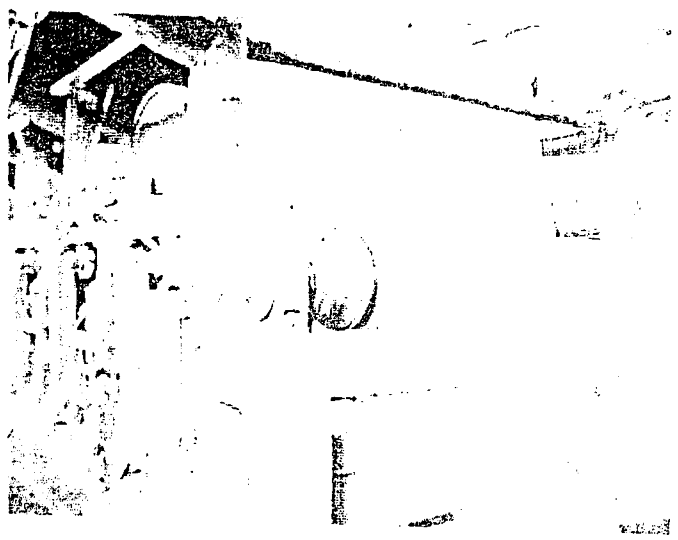
Only 14 machines have capacities 50 000 t/a or more



PM size and speed - economy of scale

Mills and machines are relatively small in India, technology is outdated and quality of raw materials and end products is low.

- Average and maximum mill and machine capacities are small in India. New machines should be about 50 % of international size.
- With bigger machines investment costs, production of utilities, effluent treatment and all fixed **costs would be lower per produced ton of paper**. Especially bigger machines can afford better automation (QCS and DCS).



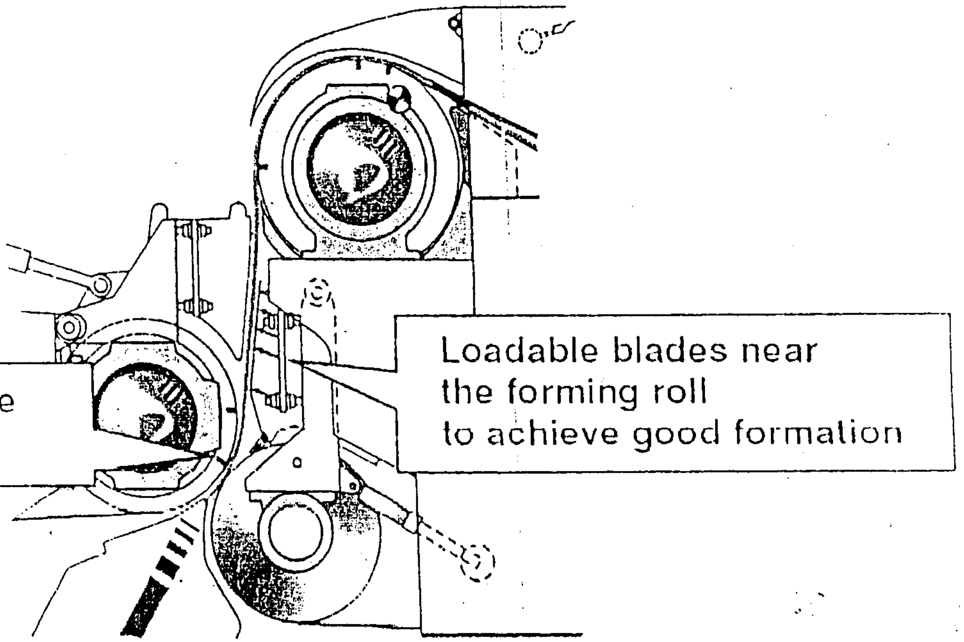
- **High speed machines** (>1000 m/min) are needed for cultural papers and one-layer boards. With a higher design speed, technology and paper quality will be closer to the international standard. Machine width can be less than internationally.
- To be internationally competitive **second hand machinery should be imported very selectively**.
- Machines representing old-dated technology should be **gradually shut down.**
- Mills and machines have to focus on **few grades** to be more effective

High speeds – better quality with gap former

Project Number 52A0087
Final Report

There are only some twin wire machines in India. Best roll and blade gap formers improve paper quality and drainage, which are important for paper made of Indian raw materials. Roll and blade gap formers give a good combination of the following properties:

Formation
Retention
Drainage
Orientation
Two-sidedness
MD&CD profiles



Suitable wrap angle of forming roll

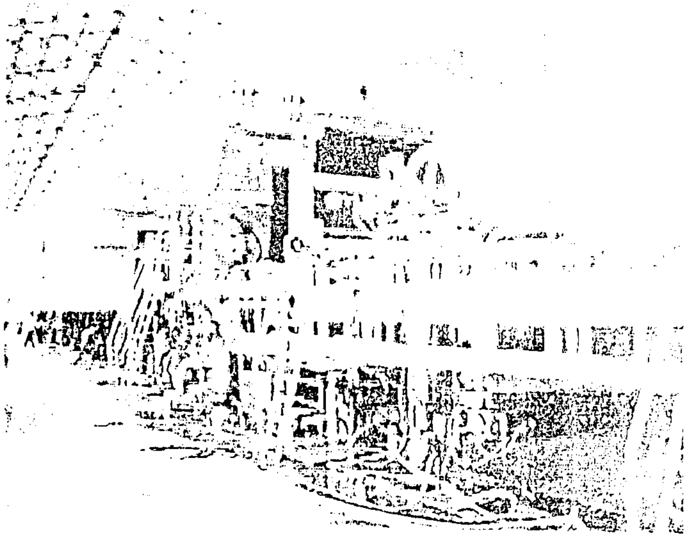
Loadable blades near the forming roll to achieve good formation

Source: Metso Paper

Wire sections of Indian paper industry

Old technology in wire section increases cost of papermaking. Filler content must be low and long fibre content high. Paper formation is not best possible and there are easily pinholes in the paper. Reduction of basis weight is difficult.

- Headbox technology is old: open headboxes, rectifier rolls, no automatic CD profile control.
- **Wire section** is normally Fourdrinier with dandy roll. Board machines are still mainly cylinder vat machines. Several machines are without suction pick-up. Even metal wires are used.
- There are only **few twin wire** machines. Even the newest machines can have machine size and technology from 1970s.
- With Fourdrinier and dandy roll it is impossible to get **high filler content** because filler is enriched to the top side. This increases dusting and reduces dry and wet pick. Also two-sidedness and curl of Fourdrinier paper are big problems.
- Low speed Fourdrinier paper is not highly oriented (tensile ratio 1.5 – 2.5). Paper made with a high speed gap former can have tensile ratios from 3 to 4. A **high tensile ratio** improves runnability and gives opportunities to reduce long fibre and to increase filler content



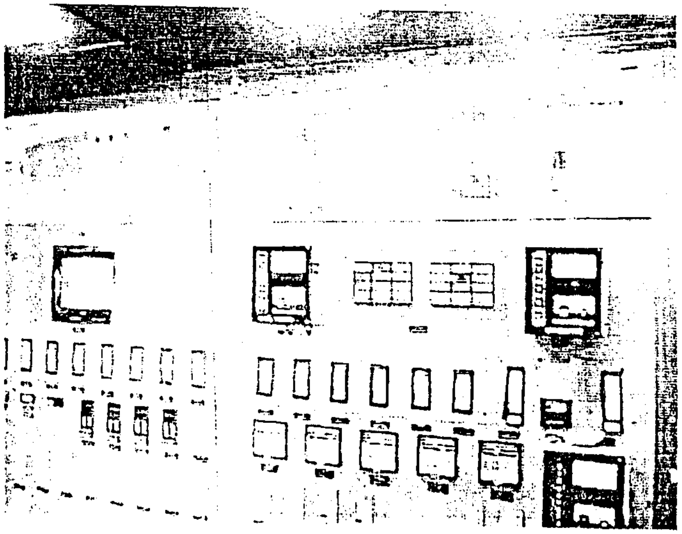
JAAKKO PÖYRY
Jaakko Pöyry Consulting

Improvement of paper quality

Domestic raw material quality & availability (fibres, pigments and chemicals) must be improved. Level of quality control systems and automation should be better.

- The level of **quality measurement and control** should be better. Cost of automation for a small machine is almost same as for a big machine. Bigger machines can afford better quality control systems (QCS and DCS).
- Classification of products and mills should be based on the customer **requirements** and their **converting processes**. Now the thinking is based too much on raw materials and mill size.

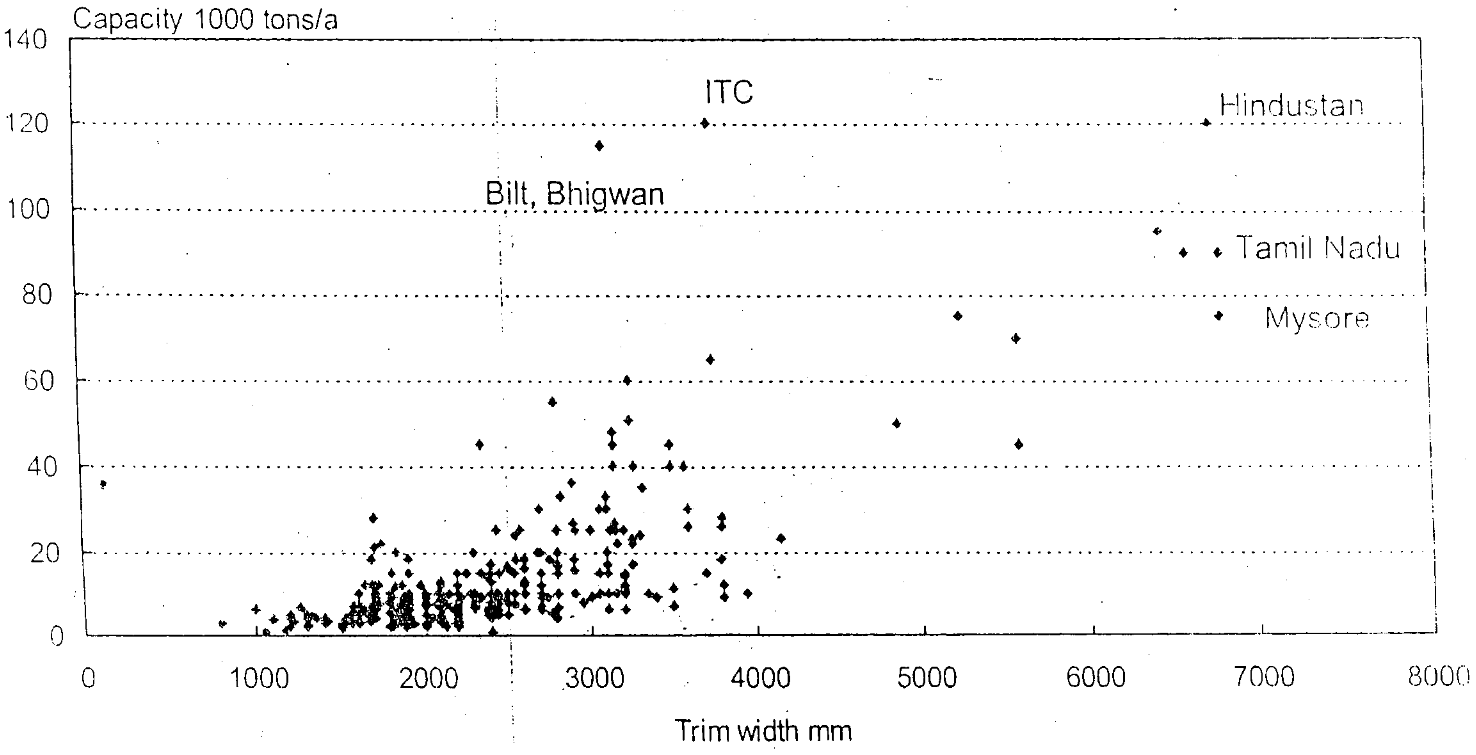
- **Bleaching** sequences should be improved to get the standard ISO-brightness of 89 %.
- **Mills must have export** to be able to improve quality and to meet the international competition and development.
- Indian paper industry needs more **international contacts**, joint ventures, training and applications instead of 100% Indian owned companies and own R&D. Input to **R&D** should be totally (mills and CPPRI) about **1% of turnover.**
- **Internet and mobile connections** from all mills should be working.



Paper machines in India 2002

Project Number 52A0087
Final Report

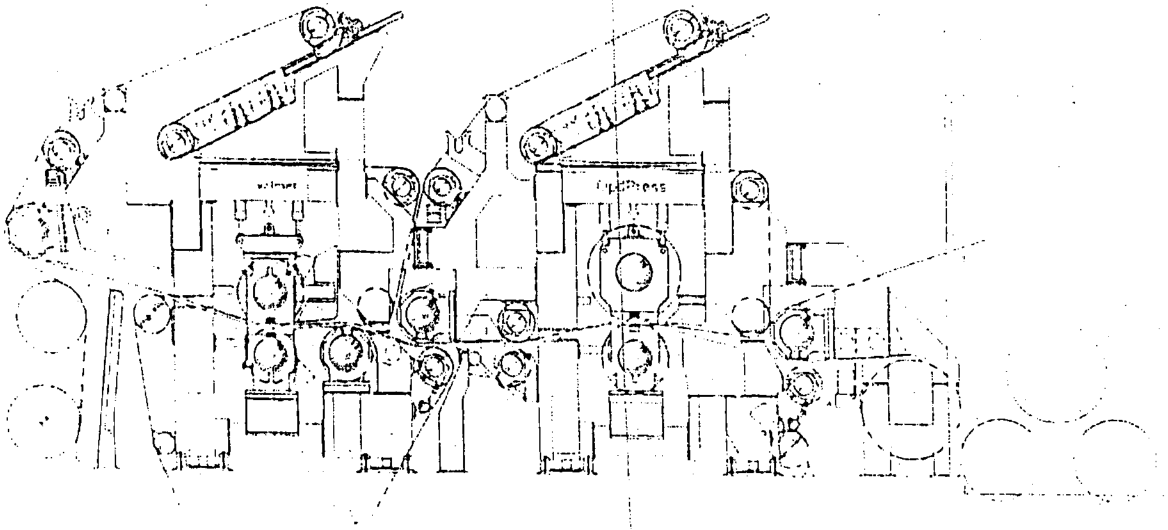
Most Indian paper machines have a trim width less than 3 metres



Better runnability with shoe presses

A two-nip press section with one or two shoe presses is today a standard solution for all paper and board grades. For Indian slow machines only one shoe nip might be best technology.

OptiPress
Roll Press and Shoe Press with Transfer Belt



Source: Metso Paper

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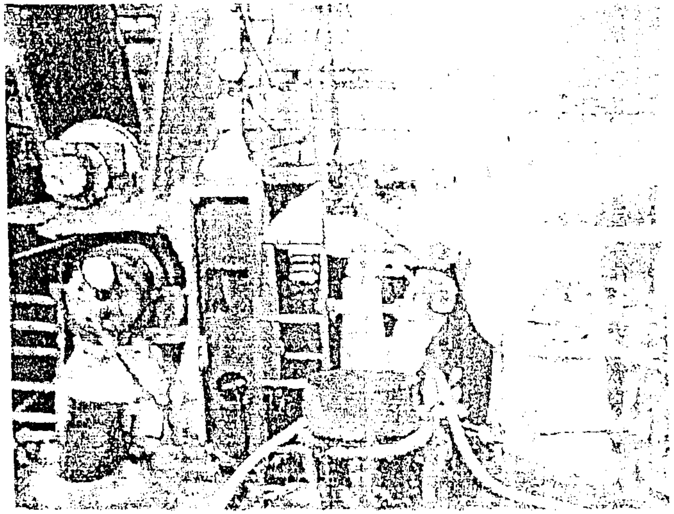
Press sections of Indian paper industry

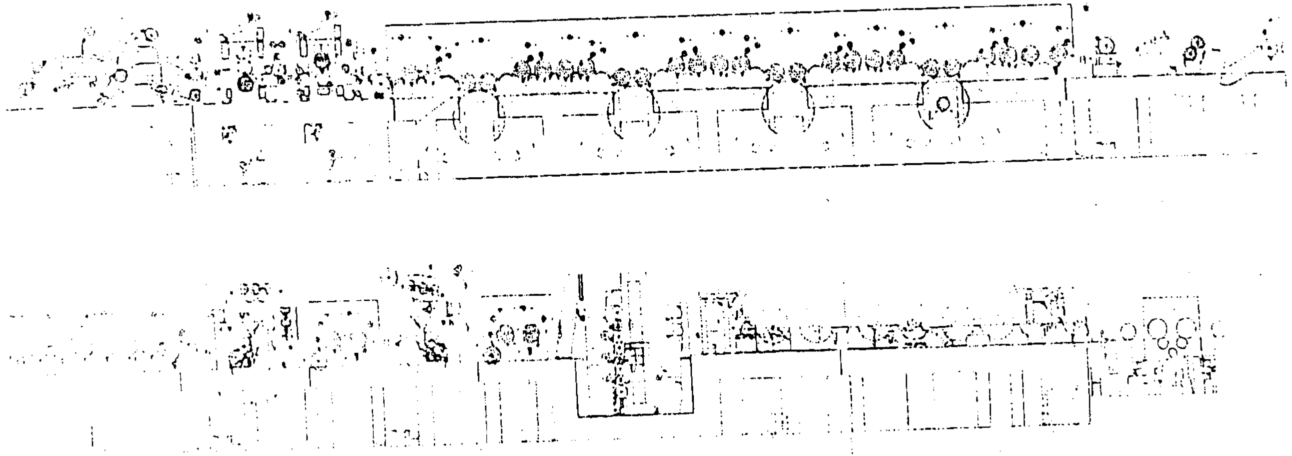
Open draws in press section increase cost of papermaking. Filler content must be low and long fibre content high. Solids content after press section in India is 5-10 %-unit lower than in developed countries. This increases steam consumption 25 – 45 %.

- Even if there is suction pick-up, open draw follows after first or second nip. Presses are usually pneumatically loaded with **very low linear loads**.
- A good press section is more important in India than in other countries because **fibres are short**. Initial wet web strength with Indian fibres is extremely low and would require best possible press sections.
- A good runnability in the press section allows **higher filler contents**. A higher filler content improves solids content and **reduces steam consumption** in the drying section.
- Indian agro fibres have very high water retention value. These fibres require time to dewater. Extended nip is the only good solution. A high speed machine with one-nip shoe press (or maximum two nips) would be the most suitable concept for Indian raw materials.



New drying concepts

New drying concepts include web supported run with vacuum pick-up to dryer section and single-tier dryers. Hood is closed and there are efficient air and infra red dryers.



Source: Metso Paper

New drying concepts Online coating Online calendering

Drying sections of Indian paper industry

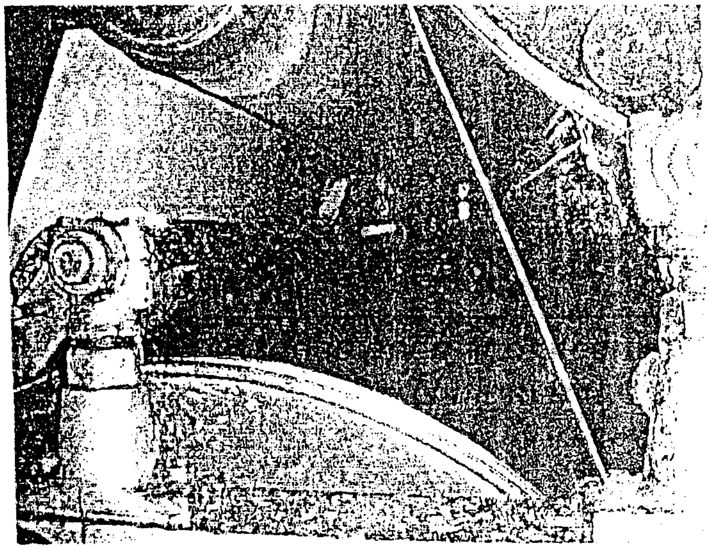
Project Number [illegible]
Final Report

Unsupported web run is normal in Indian dryer section. Hood is open and steam consumption is high. Humidity profiles are poor, paper roughness is high and CD-profiles uneven. Moisture content of final paper is low and calendering very hard.

Web run in India is normally **unsupported.** This is OK when speed is low. When speed is higher than 600 m/min there should be some single tier dryer groups to avoid web flutter and breaks.

Hood in India is usually open. The **open hood** means that web is dry at the edges and moisture profile in the cross machine direction is poor.

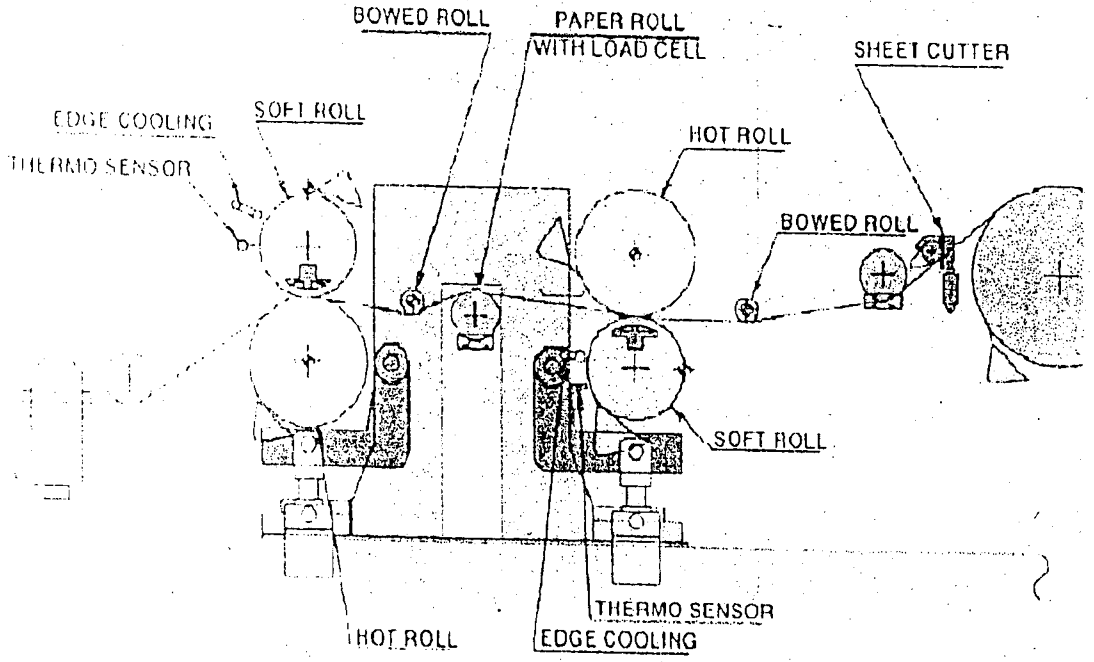
- To avoid calender blackening with uneven CD profiles the average moisture must be low. This increases paper roughness and decreases strength properties in the calendering. Web is dusty, **fibre rising** and roughening in the web offset printing is very high.
- In many cases there is **no on-line measurement of web moisture** at reel. Before size press the moisture measurement is uncommon. There are only few automatic CD-controls for moisture in India.



World class calendering technology

Project Number 52A0087
Final Report

For newsprint and woodfree papers ***state-of-the-art calendering technology is soft calendering or multinip calendering with soft rolls.*** *Two stacks are normally not needed today. There are normally 2 - 6 rolls in the stack.*

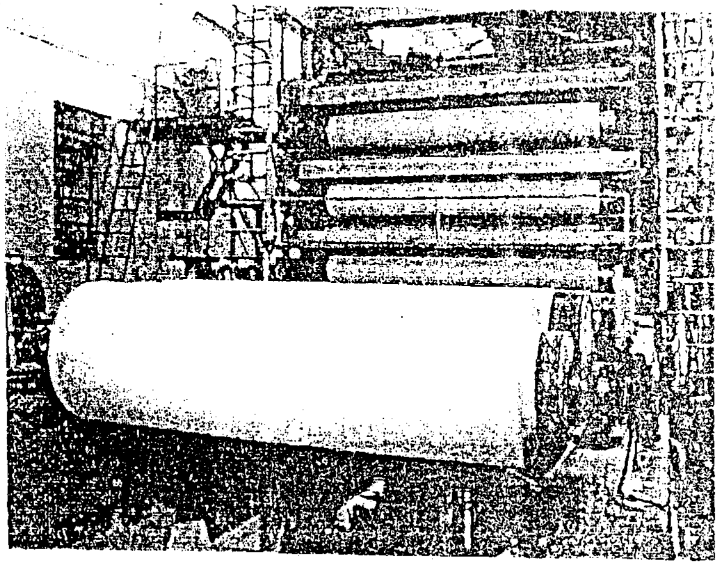


Calendering technology in Indian paper mills

Project Number 52A0587
Final Report

Soft calenders are highly recommended to Indian paper industry. However, these require good CD-profiles and more investments in the process line.

- Indian calenders are normally **hard nip** calenders with four or more rolls.
- Soft calenders could **save bulk** and improve opacity, strength and smoothness. These are critical properties of Indian papers.
- However, soft calenders require **good CD profiles** because the possibilities to control caliper profile are not as good as with a hard nip calender.
- To improve the situation it is not enough to install a soft calender, but there should be a comprehensive **study of CD-profiles.** According to this kind of study it would be easier to consider the required investments with a new calender. This would be a key research area for CPPRI in the future.



Global Competitiveness of Indian Paper Industry : Constraints

High cost of transportation and power

Low scale of operation

Inferior pulp & paper quality

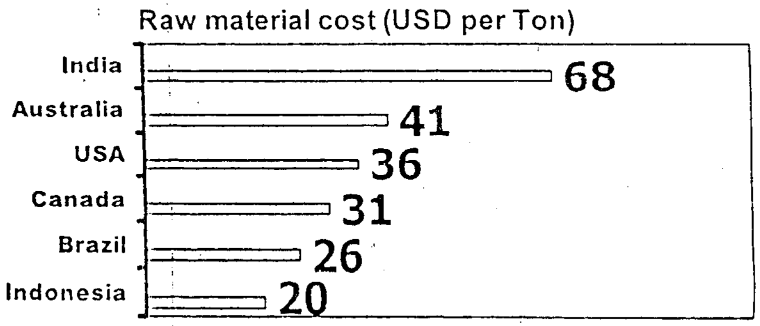
Low capacity utilisation

State of art equipment & machineries

High capital costs

High costs of raw materials

Only 20% of the total used paper is recycled in India compared to a global recycling average of 40%



Key factors that will affect growth of Indian Paper Industry

Global Focus

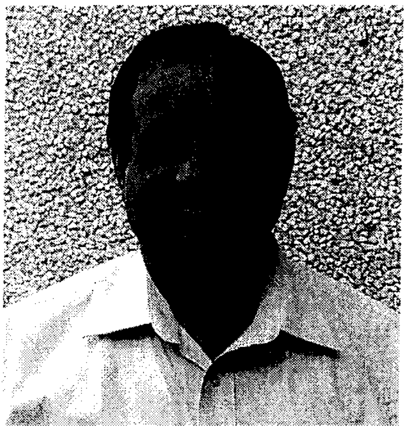
Technology

Pulp & Paper Industry

People & Business

Energy & Environment

IMPORTNACE OF CALIBRATION AND SUITABLE METHODOLOGY FOR PULP & PAPER MILLS



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IMPORTNACE OF CALIBRATION AND SUITABLE METHODOLOGY FOR PULP & PAPER MILLS

Y. V. SOOD

1. INTRODUCTION

Testing raw materials, end products and intermediates of manufacturing process is a common feature for all industrial processes. In one form or another, testing has been performed as long as products have been manufactured. Today, testing is a very important part of all industrial activities. As industrial processes become more sophisticated and a tolerance for property variations of products becomes tighter, efficient and relevant testing increases in importance. Testing tries to describe numerically certain properties or features of the product, its intermediate, or both. In industrial production, the reasons for testing may be very different. Testing may use intermediates or final products to control process conditions. The intent may also be quality control of the final product for consistency and correspondence with relevant quality specifications. Testing may also try to obtain property values for use in marketing a product. In all these cases, usage of properly **calibrated instruments** is utmost important.

Today, an increasing amount of testing occurs directly on-line during the production process. On-line instruments do certainly enhance the possibility for efficient process and product quality control. Therefore, industry wants to perform process and product control on-line as much as possible. A lack of proper sensors and gauges is in many cases a deterrent to further development of process control.

Despite increasing on-line testing, the need for traditional laboratory testing will always remain for the following reasons:

- All necessary tests are not possible on-line.
- On-line testing equipment require calibration with laboratory tests.
- On-line testing does not include conditioning of the samples to standardized conditions.
- A customer may require very specific tests on purchased products.

To ensure the correct testing and functioning of the instruments it is utmost important that these are calibrated according to standard methods. The instruments not properly calibrated are liable to affect the proper quality assessment of end product.

Quality of any end product has become one of the most important factors in global trading. Evaluation of quality is closely linked with proper calibration of the testing machines. Finish Pulp & Paper Research Institute, Helsinki &

PIRA UK etc have been providing calibration service to the pulp & paper mills in their countries for quite a long time. This helps the mills to ensure correct quality evaluation of pulp, paper & chemicals etc. It generates a confidence among the users. In India many of the pulp & paper mills have very old and obsolete testing machines. Ensuring the accurate performance these machines need regular calibration and performance check. **The Comparative Calibration Services** are required to help the pulp & paper mills to check the performance of testing machines so that the desired quality parameters of the raw material and end products are ensured. In the comparative calibration service calibration is not done at the site however standard samples are supplied to the clients. This is the practice being followed by Finnish Pulp & Paper Research Institute, Finland and PIRA, U.K.

2. TECHNICAL APPROACH

CPPRI has started the comparative calibration services. The following steps are adopted for comparative calibration services.

- The different grades of paper having good formation in reels are procured from different mills and are subjected to seasoning (conditioning under controlled atmosphere) prior to be used as standard sample.
- The samples are evaluated for different characteristics using calibrated instruments.
- Standard comparative samples are prepared and sent to the mills for evaluation.
- The result received from mills are evaluated and mills are informed about the
  - ❖ Action Limit
  - ❖ Performance of the equipment

3. PREPARATION OF PAPER SAMPLES

Paper is not homogenous material. In paper testing, the measurement is often repeated several times to determine an average result that describes the level of the property better than single measurement. A statistical distribution always relates to the test result. The size and type of distribution depend not only on the homogeneity of the material and the number of repetitions of the test but also on other factors such as equipment and operator. For the preparation of comparative paper samples, the paper samples selected are those which fell into **Cases A, B, & C** of **Fig. 1**.

Case A

The result and its uncertainty are both within the limits.

Case B and C

The result is within the limit but a small part of the uncertainty is outside the limit. The result indicates that compliance is more probable than non-compliance.

Case D and E

The result is on the limit itself and half the uncertainty is within the limit and the other half outside the limit. The result indicates that compliance is as probable as non-compliance.

Case F and G

The result is outside the limit but a small part of uncertainty is within the limit. The result indicates that non-compliance is more probable than compliance.

Case H and I

Both the result and the uncertainty are outside the limit. The product therefore does not comply with the specification.

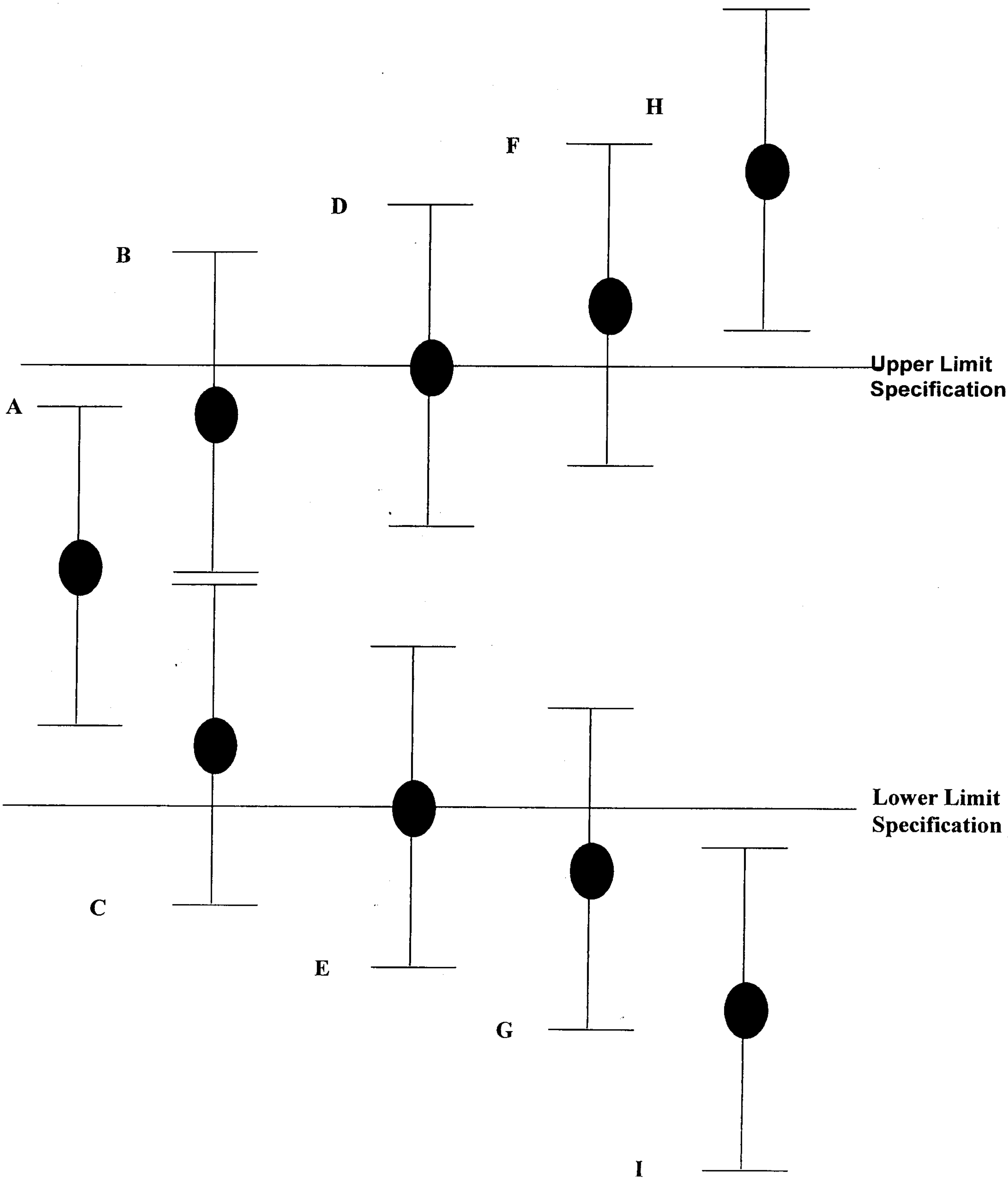


FIG.1: A RESULT & ITS UNCERTAINTY COMPARED WITH A GIVEN LIMIT SHOWING NINE

The care is taken to avoid the hystersis effect in paper samples. The strength properties of paper are dependent on ambient temperature and relative humidity. More precisely it is the actual moisture content in the paper, regardless of how it has obtained, which affects the strength. For the test material to attain a stable equilibrium moisture content in a standardized atmosphere, the test piece must be conditioned for a sufficiently long period of time. It is also important that the conditioning to standard atmosphere always take place by starting from lower humidity (about 30%) so called pre conditioning, in order to attain a reproducible equilibrium moisture content (**Fig. 3**). This is because of the so called moisture hystersis effect on the fibre material. Difference more than 2% can be obtained because of the moisture hystersis effect (**Fig. 2**).

Internationally it has been recommended that the correct equilibrium moisture content is that which is obtained on absorption. (**Fig. 3**).

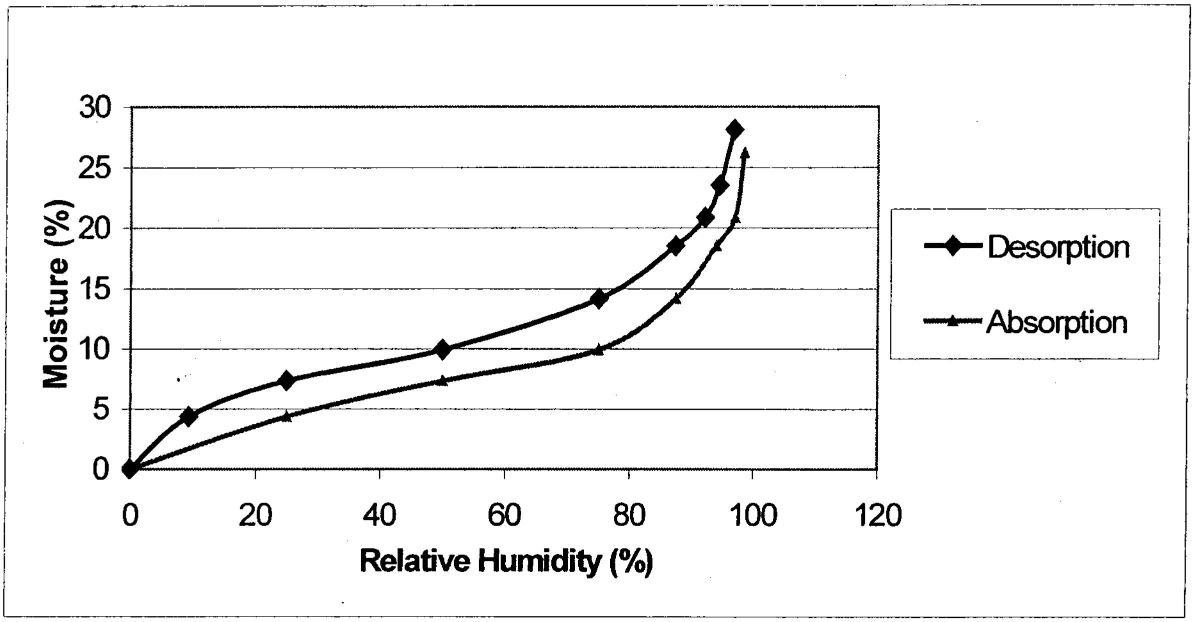


FIG. 2: HYSTERSIS EFFECT ON MOISTURE CONTENT

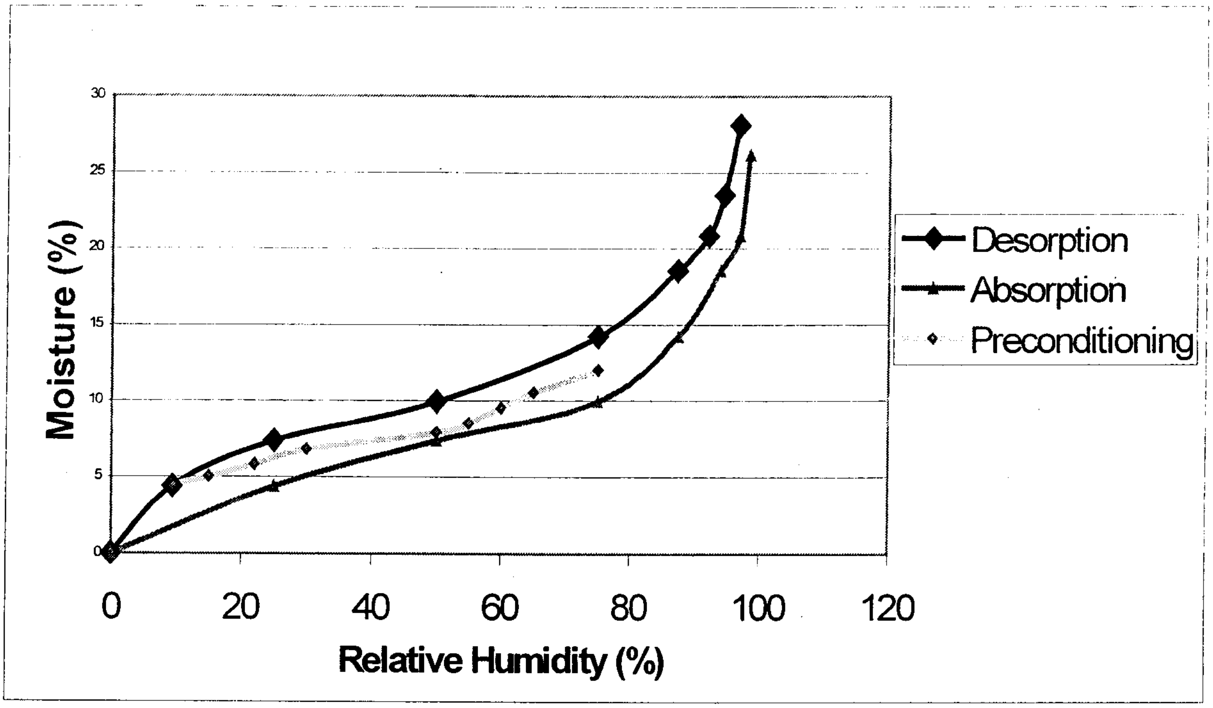


FIG. 3: HYSTERSIS EFFECT NULLIFIED ON PRECONDITIONING

4. EFFECT OF MOISTURE ON THE PAPER CHARACTERISTICS

The effect of moisture on different paper characteristics is shown in **Fig. 4**. The tensile strength increases slightly to a maximum at 30-35% relative humidity and then decreases quite rapidly at higher relative humidities. This decrease can be attributed to a weakening of fiber-to-fiber bonding.

Tearing resistance increases over the entire range of relative humidity. However, it is obvious that at some point above 80% the curve will fall off rapidly, due to the disruption of interfiber bonding. The paper becomes more stretchable and plastic with increasing moisture content. It is believed that this enables paper to distribute tearing stresses over a large area, thereby increasing the amount of stress absorbed before failure occurs. On the other hand, it is thought that the rigidity and low stretch characteristic of dry paper localizes tearing stresses, resulting in a low tearing strength.

Stiffness continuously decreases with increasing moisture content. This is primarily a result of increased fiber flexibility. Softness, which is inversely related to stiffness, increases substantially with increased moisture content. Consequently, in papers that require softness, additives are sometimes employed that encourage high moisture content.

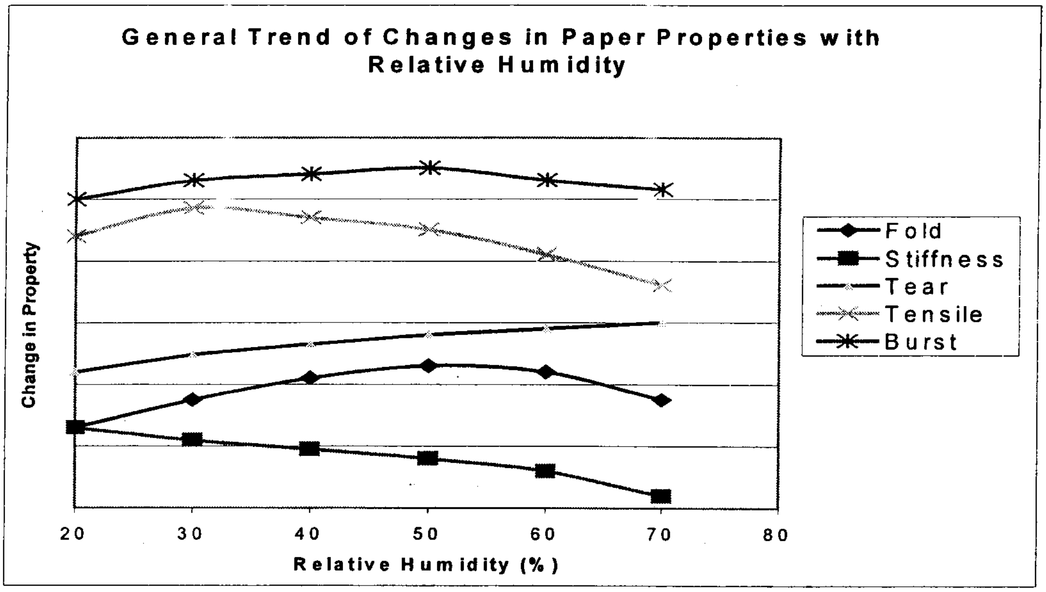


FIG 4: GENERAL TREND OF CHANGES IN PAPER PROPERTIES WITH RELATIVE HUMIDITY

The bursting strength curve shows a broad maximum between 30% and 60% relative humidity. This can be interpreted in the following way.Bursting strength is thought to be primarily a function of tensile strength and stretch. From 20–35% relative humidity, both tensile and stretch increase, producing an increasing burst; from 35–50% relative humidity, stretch continues to increase and tensile falls off at an increasing rate, but stretch continues to dominate and burst continues to rise although at a decreasing rate. Finally, above 55% relative humidity, the decrease in tensile strength is greater than the increase in stretch, and bursting strength decreases continuously.

Folding endurance is strongly influenced by moisture content, also, increasing markedly up to 50–60% relative humidity and then falling off thereafter. Even though the curve in **Fig. 4** shown above looks fairly straightforward, it cannot be analyzed in a complex function involving tension, compression, the stress-strain properties of the fibers, fiber flexibility, and probably other factors yet to be discovered.

5. CALIBRATION AND OBJECTIVE OF TESTING

The main objectives of testing are as under:

1.	Quality Control	(i)	Establish and maintain quality
		(ii)	Control off-grade production
2.	Process control	(i)	Control of raw material
		(ii)	Control of process
		(iii)	Control of wastage
		(iv)	Improve process efficiency
3.	Process monitoring	(i)	Assess performance
		(ii)	Compare with other mills
		(iii)	Pinpoint problem areas
4.	Monetary control	(i)	Establish cost
		(ii)	Provide consideration for alternatives.

Before a test instrument is used, it must be calibrated so that it can produce results that are internally consistent, and which can be compared with results obtained on similar calibrated instruments by other when the same test method is used. Data generated by an uncalibrated or out of calibration instrument can cause untold damage in terms of wrong decisions and the resultant misdirection of effort and capital. An instrument calibration program is required if measurements are to be used to improve quality, product, and profitability. Apart from the calibration, precision and accuracy of the test method plays an important role. Precision is a statistical term used to describe the variability of introduced test values of some measured property about the average or mean value.

The data variability that results when a test method is used in a specific testing application has many sources. The sources are

- Sampling variability
- Equipment maintenance and calibration
- Difference among users

Sound decision making requires data of known precision. Decision made on imprecise data, particularly when the imprecision is unrecognized, can be costly.

The accuracy of test result is also important. The term accurate is used to describe the agreement of test results with some "absolute" correct value. Because of the hygroscopic viscoelastic nature of paper and the products made from it, there are few paper properties that can be treated as absolutes. Most properties must be considered in relationship to a specific test method and the accuracy defined on these terms. For example, because paper is hygroscopic, the value of basis weight as a function of relative humidity with which the paper is in equilibrium at the time of basis weight is determined. Unless a "standard relative humidity" is used for measurement, numerous "basis weight" will result. Similarly, because paper is viscoelastic, values determined vary. Mechanical properties such as tensile and bursting strength vary as a function of rate at which stress is applied to the paper tested, and the equilibrium relative humidity at which the testing is conducted.

Instrument calibration and maintenance are both key to providing reliable "accurate" test results. For technical control of mill operations it is important that tests carried are sufficiently accurate. A poor test or an improperly made test is sometimes worse than no test at all. All testing procedures can be characterized by following:

- Their sensitivity (or instrument readability)
- Precision (a measure of the variation that can be expected when repeated tests are made on the same specimen)
- Accuracy (the difference between the test value and true value)

There is sometimes confusion regarding the distinction between precision and accuracy; precision is an assessment of test reproducibility, but says nothing about the relationship of the test value to the true value. For example, if the instrument is out of calibration and if a non-representative sample is being tested, the test result in either case may be precise, but probably not accurate. Factors affecting the precision or the accuracy of a test value are

1. Instrument readability
2. Instrument or test sensitivity
3. Sampling error
4. Procedural differences
5. Instrument calibration
6. Variation in correlation between measured property and desired property
7. External factors

The precision of any test result can be defined statistically in terms of standard deviation. It is frequently useful to convert the standard deviation into percentage of median test value i.e. co-efficient of variation. One way to improve test precision is to increase the number of têst replications. However, greater replications mean higher cost for some technician testing. The number of replications specified for some common tests in different standards is listed in **Table - 1**. Strict adherence to the number of replications is desired to get proper results.

TABLE -1

NUMBER OF REPLICATES SPECIFIED IN DIFFERENT STANDARDS FOR DIFFERENT PROPERTIES.

Property	TAPPI	SCAN	APPITA	ISO	BIS
Bulking Thickness (piles)	10(5)	5(4)	10(8)	20(5)	5(5)
Grammage	-	20	20	20	10
Burst	10	8	10	10	nm*
Tensile &Stretch	10	8	10	10	10 or 20
Folding endurance	5	5	10	10	10
Cobb	-	-	-	5	nm*
Brightness	-	-	-	10	
Opacity	-	-	-	5	
Air resistance, Gurley	5	-	5	10	
Roughness , Bendtsen	-	-	-	10	
Stiffness	5	-	5	4	

nm* - Not mentioned.
In most of BIS standards, the number of replications to be carried out has not been mentioned.

5.1Accuracy of different tests

The variation, which is expected in the determination of different paper properties, is given as under (**Table-2**).

TABLE-2

REPRODUCIBILITY OF DIFFERENT PAPER PROPERTIES.

Property	Repeatability *	Comparability**	Reproducibility***	Co-eff. of variation
Tensile	4.0	-	15 (Ref. 1)	2.6 to 11 (Ref. 10, 11)
Bursting Strength	5.4	9.5	14.3 (Ref. 2)	4 to 10 (Ref. 12)
Thickness	1.25	-	5.5 (Ref. 3)	0.1 to 0.5 (Ref. 13)
Double fold	15	34	40 (Ref. 4)	-
Tear	1.5	2.5	4.5(Ref. 5)	2 to 9 (Ref. 14)
Stiffness	3 to 5	7 to 12	9-20 (Ref. 6)	2 to 5 (Ref. 5)
Cobb	-	-	10 (Ref. 7)	-
Air-resistance	11	-	2 (Ref. 8)	-
Opacity	0.64	-	0.77 (Ref. 9)	-

***Repeatability** – A limit within which agreement may be expected 95% time between two test results obtained under essentially the same conditions and from same homogenous sample of material.

** **Comparability** – A limit within which agreement may be expected 95% the time between two test results obtained under essentially the same conditions from samples of different materials being compared as to a particular property and having actually the same level of the measured property but differing perhaps markedly in other properties.

*** **Reproducibility** – A limit within which agreement may be expected 95% of the time between two test results obtained in different standard laboratories for the same homogenous sample of material.

It clearly indicates that double fold is the most unreliable test. In our paper testing laboratory, we have observed that generally for most of the paper & board tests, co-efficient of variation lies between 5% and 10%. Exceptions are grammage and thickness, which give less; fold & stretch which give more.

In paper testing it is usual way to employ the mean value of a property as a guide to judge the suitability of the material. Where the standard deviation or co-efficient of variation result is calculated, this is normally done to determine the confidence limits of the mean result. Although for some properties such as substance, roughness and stiffness this data is normally adequate, for other properties the mean value is in itself of little utility. The test value that is required is the minimum value of the property that is likely to be encountered in practice. This is true for instance for tensile strength and pick resistance. Occasionally very low values of these properties could result in web breaks or the necessity for frequent wash down on printing presses although the average value of the relevant properties could be so high that trouble might not be anticipated if mean alone were considered.

For example, if we have two paper Samples A & B which have mean tensile strength of 5 kgf and standard deviation of 0.5 kgf and 0.6 kgf (**Table -3**).

TABLE –3
PAPER SAMPLES WITH SAME MEAN VALUE BUT DIFFERENCE IN STANDARD DEVIATION.

For A	For B
90% of paper will have tensile strength between 4.5 & 5.5 kgf	90% paper will have tensile strength between 4.4 & 5.6 kgf
95% strength between 4.0 and 6.0 kgf.	95% strength between 3.8 and 6.2 kgf.
99.8% strength between 3.5 and 6.5 kgf.	99.8 % strength between 3.2 and 6.8 kgf.

If minimum tensile load desired in converting operation is 3.2 kgf, B will give more web breaks than A.

5.2 Uncertainty, repeatability and reproducibility

Uncertainty of a measurement is a parameter associated with the result of a measurement that characterized the dispersion of the values reasonably attributed to the measurand. The measurand is a particular quantity subject to measurement.

The uncertainty of a result is a comparison of many uncertainty components. It depends on material tested, sampling, principle of testing method, instructions for performance, experience and competence of the personnel, quality of the equipment and testing environment etc. Factors arising from systematic effects such as components associated with corrections and reference materials may also contribute to the dispersion.

A measurement result always consists of a value with information about measurement uncertainty. The quality of the measurement result depends on the error structure in the measurement process, while quality of the information about measurement uncertainty is determined by our knowledge about the error structure. A measurement that is not accompanied by some form of information about measurement uncertainty in therefore of no use as a measurement result. The paper industry has lacked guidance as to low measurement uncertainty should be estimated in physical testing. Measuring correctly is important, but for us in the paper industry, it is even more important to measure consistently. In other words, a measurement performed on one occasion should be comparable to measurement performed on other occasion in the same laboratory as well as in other laboratories. The standard published SCAN G.6.00 contains all the relevant information in this regard. A way of controlling measurement uncertainty is following a regular calibration schedule. The frequency of calibration is determined by the requirements for measurement quality.

Repeatability of result of measurement is the closeness of agreement between results of successive measurements using the same material under the same conditions. This mean the same operator repeats the measurements within a short period of time without any changes in measurement procedure, equipment or test conditions. Any variation noted this way describes the repeatability of the measurement.

Reproducibility of result of measurements is the closeness of the agreement between the results of measurements using different conditions of measurements when defining reproducibility, any conditions can change including the principle of the method. Statements about reproducibility therefore require a specification of the conditions.

6. MILLS/ORGANIZATION SELECTED FOR PARTICIPATION

CPPRI has started comparative testing calibration services about 2 years ago.Around twelve mills with well equipped paper testing laboratories were chosen to try this CPPRI comparative testing calibration service methodology.

These mills were sent the paper samples and the test results obtained from these mills were analyzed to check the performance of their instruments. The findings were conveyed to them for taking appropriate action at their end.

7. PROPERTIES COVERED IN THIS SERVICE

The properties and the relevant standards followed covered under this service are given in **Table – 4**

TABLE – 4
PROPERTIES AND THEIR RELEVANT STANDARD TEST METHODS.

Property	Standard Test method
Tearing Strength	ISO 1974:1990
Tensile Strength	ISO 1924
Bursting Strength	ISO 2758
Double Fold	ISO 5626
Brightness	ISO 2470
Smoothness	ISO 8791-2
Thickness	ISO R534
Gloss	TAPPI 480-om-99

8. TEST RESULTS OBTAINED AND OBSERVATIONS

The results obtained from the mills were analyzed.

The property was evaluated as per standard method. The action limit was evaluated using formulae

Action limit: Mean ± 2.0 S.D.

The report was sent to the mills in the following format

Format in which the results were sent to the mills

Your Reference —
Our Reference —

Property	Action Limit	Mills' Results	Comments
X	A to B	A+, B–	Results are in the limit
		A–, B+	Results are outside the limits and probable cause of variation

The mill took appropriate action to rectify the instrument .

The analysis data obtained for different characteristics is depicted as bar diagram (**Figs. 5–12**).

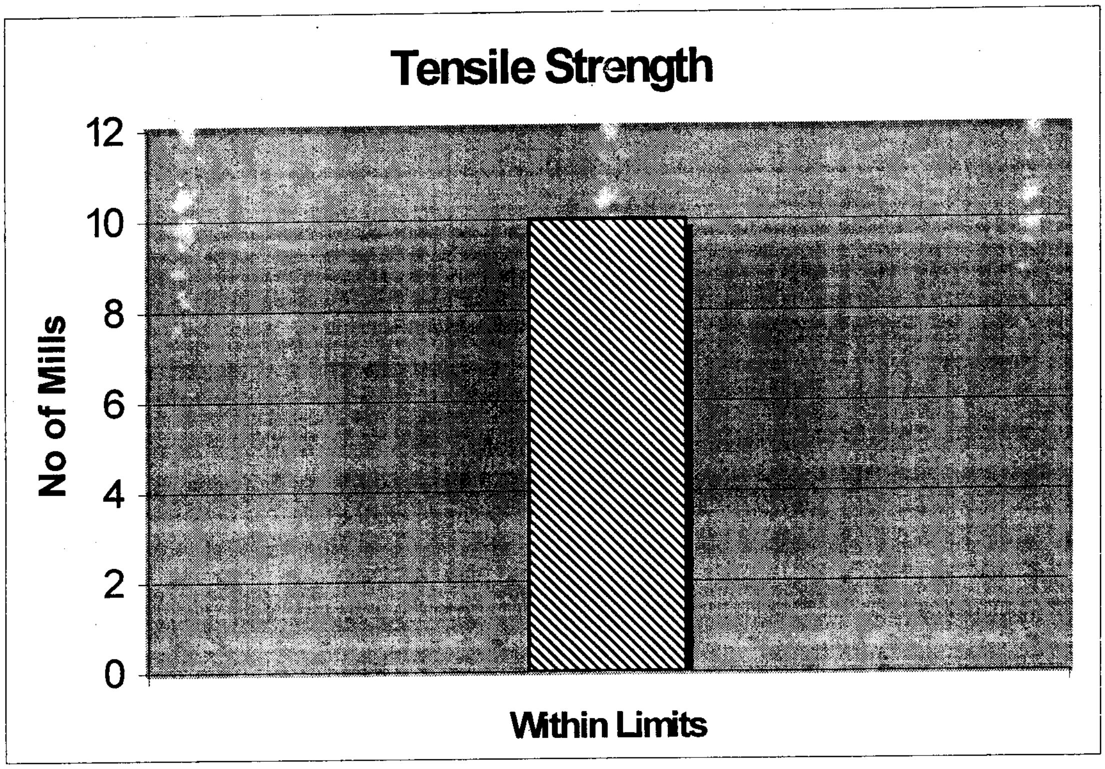


FIG. 5: TENSILE STRENGTH TESTING IN LABORATORIES OF DIFFERENT MILLS

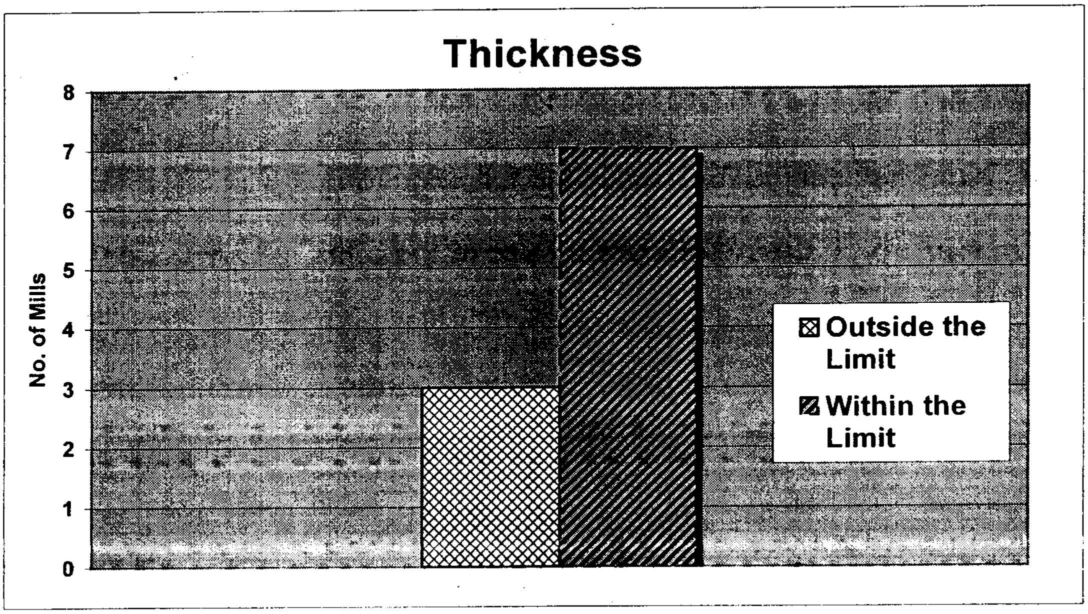


FIG. 6: THICKNESS TESTING IN LABORATORIES OF DIFFERENT MILLS

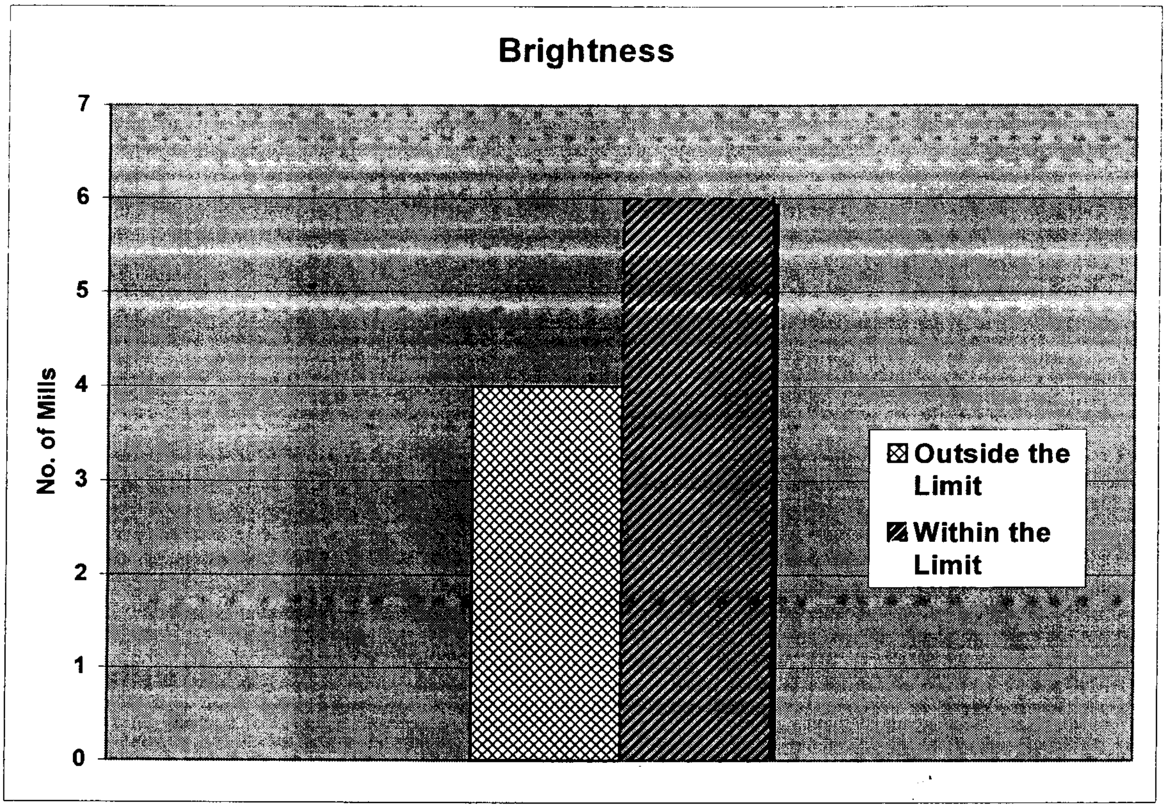


FIG. 7: BRIGHTNESS TESTING IN LABORATORIES OF DIFFERENT MILLS

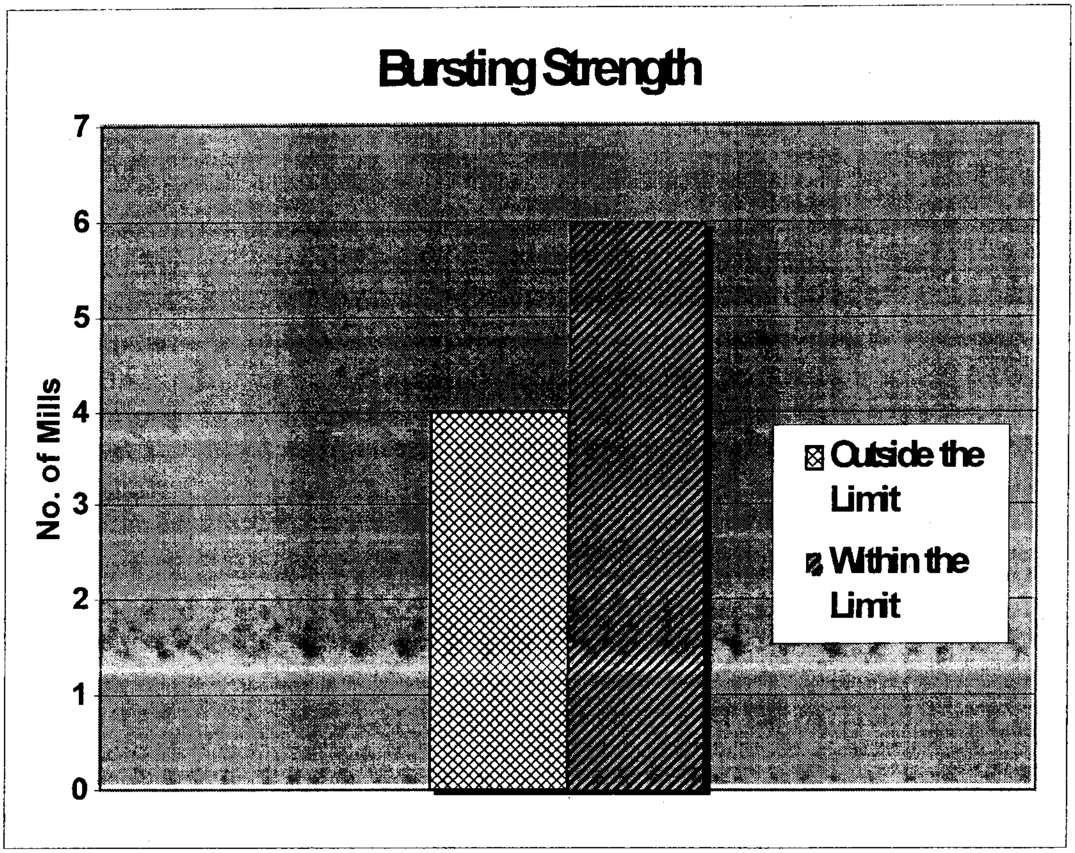


FIG. 8: BURSTING STRENGTH TESTING IN LABORATORIES OF DIFFERENT MILLS

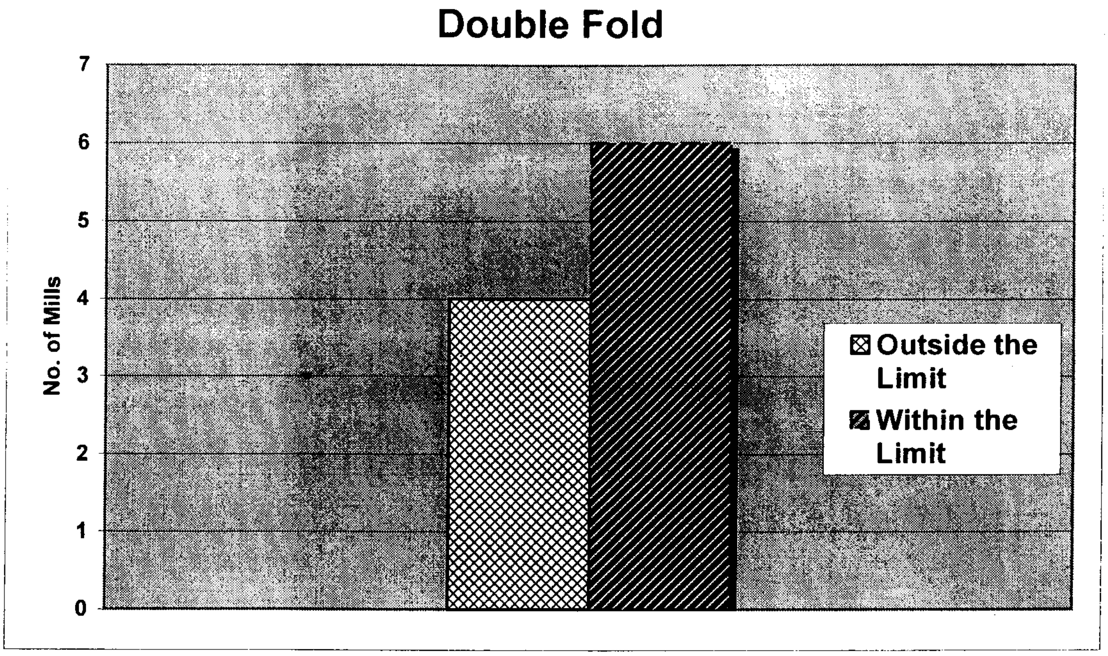


FIG. 9: DOUBLE FOLD TESTING IN LABORATORIES OF DIFFERENT MILLS

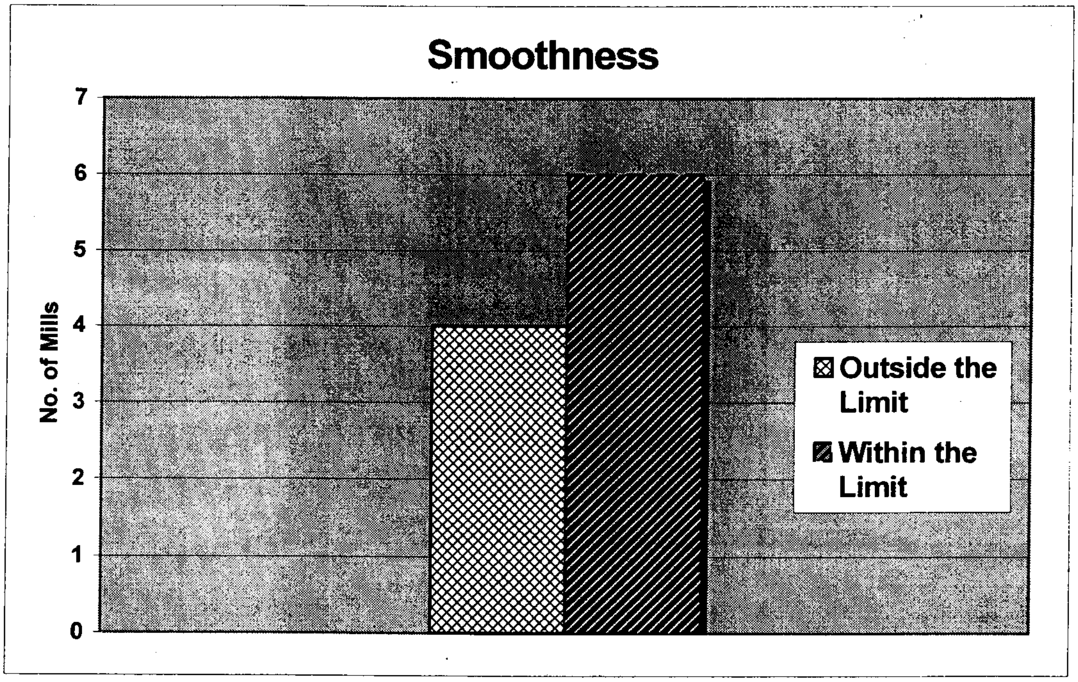


FIG. 10: SMOOTHNESS TESTING IN LABORATORIES OF DIFFERENT MILLS

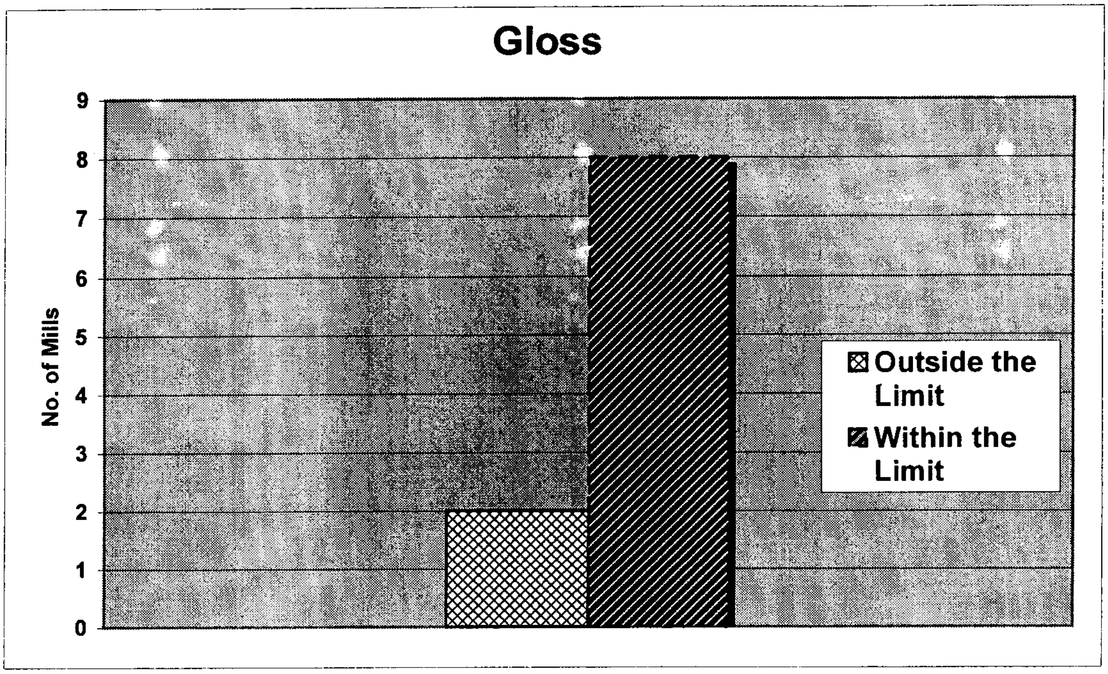


FIG. 11: GLOSS TESTING IN LABORATORIES OF DIFFERENT MILLS

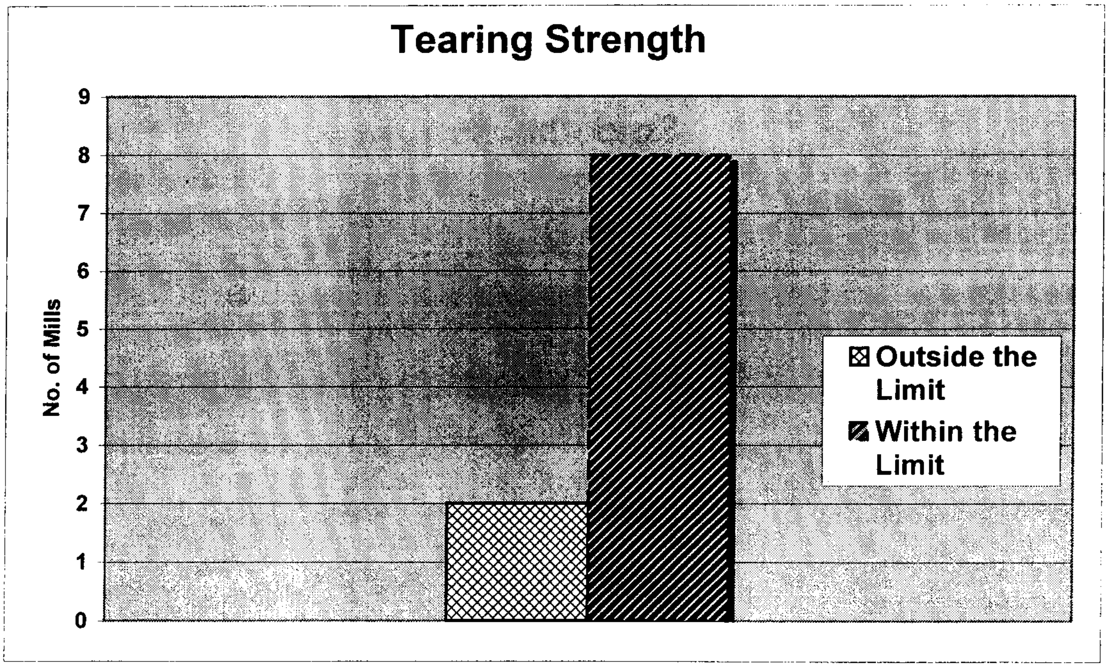


FIG. 12: TEARING STRENGTH TESTING IN LABORATORIES OF DIFFERENT MILLS

Results indicated that the major variation in results obtained is for the following properties

- Brightness
- Bursting Strength
- Double Fold
- Smoothness
- Thickness

The probable reasons for the variability may be as under:

8.1 Brightness measurement

Some of the mills still have not adopted the amendment issued for ISO standard test method. In the latest amendment it has been recommended that a part of UV reflection should also be included in the brightness value. It is important to realize that fluorescence from a fluorescent whitening agent is a blue radiation that influence the brightness value if the measurement is not equipped with an UV filter that eliminates the fluorescence. For this reason, brightness often differs between instruments. There is a currently (1997) a proposal to adjust UV content is illumination according the to the CIE illumination C when measuring the brightness of fluorescent materials. The new generation spectrophotometers have provision for this adjustment of UV content. There are three brightness values for fluorescent materials.

- C brightness (ISO brightness)
- D_{65} brightness
- Brightness with fluorescent eliminated
-

In paper trade different brightness are referred.

The designations used are:

Directional Brightness
Tappi T452 ($45^0/0^0$) GE brightness, IPC brightness

Diffuse brightness
ISO 2470 ($d/0^0$) Elrepho brightness, ISO brightness
CPPA brightness

No direct method of transforming values from the one to the other is possible. The difference is usually not greater than 2%.

8.2Bursting strength

Bursting strength is a complex paper property, which takes into consideration tensile strength, stretch as well as density of the paper. For this property the sheet clamping pressure is quite important and the bursting strength value is dependent on the clamping pressure. The mills are required to take appropriate action in this regard.

8.3Double fold

Double fold is very sensitive paper property. It depends on sheet structure, atmospheric condition, loading rate and the type of instruments. The mills are having different type of instrument viz. Kohler Molin, MIT, Schopper type. It is quite difficult to correlate all these instruments. The variation in this characteristic is always expected.

8.4Bendtsen smoothness

The variability obtained in this parameter is probably due to lack of auto clamping and damaged measuring head in the instruments. The manual handling of measuring head is more prone to operator's skill. The operator personal skill in putting the measuring head on the paper surface and time taken to read the reading affects the results.

8.5Thickness

Thickness is a critical measurement of the uniformity of paper or board today, when thickness is monitored by on-line equipment, it is vital to have an accurate thickness tester in the laboratory, in order to calibrate and verify the operation of the on-line equipment. In spite of this, the micrometer is one of the most overlooked instruments in many laboratories.

The micrometer should confirm to standard in:

- Lowering speed of the upper measuring face
- Contact pressure
- Good parallel alignment between the measuring faces

The conditions specified under different standard test methods are listed in **Table-5.**

TABLE -5

THE CONDITIONS OF MEASURING THICKNESS AS SPECIFIED UNDER DIFFERENT STANDARD METHODS.

Standard Method	Dead Weight Load, kg	Surface Size, cm^2	Lowering Speed, mm/s
SCAN P7	2	2	1
ISO 534	2	2	2
Tappi T411	1	2	0.8

The variation in the results obtained is probably due to different testers available within the laboratories. The variation observed was more in case of bulking thickness values.

8.6The Remaining Properties

Tensile strength, tearing strength and gloss — most of the mills have given satisfactory results.

9.0COMPARATIVE CALIBRATION SERVICES TO PAPER MILLS

CPPRI is providing comparative calibration samples on chargeable basis to mills on demand.

9.1Recommended Calibration Schedule for paper testing instruments

For proper functioning of the instruments, the schedule to be followed is given below

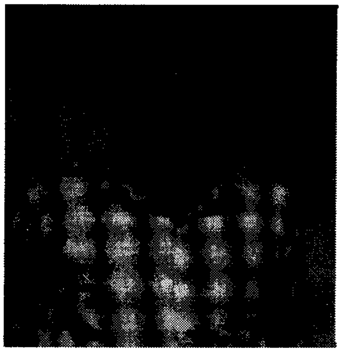
Parameter	Schedule
Thickness Tester	
Parallelism of jaws	Quarterly
Accuracy of gauge using standard samples	Quarterly
Lowering speed	Two years
Bursting Strength	
Diaphragm Condition	Monthly
Glycerin level	Monthly
Accuracy of gauge using standard samples	Quarterly
Clamping pressure	Quarterly
Check value in case of electronic pressure gauges	Quarterly

Tensile strength	
Load cell accuracy	Quarterly
Functioning of stretch measuring device	Quarterly
Checking load with standard samples	Quarterly
Double fold	
Jaws condition	Weekly
Number of strokes per minute	Half yearly
Checking with standard samples	Quarterly
Tear tester	
Pointer friction	Monthly
Clamping section	Quarterly
Pendulum factor	Half yearly
Knife cut	Quarterly
Values using standard samples	Quarterly
Brightness tester	
Calibration with standard plates	Daily
Calibration using standard samples	Quarterly
Bendtsen smoothness tester and Porosity tester	
Level of instrument	Weekly
Accuracy using standard samples	Quarterly
Gloss tester	
Calibration with standard plates	Daily
Verification with standard samples	Quarterly

10. References

1. Tappi T 404-os-76
2. Tappi T 403-os-76
3. Tappi T 411-os-76
4. Tappi T 423-su-68
5. Tappi T 414-ts-65
6. Tappi T 489-os-76
7. Tappi T 441-os-69
8. Tappi T 460-os-75
9. Tappi T 425-os-75
10. BS 4415 : 1969
11. ISO Recommendation ISO/R 1924-1971
12. Appita P 403 rp - 73
13. Appita 427 Ts - 72
14. Appita P 400 m - 73
15. Appita P 431 rp – 73

IMPORTANT PHYSICAL PROPERTIES OF PAPER



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IMPORTANT PHYSICAL PROPERTIES OF PAPER

P. C. PANDE

1. INTRODUCTION

The properties of paper are interrelated, when a change is made to improve one property, some other property often made poorer. The good papermaker tries to choose the middle ground where the combination of various properties gives the best paper. The physical properties include the common tests i.e. tensile, burst, tear, fold, stiffness, density, weight and thickness. These are the important properties of paper.

General physical properties of paper are following—

- Basic properties
- Strength properties
- Stiffness properties
- Structural properties
- Surface properties
- Absorption properties

2. SAMPLING

Before testing the paper sample it is necessary to obtain samples that are representative of the paper sample to be tested. Paper is delivered in rolls, the sampling uses the outer surfaces of roll. Determination of the number of rolls to be sampled uses statistical rules. According to the most frequently applied principle, the number of rolls sampled should be relative to the square root of the total number of rolls in the consignment. From each roll sampled, a few sheets of paper are cut with a minimum size of 300X450 mm. The sheets are combined into one sample that is randomized by careful mixing.

3. CONDITIONING

Moisture content has a very pronounced effect on the properties of paper. Fibers are hygroscopic, paper easily absorbs moisture from the atmosphere or releases moisture if atmosphere is dry. Moisture content of paper influences almost all its physical properties. Paper sample therefore requires careful conditioning before testing.

According to ISO 187, the conditioning climate is 50±2% RH and 23±1°C. For tropical countries, the conditioning climate is 65±5% RH and 27±1°C. Time of conditioning should be sufficient to bring the paper to a uniform moisture content which usually requires 4 to 12 hours.

4. BASIC PROPERTIES

The basic properties of paper are moisture content, basis weight, thickness (caliper), density, bulk and filler content.

The moisture content of paper is dependent on the relative humidity of the air in contact with it. Determination of moisture content of paper involves weighing a sample of paper before and after drying at 105±2°C (ISO 287). Basis weight is the weight per unit area of the paper and boards expressed as g/m^2. Weight affects all physical properties. Uniformity in the weight is also important

The normal expression for thickness of paper is 'µm' (micron). Measurement uses a thickness gauge with a paper sheet or a pad of sheets compressed with a given pressure between two parallel plates. The preferred pressure is 100 kPa (ISO 534). Uniform thickness is very important for printing papers.

Apparent density is the mass per unit volume of the paper calculated as the ratio between basis weight and thickness. The unit is g/cm^3.

Density of paper is important fundamental property. It is related to porosity, hardness and strength of paper. Density can be reported either as the apparent bulk density based on bulking thickness or apparent density based on the single sheet thickness. Bulk is the inverse of density.

Bulk =1/density, expressed as cm^3/g.

Determination of the filler content of paper involves igniting the sample at 900°Cor preferably at 525°C if the filler consists of $CaCO_3$ that decomposes at higher temperature.

In machine made paper, there are more fibers aligned in the machine direction than in cross direction of the paper. So this influences the properties of paper that therefore are different in different directions of paper sheet.

5. STRENGTH PROPERTIES

The important strength properties of paper are following—

- Tensile strength
- Bursting strength
- Tearing strength
- Folding endurance
- Surface strength
- Zero-span tensile strength

- Z – directional strength

5.1 Tensile strength

It is the maximum force per unit width that a paper strip can resist before breaking when applying the load in a direction parallel to the length of the strip. Measurement uses testers applying either a constant rate of loading or a constant rate of elongation for loading of the strip.

In the tensile strength tester, the test piece is stretched to the point where rupture occurs. The maximum tensile force the test piece can withstand before it breaks and the corresponding elongation of the strip are measured and recorded.

Tensile strength expression uses kN/m.

From the tensile strength measured, calculation of the tensile index uses the following formula—

Tensile index = Tensile strength/Basis weight
The unit of tensile index is Nm/g.

The elongation or stretch at break is the increase in length of the strip to its breaking point expressed in percentage of the original length. Tensile strength of a paper depends on fiber strength but primarily on the degree of bonding between fibers.

5.2Bursting strength

Bursting strength is the maximum pressure that a paper can resist without breaking with pressure applied perpendicular to the plane of the test piece. A unit for bursting strength is kilopascal, kPa. Calculation of the material related burst index uses the following formula—
Burst index= Bursting strength/Basis weight

Burst index is expressed in kPa-m^2/g.

The most common tester used for bursting strength measurement is the Mullen tester. A test piece placed over a circular elastic diaphragm is rigidly clamped at the periphery but free to bulge with the diaphragm. The hydraulic fluid pressure increases by pumping at a constant rate to bulge the diaphragm until test piece ruptures. The bursting strength of the test piece is the maximum value of the applied hydraulic pressure.

5.3Tearing strength

Tearing strength is the mean force required to continue the tearing of paper from an initial cut in the sheets. If the cut is in the machine direction, results will be obtained as machine direction tearing resistance. The tearing strength is highly dependent on the fiber orientation of the sheet.

The unit for tearing strength is Newton (N) or milli Newton (mN). From tearing strength, calculation of the tear index uses the following formula—

Tear index= Tearing strength/ Basis weight
Tear index units are mN-m^2/g.

The most frequently used single tear tester is of the Elmendorf type. The work done in tearing the test piece is measured by the loss in potential energy of the pendulum. The tearing strength of a paper depends on the following factors—

- Fiber length
- Fiber strength
- Degree of bonding between fibers
- Degree of orientation of fibers in the paper

Longer and stronger fibers provide higher tearing strength

5.4Folding Endurance

Folding endurance or folding strength is the ability of a strip of paper to resist breaking when folded under a certain load. The folding strength is expressed directly as the number of double folds that the paper can withstand. Folding strength relates to the tensile strength, elasticity and elongation of the paper. Higher tensile strength and elongation of a paper shows higher folding strength.

The most common folding testers are the following—

- Schopper
- MIT
- Kohler Molin
- Lhomargy
-

Folding strength indicates the effect of paper aging. It is important test in testing endurance of banknote paper.

5.5Zero-span tensile strength

This test indicates the strength of the individual fibers rather than the strength of the paper itself. Normal tensile testing indicates the combined effect of the strength of the fibers, then bonding and variation in the structure of paper. The zero-span tester is equipped with special clamps to bring the span length as close to zero as possible. The Pulmac tester was developed by Cowan & Cowdrey for zero-span testing. Careful zero-span testing requires testing at several different span lengths. The zero-span tensile strength value is a good indicator of the average strength of individual fibers. It is therefore a useful test to indicate the effect of pulping processes, beating etc. on the fiber strength.

5.6 Surface strength

The surface strength of the paper is the ability of paper to resist a force pulling out fibers or fiber bundles from its surface. A low surface strength may create linting problem and runnability problem.

Methods of testing surface strength of paper usually use high viscosity, tacky oils or printing inks. This unit prints the paper at a continuously increasing speed using a very tacky oil. The printing speed at which picking of the surface begins is measured. The product of this speed and viscosity of the oil is a measure of surface strength of the paper.

Another test is Dennison wax pick test. In this test sealing wax sticks with different picking values are fastened on the paper by melting. The wax number at which the paper surface does not break when removing the wax piece indicates the surface strength.

5.7Z-directional strength of paper

Z-directional strength refers to the ability of paper to resist tensile loading in a direction perpendicular to the plane of paper (z-direction). Paper or board will often be loaded in the z-direction. Such cases are offset printing and certain converting operations such as corrugating, folding and plastic coating.

In a direct measurement of the z-directional strength of the paper sample is mounted with double sided tape or glue between two metal pieces which are forced together under load to ensure that plates are well attached to the paper. This assembly is mounted in a tensile tester and loaded until the sheet splits. The z-direction strength is the load at break divided by the area of sheets.

6. STIFFNESS PROPERTIES

Stiffness relates to the elastic properties of a material. Two types of stiffness are usually measured—

- Tensile stiffness
- Bending stiffness

6.1Tensile stiffness

For tensile stiffness, the external load acts parallel to the plane of the sheet. Tensile stiffness can be determined from the results of the tensile test. It is described by the following mathematical formula—

$T_s = (\Delta F/b) \times (l_0/\Delta l)$

where, 'T_s' is tensile stiffness

'ΔF' is tensile stress

'b' is width of the test piece

'Δl' is length increase of test piece

'l_0' is the original length of test piece

6.2Bending stiffness

Bending stiffness describes the ability of a paper or board strip to resist a bending force applied perpendicularly to the free end of a strip clamped at the other end. The force required to bend the strip to a specified angle is the bending resistance. The strip dimensions and the bending angle remain constant.

6.3Stiffness by the resonance method

The principle of the resonance stiffness method is that the natural vibrating frequency of a material during free vibration depends on the bending stiffness, length of freely vibrating sample and weight of material. In resonance method the test piece is clamped at one end and forced to vibrate at a constant frequency. The length of the free end of the test piece varies until it goes into resonance with this frequency and this length is measured. The bending stiffness can be calculated from the resonance length, frequency and basis weight of the material according to the following formula—

$S = 20\,(L/100)^4\,(W/100)$

Where,

'S' is bending stiffness, mNm

'L' is resonance length of the strip, mm

'W' is basis weight, g/m^2

7. STRUCTURAL PROPERTIES

Basis weight, thickness, density or bulk and air permeance are among the important structural properties of paper. The first three properties have already been described.

The air permeance of paper is normally measured using the air leakage instruments of different types. These instruments measure the flow of air through a defined area of paper.

The common air permeance testers in use today are the following—

- Bendtsen and Sheffield testers which measure the air flow in milliliters per minute
- Gurley tester, which measure the time required for 100 ml of air to flow through the paper sample.

8. SURFACE PROPERTIES

The most important surface properties of paper are—

- Roughness or smoothness
- Surface strength

8.1Roughness or smoothness

The principle generally applied for this purpose measures the ability of paper surface to resist an air stream flowing between the paper surface and an edge (measuring head) pressed against it. The features of this measurement are the following—

- Pressure difference used to create the air flow
- Pressure with which the measuring head presses against the paper surface
- Area of the measuring head pressed against the paper

The result obtained is directly reported as an indication of paper smoothness or roughness.

The most common testers used are the following—

- Bendtsen tester measuring roughness
- Sheffield tester measuring roughness
- Parker Print Surf measuring roughness
- Bekk tester measuring smoothness

9. ABSORPTION PROPERTIES

The absorption properties are important for printing of the paper. Many methods are available for testing these properties. An important requirement of all these tests is that the medium used for absorption into the paper should simulate as closely as possible the end use situation.

The means used are the following—

- Water
- Oil of different composition and viscosity
- Printing inks of different types

The most common absorption test is 'Cobb test' and 'Klemm test'. The 'Cobb test' measures the amount of water absorbed by the sample during a certain time from an excess of water in g/m^2. This test has use primarily as an indicator of the degree of sizing, i.e. the water repellency of the paper.

The 'Klemm method' measures the capillary rise of the water in a strip of paper perpendicular to the water surface.

The 'absorbency test' is IGT oil absorbency test. A drop of oil is applied to the paper surface. The absorbency of printing inks is measured with 'Printability tests'.

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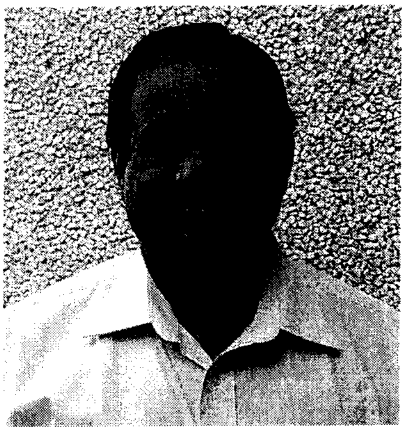
EVALUATION OF PULPS FOR PHYSICAL AND STRENGTH CHARACTERISTICS



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EVALUATION OF PULPS FOR PHYSICAL AND STRENGTH CHARACTERISTICS

Y. V. SOOD, S.TYAGI & A. KUMAR

1. INTRODUCTION

To control the quality of any industrial process it is important to evaluate the raw material, end products and intermediate manufacturing processes. To do so an efficient, accurate and relevant testing is needed and its importance has increased now a days due to global competition and the processes becoming sophisticated with tighter tolerance variation. For any paper grade, pulp quality is the major contributing factor. So it becomes necessary that the pulps are evaluated for various characteristics according to standard procedures. The importance of such evaluation become more important when a paper mill is using purchased pulp and paying substantial amount for it. Basically there are two broad objectives for pulp evaluation.

- Testing the quality parameters of specific pulp
- Comparing the paper making potential of different pulps.

2. PULP EVALUATION METHODS

Pulp evaluation methods have been formulated by different organizations like ISO, SCAN, TAPPI, and APPITA. The list is given in **Table - 1** . All these standards are similar in their main principles but differ in some details. One of the main differences amongst them is the atmospheric conditions used during testing. Different standard atmospheric conditions have been prescribed in different standards (**Table - 2**).

3 SAMPLING OF PULP FOR TESTING

For proper testing representative sampling of pulp is important. The selection of sampling method and actual sampling must be carefully done keeping in view that pulp itself is heterogeneous in nature due to variation in wood quality, which is changing from time to time in the process. The sample should be stored properly.

It depends on pulp type. Mechanical pulp slurry starts to deteriorate at room temperature in matter of days. Even in cold room conditions, the storage time has limitations. Chemical pulps can be stored for longer time especially if it is bleached. For longer storage one possibility is freezing, but properties of chemical pulp change in the freezing and defrosting process (1,2). With mechanical pulp, freezing changes are minimum. The storage time can also be extended using biocides. Small dosage of formaldehyde may be used to prevent degradation. Pulp dried to 90% of solid can be stored for long period at room temperature.

4. DISINTEGRATION OF PULP

Proper disintegration of pulp is important for the correct evaluation of its characteristics. Dried pulp requires soaking before disintegration. The soaking time depends on the dry matter contents in the pulp. Higher dry content necessitates longer soaking time. Different disintegration conditions have been prescribed in ISO 5263-1995 (E) standard for different pulps (Table III). Mechanical pulps often require hot disintegration to remove latency. Hot disintegration at temperature approximately 85°C is used for mechanical pulps.

5. DIFFERENT TYPES OF LABORATORY BEATERS AVAILABLE

The common laboratory beaters used in pulp evaluation are

- PFI mill
- Valley beater
- Jokro mill

Some of the features of these beaters are

5.1PFI mill

In PFI mill beating occur between a bar roll and a smooth beater housing both rotating in the same direction but with different speed. The beating consistency is 10% compared with normal low consistency refining of about 2 to 5% in paper mills. The advantage with PFI mill is that single beating uses small amount of pulp i.e. 30 g. So it is possible to evaluate small quantity of pulp.

5.2Valley Beater

Valley beater has a beater roll and bed plate with loaded lever arm, which controls the beating. Pulp consistency in the beater is about 1.5 %. The benefit of the valley beater is that the large amount of pulp in beating (360 g OD) which allows sampling from the beater at selected time interval to provide a complete beating using a single beating.

5.3Jokro mill

The Jokro mill is similar to PFI mill by having no counter bars in the beater housing. Beating occurs between bar surfaces and smooth housing. Pulp consistency is 6%. Jokro mill has six beating chambers each taking 16 g OD pulp.

6. BEATING ACTION IN DIFFERENT LABORATORY BEATERS

The beating action is different in all these three beaters. The main differences are listed in table IV.

In many cases the PFI mill and valley beater classify pulp in the same order, but in some cases the results differ (3,4,5).

7. DESCRIPTION OF LABORATORY HANDSHEET FORMER (BRITISH HANDSHEET MAKING MACHINE)

7.1 Principle

A circular square or rectangular sheet is formed from a pulp suspension on a wire screen under suction. The sheet is subjected twice to a pressure of 410 kPa. The sheet is dried in conditioned air and in contact with a drying with a drying plate, to which it adheres so that it

7.2Equipment

The equipment **Sheet former** mainly consists of three main parts.

I. Upper section

Upper section comprising a stock container, with a mark located 350 mm ± 1mm above a wire screen is furnished with a rubber gasket to prevent leakage. The cross-section of the container shall be circular, square or rectangular, and constant throughout the height. If the container is rectangular, the shorter side shall be not less than 120 mm and the ratio of the longer to the shorter side shall not exceed 2.5. if the container is circular it shall be not less than 158 mm in diameter. It shall be of such height that water will not splash over the edge when the stirrer is operating.

II. Lower section

Lower section comprising a drainage vessel, consists of an upper and a lower part. The upper part shall have the same cross section as the stock container and its shape shall be such that the flow of liquid through the wire screen is uniform over the whole area. The lower part may be of smaller cross-section but shall be positioned symmetrically in relation to the upper part. The lower part shall be fitted with a valve, which is connected to a draining pipe with a water seal at its lower end. The vertical distance from the top of the wire screen to the overflow of the water seal shall be 800 mm ± 5 mm. the lower part and the drainage valve shall be large enough to permit water in the stock container between the level mark and the wire screen to empty within 4.0s ± 0.2 s

III. Frame

with a perfectly flat plain-woven metallic wire screen, to be placed horizontally between the upper section and the lower section. The screen shall be clean, undamaged and fitted without wrinkles and corrugations. It shall have a nominal size of aperture of 125 um,

7.3 Stirrer

It is made of any non-corroding rigid material consisting of a perforated plate and furnished with vanes to keep the plate parallel to the wire screen and to minimize swirling during stirring. The total area of the holes (diameter 10 mm to 20mm) shall be about 30% of the area of the plate, the holes shall be evenly spaced. The dimensions of the plate shall be such that there is a clearance of 2 mm to 3 mm between the plate and the stock container.

7.4Couch roll

(mass 13.0 kg length 178 mm, diameter 102 mm) and a couch plate to protect the sheets.

7.5 Blotters

These are made of fully bleached chemical pulp or rag pulp having neutral pH, and free from sizing agents. Chemical additives visible contraries and fluorescent contaminant. The grammage of the blotters shall be 250g/m2 ± 25 g/m2, the klemm absorbency measured in accordance with shall be 70 mm ± 20 mm and the dimensional changes caused by soaking.

7.6Drying plate

It is of the same size as the formed made of corrosion-resistant metal or another suitable material such as rigid plastic glazed or polished on at one side.

7.7 Press

It is capable of exerting a uniform pressure of 410 kPa ± 10 kPa over the area of a laboratory sheet and of maintaining this pressure for 5 min. the maximum number of laboratory sheets to be pressed simultaneously shall be adjusted to the capacity of the press.

8. PREPARATION AND EVALUATION OF THE HANDSHEETS

The handsheets can be prepared and evaluated by any standard test method like ISO, TAPPI, APPITA or SCAN. Depending on the model of sheet former round or squared sheets are formed. In some places Rapid Kothen Sheet Former is used. The properties achieved with a Rapid Kothen paper Former differ to some extent from the results with the conventional method (10). When comparing the pulp evaluation results with paper from paper machine one should remember that there is clear difference between handsheets and paper from paper machine due to foaming step. The orientation of fibre in handsheets is isotropic whereas in case of paper on paper machine it increases from upper side to wire side. The fibres content in the wire side of handsheets is more whereas it is reverse in the case of paper made on paper machine. To get proper results it is important that the number of replications for each test is sufficient. ISO standard has prescribed the minimum number of replication for each test, which is given in table V. To get these the practice of cutting handsheets being followed in CPPRI is given in **Fig. 1.**

9. INTERPRETATION OF PULP EVALUATION RESULTS

To get more information from the pulp evaluation results it is important that comparison is made properly. The practice of comparing the strength properties of pulp at constant freeness has been criticized for many years. The shortcomings of freeness test have been summarized by Clark (6). He has proposed the use of sheet density as a more useful basis for comparison. The reason for this choice has been summarized in reference (7). Rydholm (8) has suggested that the relationship between tensile and tear is also a satisfactory indication of pulp strength and usefulness has been further demonstrated by Mcleod (9).

As we know that the main purpose of beating is to improve the wet conformability of the paper making fibres. At the same time, specific bond strength, fines contents, fibre strength and stretch properties may be altered. Whether this alternation is desirable or otherwise depends on the end use of the paper. The effect of processing conditions e.g. pulping, beaching, refining on the fibre and pulp properties or their inter relationship can be estimated from the results of a normal pulp evaluation. The ways of obtaining such information are

- The three parameters which are not affected by intrafibre bonding (apparent density, air resistance and freeness) are all primarily controlled by wet fibre conformability the property level being modified by the fibre content. As wet fibre conformability increases, the fibre mat is more readily compacted, both by drainage forces and by wet pressing. This leads to an increase in sheet density and to a reduction in effective pore diameter. As the flow of fluids through porous media is much more influenced by pore diameter then by pore length, the overall effect of reducing sheet thickness and pore diameter is to reduce the permeance of the fibre mat, both to air and to water. The presence of fibre in the pulp produces significant changes in all these properties. The voids in the sheet are filled by small patches, giving an increase in sheet density and reduction in effective pore diameter. Keeping this in view by plotting a graph between log (air resistance) against sheet density the relative rates of development of conformability and fines production in pulp can be assumed. The way, in which the relationship is changed, either by changes in pulping or bleaching conditions or by modification to the beating process can be seen from such graph. For example in **Fig. 2**. for treatment. There is greater fine production for a given degree of development of wet fibre conformabilities than treatment B.

- By plotting graph between tensile index and sheet density the changes in the specific bond strength of the pulp can studied. Most of the mechanical strength properties of paper like tensile index, tear index, burst index, stretch, folding endurance are largely dependent on inter fibre bonding although modified by other factors such as fibre length, fibre strength etc. Fibre coarseness is also important but it is not affected much by process conditions. The effect of inter fibre bonding is related to the inter fibre contact area and to the specific bond strength. When comparing the pulp with fibres of approximately in same dimensions one may assure that the area of inter fibre contact can be related to fibre conformability and hence to sheet density. Therefore, change in specific bond strength will lead to a change in tensile strength and sheet density. The effect of fibre strength on the paper strength/sheet density relationship becomes more evident as inter fibre bonding increases. In a well bonded sheet, failure may occur as a result of fibres breaking than through the breaking of inter fibre bonds. If the fibres have been weakened, the probability of fibres breaking is higher. In these circumstances the strength of a well bonded sheet will be lower than it would be. Inter fibre bond breaking was the sole factor controlling strength.

Consider **Fig. 3** which shows the relationship between tensile index and sheet density for three pulps A, B, C. The tensile index in case of B and C is higher which shows belle bond strength. From the difference in slopes it is possible to get information that whether improvement in bond strength will help to improve the bonding properties or not. If the difference in slope is less then initial bond strength is already higher which indicates less improvement is possible.

The relationship between tensile/tear index provides a very sensitive indication of fibre damage especially in long fibre pulps. The damage may be brought about by pulping or beaching or by refining. **Fig. 5** illustrate that pulp B is better than A.

By plotting stretch against tensile index it is possible to detect the effect of processing correlation on the development of stretch by mechanism other than a simple increase in inter fibre bonding. Papers such as bag and sack krafts and kraft wrapping are often required to have high tensile energy absorption-a combination of tensile strength and stretch. The stretch is largely controlled by inter fibre bonding. An examination of tensile index/stretch relationship will identify. Those pulps likely to give a superior product because of belle stretch characteristics at a given level of inter fibre bonding. Pulp B is better than pulp A. The advantage of using the tensile index/stretch relationship for purpose of comparison is that the basic effect of increasing inter fibre bonding on stretch may not mark its effect on other factors which influence stretch properties.

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TABLE –1

DIFFERENT STANDARDS FOR THE EVALUATION OF PULPS

Content	ISO	SCAN C chemical pulp M Mechanical pulp	TAPPI	APPITA
Sampling for Testing	7213			
Dry matter content	638	C 3	T 412	
Stock concentration	4119	C 17, M 1	T 240	
Standard water for physical testing	14487	CM 58		
Laboratory wet disintegration	5263	C 18, M 2	T 205	
Hot-distigration of mechanical pulp		M 10	T 262	
Fibre length by classification (McNett)		M 6	T 233	
Fibre furnish analysis	9184	G 3, G 4	T 401	
PFI-mill beating	5264-2	C 24	T 248	
Valley-beating	5264-1	C 25	T 200	P 209-TS 75
Jakro-mill beating	5264-3			
Canadian Standard Freeness	5267-2	C 21, M4	T 227	
Schopper-Riegler	5267-1	C19, M 3		
Sheet forming ➢ Convential method ➢ Rapid Kothen method ➢ Sheets for diffuse blue Reflection factor (ISO brightness)	 5269-1 5269-2 3688	 C26, M5 CM 11	 T 205 T 218	 P 203-M 75
Measurement of diffuse blue reflectance factor (ISO brightness)	2470	P 3	T 452 T 525	
Standard atmosphere for conditioning and testing	187	P 2	T 402	
Laboratory sheets ➢ Determination of physical properties	5270	28, M 8	T 220	P 208-M 75

ISO- International Organization for Standardization
SCAN- Scandinarran Pulp, Paper and Board
TAPPI- Technical Association of the Pulp and Paper Industry (USA)
APPITA- Australian Pulp and Paper Industry Technical Association

TABLE - 2

ATMOSPHERIC CONDITIONS PRESCRIBED IN DIFFERENT STANDARDS FOR PAPER TESTING.

Country	Standard method	Relative humidity (%)	Temperature °C
Australia	APPITA P 415 m-73	65±2	20±1.7
Britain	BPBMA P T3 Sm-1951	65±2	20±1.7
Canada	CPPA A, 4; June 1973	50±2	23±2
France	AFNOR NF Q03-010, March 1958	65±2	20±2
Germany	DIN 50014, Dec. 1959	65±2	20±1
Holland	HCNN N 1108, May 1951	65±2	20±5
India	BIS 1060 (Part I)	65±2	27±2
Scandinavian countries	SCAN P 2:61	65±2	20±1
		50±2	23±1
U.S.A.	TAPPI T 402 OS-70	50±2	23±2
USSR	SSSR GOST 7497-55	65±2	20±5
International	ISO 187-1961	50±2	23±1
		or 65±2	27±1
		or 65±2	20±1

TABLE - 3

CONDITIONS TO BE USED IN STANDARD PULP WET DISINTEGRATION APPARATUS FOR PULP EVALUATION AS PER ISO 5263-1995 (E) METHOD

Kind of pulp	Dry matter content	Pulp weight OD (g)	Minimum soaking time	Disintegration volume	Number of revolns.
Chemical	<20	30	-	2000	10000
	20 to 60	30	30 min	2000	30000
	>60	30	4 h	2000	30000
Mechanical	<20	60	-	2700	10000
	20 to 60	60	30 min	2700	30000
	>60	60	4 h	2700	30000

TABLE –4

DIFFERENCE IN BEATING ACTION IN PFI MILL, VALLEY BEATER & JOKRO MILL

Action	Valley beater	PFI mill	Jokro mill
Removal of primary walls/outer layer	Main	Less intense	Less intense
Internal and external fibrillation	Little	More	More
Fibre breakage	Significant	Less	Less

TABLE –5

MINIMUM NUMBER OF SHEET PIECE TO BE TESTED FOR DIFFERENT CHARACTERISTICS

Property	Standard method	Target grammage	Test piece diameter (mm)		Minimum number of test piece
			Length	Width	
Tensile Index	ISO 1924-2	60 or 75	100±2 between clamps	15±0.2 0.1	8 from at least 4 sheets
Tear Index	ISO 1974	60 or 75	According to apparatus		$2^{(1)}$ from at least 4 sheets
Burst Index	ISO 2758	60 or 75	Wide enough to securely clamped		8 from at least 4 sheets
Folding endurance	ISO 5626	60 or 75	According to apparatus 15±0.1		6 from at least 3 sheets
Resistance to beating	ISO 2493	(2)	≥70	38±0.2	6 from at least 2 sheets
Flat crush resistance	ISO 7263	(2)	≥150	12.7±0.1	10 from at least 2 sheets
Compresion-Ring	ISO 12192	(2)	150 to 152.5	12.7±0.1	10 from at least 2 sheets

(1) One test piece consists of four cut from at least two trimmed sheets

(2) Unspecified

TABLE – 6

AS PER ISO 5270-1998 (E) THE DIFFERENT PROPERTIES SHOULD BE EXPRESSED AS

S. No.	Property	Formula	Units of Expression	Report to nearest
1	Apparent Density	$\frac{\text{Wt. of sheets (g)}}{\text{Area (m}^2\text{) x no. of sheets}}$	G/cm^3	Two significant figures
2	Tensile Index	$\frac{\text{Tensile (N)}}{\text{Width of sheet (m) x grammage}}$	N.m/g	0.5
3	Tear Index	$\frac{\text{Scale Reading (mN) x factor}}{\text{Area (m}^2\text{) x grammage}}$	MN.m^2/g	0.1
4	Burst Index	$\frac{\text{Bursting strength (kPa)}}{\text{Grammage}}$	KPa.m^2/g	0.1
5	Air permeance Gurley	$\frac{127}{\text{t (s/100)}}$	$\frac{\text{Micrometer}}{\text{Pascal second}}$	Two significant figure
6	Folding endurance	Log (Fold number)	-	Two significant figure
7	Bending resistance index	$\frac{\text{Bending resistance (mN) x } 10^6}{(\text{Grammage})^3}$	Nm^6/kg^3	Three significant figure
8	Flat crush resistance index	$\frac{\text{Flat crush (N)}}{\text{Grammage}}$	N.m^2/g	Three significant figure
9	Ring crush resistance index	$\frac{\text{Ring crush (N)}}{\text{Length (m) x grammage}}$	N.m^2/g	Three significant figure

TABLE –7

CHARACTERISTICS OF SOME PAPER MAKING BLEACHED PULPS AT 350±50CSF

Raw material	Tensile Index (N.m/g)	Burst Index (kPa.m^2/g)	Tear Index (mN.m^2/g)	Sp. Scat. Coff. (m/kg)
Mixed hard wood	48-64	2.8-4.3	8.1-9.0	34.2-36.3
Eucalyptus	60-78	3.2-4.9	7.0-10.0	36.0-42.0
Bamboo	52-60	3.0-3.9	15.5-17.0	27.0-29.2
Softwood Imported	70-80	4.9-6.0	11.5-15.0	20.7-22.1
Wheat straw	42-48	2.6-3.0	5.2-5.4	26.0-28.0
Rice straw	40-47	2.5-2.7	4.8-5.0	42.0-44.1
Bagasse	45-54	2.9-3.2	5.4-5.6	20.6-22.8

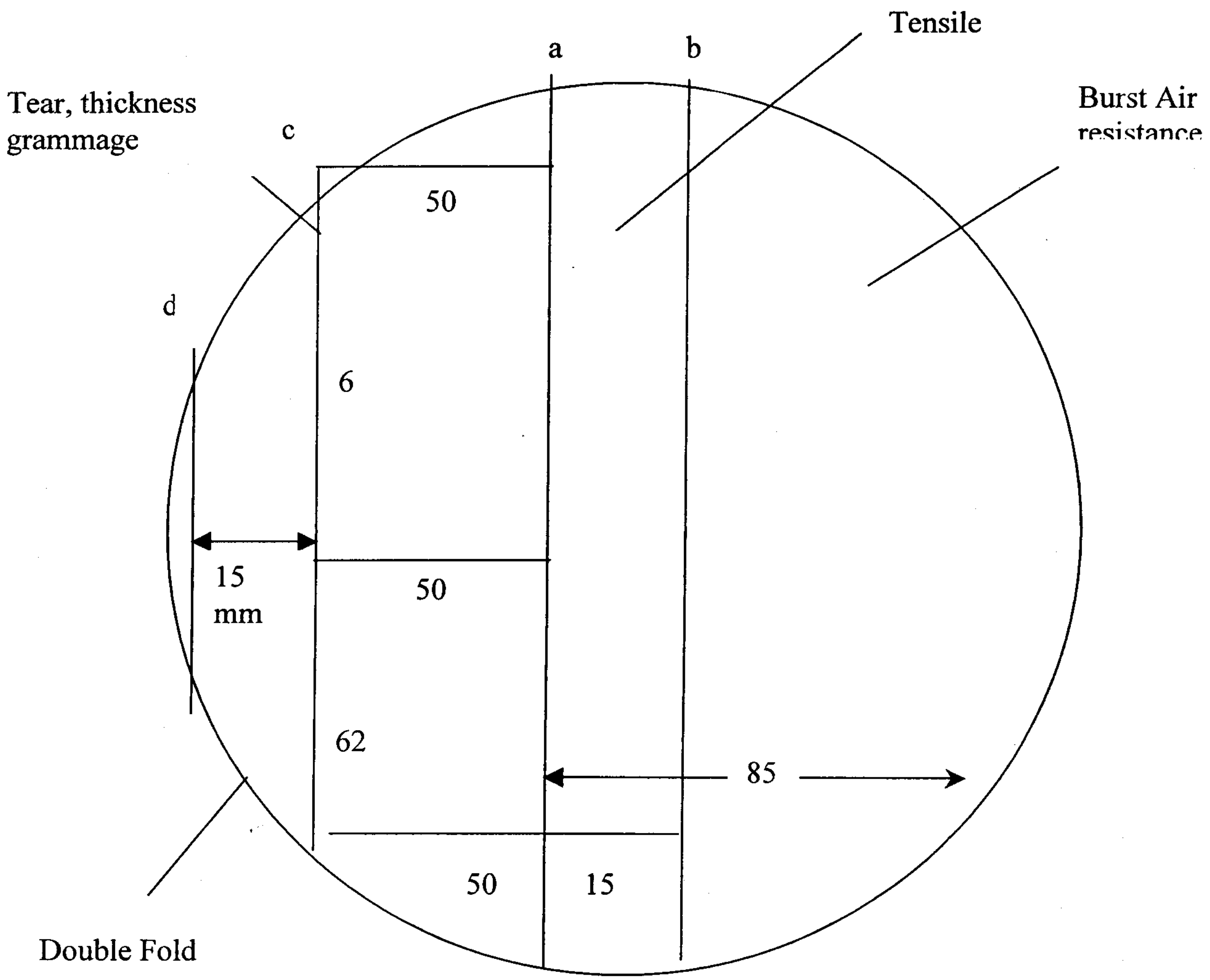


FIG. 1: CUTTING OF HAND SHEETS FOR TESTING
(a) FIRST CUT AT 85 MM
(b) SECOND CUT
(c) THIRD CUT (15 MM)
(d) FOURTH CUR (15 MM)

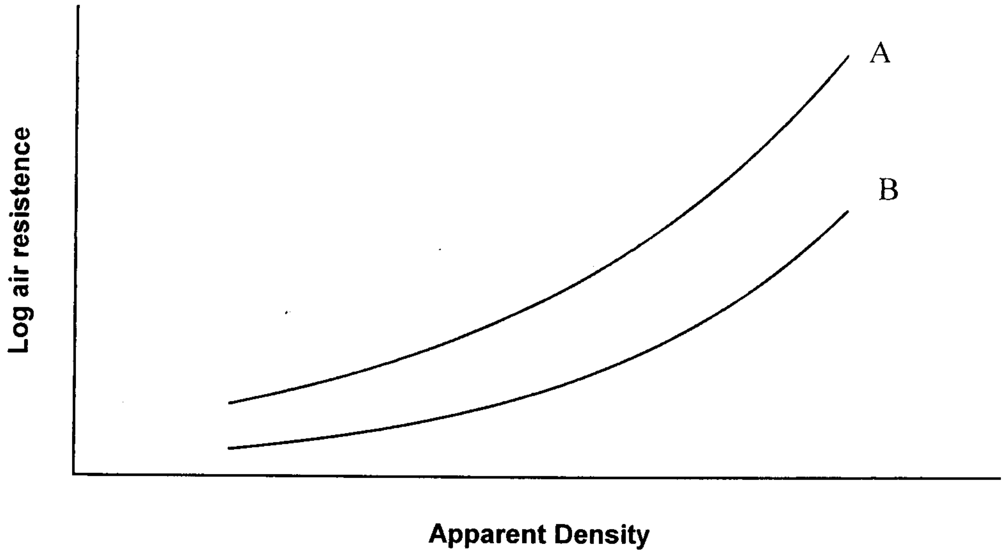


FIG.2: GRAPH BETWEEN LOG AIR RESISTANCE AND APPARENT DENSITY FOR PULP A AND B

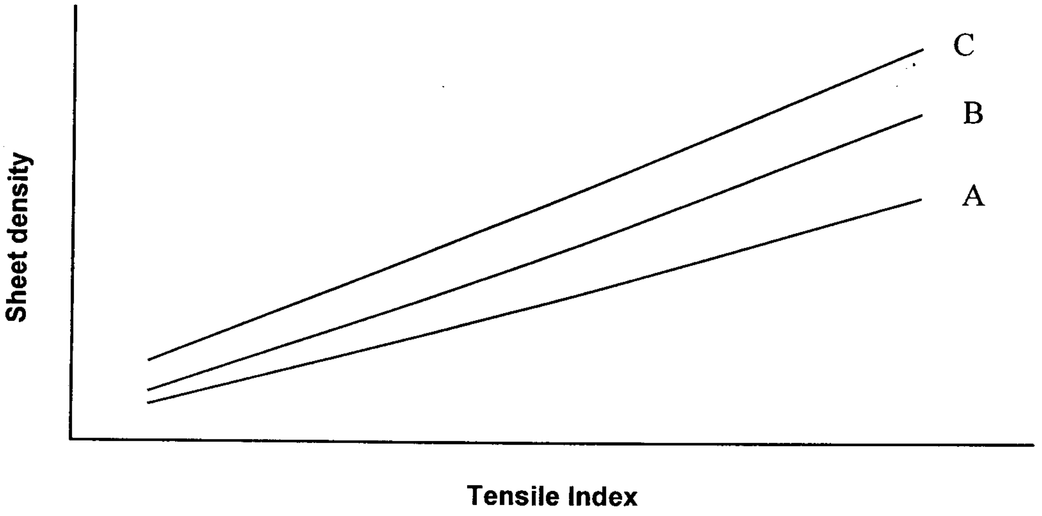


FIG.3: GRAPH BETWEEN TENSILE INDEX AND SHEET DENSITY FOR PULPS A, B AND C

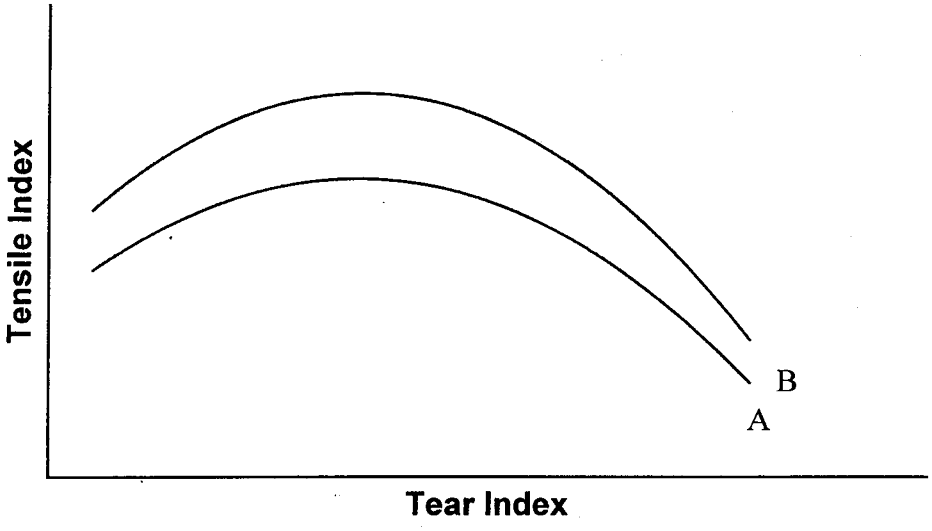


FIG. 4: RELATIONSHIP BETWEEN TENSILE INDEX AND TEAR INDEX

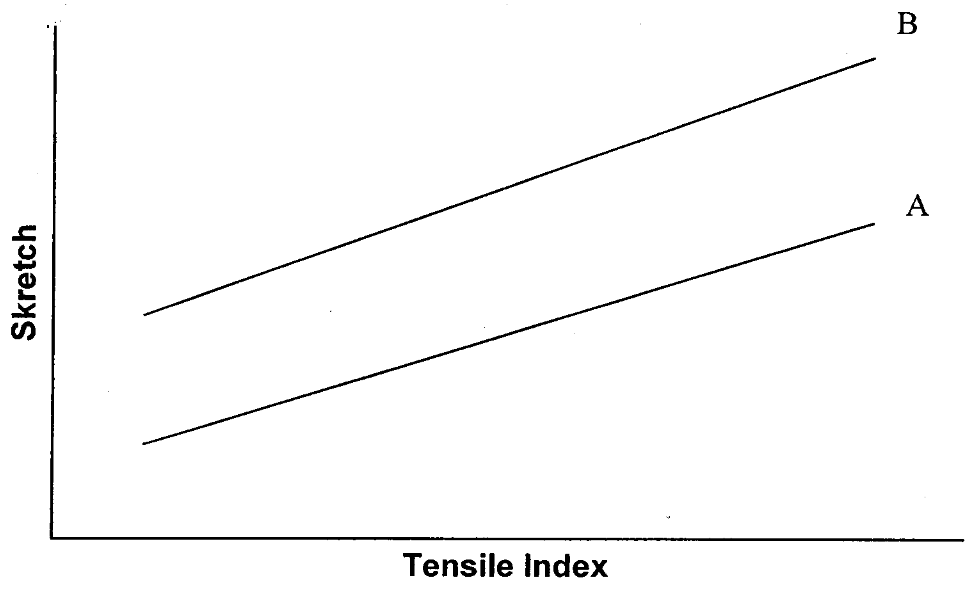
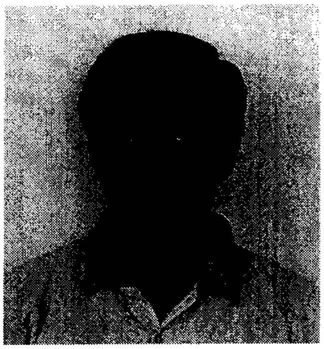


FIG.5: RELATIONSHIP BETWEEN STRETCH AND TEAR INDEX

MEASURING / STUDYING / TESTING OF PULP DRAINAGE



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MEASURING/STUDYING/TESTING OF PULP DRAINAGE

S. TYAGI & I. PAYRA

1. INTRODUCTION

The manufacture of paper consists essentially of removing water from a diluted suspension of fibre and other materials, leaving the solids in form of a coherent sheet on a fine mesh screen. This removal of water from fibre suspension is known as 'drainage'. Sheet forming is considered to be a combination of three hydrodynamic processes viz. drainage, oriented shear and turbulence, which occurs simultaneously and are not quite independent of each other– thus making the process very complicated. A further complication arises due to chemical interactions between fibres, fillers, fines and other chemical additives present in the pulp suspension. The response of a particular furnish to a wet end additive depends on the specific polymer used, presence of other additives and operating conditions. For example, addition of a low-molecular-weight high-charge-density cationic polymer to a pulp can improve the gravity drainage and decreases the vacuum drainage while high-molecular-weight low-charge-density cationic polymer may help wire dewatering. However a wetter sheet then leaves the press section. It is therefore, very important for the papermaker to attain optimal drainage at paper machine. Optimal drainage allows a broad variety of opportunities as under.

- Reduction in energy cost for water removal
- Increased runnability due to increased wet web strength
- Study of drainage during sheet formation helps the papermaker to optimize the wire section operation from both formation and retention viewpoints
- Reduction of forming fabric wear

A good drainage system at paper machine is therefore always desired and that is why laboratory study of drainage is so important for papermakers.

2. BASIC SHEET FORMING PROCESS BY DRAINAGE

Formation of wet mat of fibre in a paper machine (or in a sheet former) is considered to proceed according to following mechanisms (**Fig. 1**) depending upon the state of fibre mobility existing in the undrained suspension.

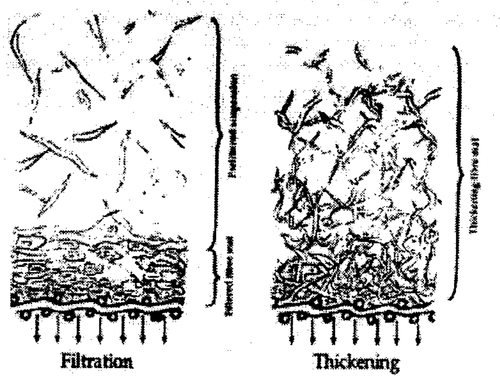


FIG. 1: DRAINAGE MECHANISMS– FILTRATION AND THICKENING

2.1 Filtration

It occurs when suspended fibres are mobile or free to move independently of one another. Thus a sharp boundary develops between the concentrated mat deposited on the mesh and dilute suspension approaching it and the concentration of undrained suspension remains essentially constant. After the formation of mat, high retention and slow drainage result. Use of drainage aids increases filtration rate.

2.2 Thickening

Thickening occurs when the fibres in suspension are entangled together to form a coherent network. This network is a compressible solid structure which steadily collapses as drainage proceeds. Water is removed simultaneously from all parts of the suspension during drainage and fiber concentration increases from top to screen mesh. This results a quicker drainage and lower retention and when aids are used, there is no change or even a loss in drainage.

There are different theories as to the main mechanisms of wet mat formation by drainage. However in general it is believed that filtration is predominant mechanism for machines running at lower speed and thickening process prevails in high speed ones.

During formation of sheet, mechanical entanglement of fibres, fillers and additives form a three dimensional network. An optimum wet end means the right balance between formation, retention and drainage. This is not very easy to achieve as all the parameters that improve one of them can adversely affect the other. That is why studying and measuring the drainage performance in regular intervals is so important from papermaker's viewpoint.

3. PARAMETERS AFFECTING /INFLUENCING DRAINAGE

Various parameters that influence drainage process on a paper machine are generally classified as und

3.1Furnish properties

- Composition
    - Fiber properties (length, mass distribution, fibrillation, flexibility)
    - Fillers (ash content, particle shape, size and density)
    - Fines content
    - Wet- end additives (molecular weight, charge density and structure)
    - Air content

- Refining degree- freeness
    - Consistency
    - Temperature
    - Rheological properties
    - Density
    - Flocculation
- Product specification
    - Grammage
    - Physical properties
    - Chemical properties
- Paper machine parameters
    - Head box conditions (turbulence and shear forces)
        i. Slice configuration
        ii. Jet position
        iii. Pressure condition (flow)

    - Machine speed
        i.Wire speed
        ii.Jet/wire speed ratio

    - Table design and drainage element conditions
        i.Wire (fabric mass, width, fabric ability to drain, air permeability)
        ii.Forming board position
        iii.Blade angle along the table
        iv.Gravity element (type, number, placement)

v.Vacuum devices (type, number, pressure gradient)
vi.Formation devices i.e. top formers

4. DRAINAGE MEASURING TECHNIQUES

Many types of equipments/ instruments are used to study drainage, some of them are similar and others are complimentary. Drainage testers serve several purposes as under.

- Pulp quality evaluation
- Prediction of drainage behaviour of a given furnish
- Prediction of retention of different wet-end additives
- Prediction of the relationship between pulp and paper properties

Drainage performance can be studied both in laboratory and also online i.e. on paper machine itself. Below are some techniques or methods to measure drainage in laboratory and online.

5. LABORATORY DRAINAGE TESTING

In laboratory, drainage for certain furnish can be studied using several instruments or equipments. Based on operating conditions, there are mainly two types of devices commonly used in laboratory for monitoring/ measuring drainage performance. These are as under.

- Static drainage tester
    - Freeness tester
    - Constant flow and constant pressure tester
- Dynamic drainage tester
    - Britt jar
    - Hercules dynamic drainage jar

5.1Static drainage tester

(i)Freeness and wetness tester

The term 'freeness' is most often used to describe the relative ability of water to flow from a pulp suspension. Freeness tests are primarily based on the technique of measuring volume of water drained as a pad of pulp is formed on a screen or porous plate (**Fig. 2**).

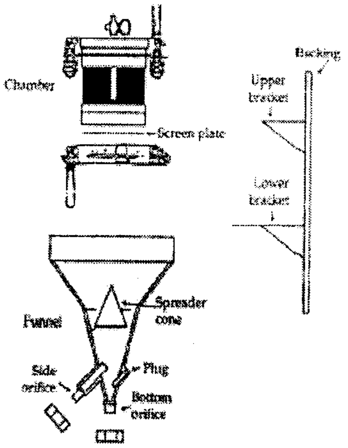


FIG. 2:CANADIAN STANDARD FREENESS

Freeness testers are by far the most common form of drainage testers in use today owing to its simplicity. Freeness tester works on the principle of drainage through a fiber mat formed during the test on a perforated screen plate of a given volume of an aqueous pulp suspension into a funnel provided with a bottom and a side orifice. Determination is made of the volume of filtrate discharged from the side orifice. The volume of the discharged filtrate, in millilitres, is the 'Canadian Standard' freeness of the pulp. The standard temperature and consistency for the test are 20^{0}C and 0.30% respectively. However corrections are made according to the ISO 5267-2:2001(E) method, when test conditions deviate from the standard ones.

Wetness tester (**Fig. 2**) works also on the same principle as freeness tester except that the determination is made in degree Schopper-Riegler numbers and not in millilitres. The filtrate from side orifice is hence collected in a measuring cylinder graduated in Schopper-Riegler numbers. However standard conditions for testing are 20^{0}C and 0.20% consistency.

Freeness testers are probably most widely used by papermakers for predicting drainage of pulp furnish as it is simplest to use and handle. However, there are certain limitations of freeness testers as mentioned below.

- The mechanisms which control drainage on a paper machine are far more complex in nature than those controlling drainage in freeness tester. Unlike in paper machine, only gravity drainage without shear takes place in freeness tester ignoring the compressible behaviour of pulp pad.
- Freeness is not a proper indication of changes to be expected in vacuum dewatering. When retention aids are added to furnish, freeness increases due to flocculation while on paper machine, wet line moves toward the head box. However solid content after couch may be lower than that without additives.

- Freeness tester basically gives a qualitative indication of how a stock will drain by gravity, and not necessarily predicts quantitative machine performance.
- Overdependence of the test result on fines content reduces its validity as a means of predicting the drainage on paper machine.

Thus it is possible that pulps of same freeness or wetness can exhibit quite different drainage behaviour on paper machine. Also it is generally believed that freeness test is more valid for drainage occurring at low pressure drop areas, rather than dewatering under greater heads. This eventually led to development of drainage testers incorporating higher pressure drops.

6. CALIBRATION OF FREENESS TESTERS

6.1CSF Tester

The calibration of the rate measuring funnel requires that two critical dimensions be adjusted within specified limits.

- The head of water in the funnel which influences the flow through the bottom orifice.

- The volume of water in the bottom section from the bottom of the core to the side orifice overflow level, given as 23.5 ml in the standard procedure. In this procedure the side orifice portion is adjusted to provide the required volume. Before every use or at least once a day (preferably the beginning of experiments in morning) a field check of CSF tester should be carried out daily. A 1000ml of distilled water at 20^0 C instead of pulp for a side orifice discharge, which may be used as a field, check on the bottom orifice. The side orifice discharge should not differ from the value by more than 5 ml. If the difference exceeds this bottom orifice shall be replaced.

6.2 Schopper-Riegler Tester

Check the position of the side orifice as fellow. Close the bottom orifice with a finger. Pour 100 ml of water at 20.0^0 C+0.5^0 C into the funnel. After the excess has escaped through the side orifice open the bottom orifice and collect the water leaving the funnel. The volume of the water shall be 7.5 ml to 8.0 ml. If this is not the case adjust the side orifice. Check that the side orifice in the right position so that the pressure head is correct.

Check the diameter of the bottom orifice as follow. Remove the spreader cone close the side orifice with a stopper and fill it by pouring about 500 ml of the water at 20^0 C into the funnel while the bottom orifice is closed with a finger. After a moment allow the excess water to escape through the bottom orifice.

6.3 Constant flow and constant pressure drainage tester

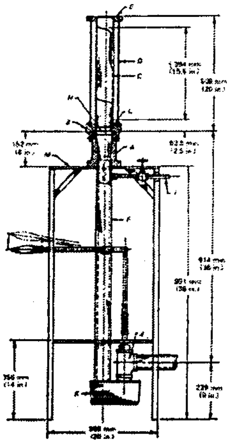


FIG. 3:CONSTANT FLOW AND CONSTANT PRESSURE DRAINAGE TESTER

It actually measures drainage time. Drainage time is the time required to form the sheet on wire– the interval between the onset of drainage of water to the point of air intrusion into the sheet.

The development of fundamental dewatering principles was the primary goal of both the constant flow and constant pressure drop approaches to the drainage of water through a forming fibrous mat.

Several Scientists worked on this kind of drainage testers to obtain specific filtration resistance and fundamental fibre properties viz. specific surface area, mat compressibility etc. However, constant flow and constant pressure drainage testers (**Fig. 3**) are not fully able to predict flow and drainage on paper machine. The turbulence and heterogeneous flows encountered in paper machine allows its use only to non-disruptive drainage part of the wet end.

6.4 Dynamic drainage tester

i. Britt dynamic drainage jar

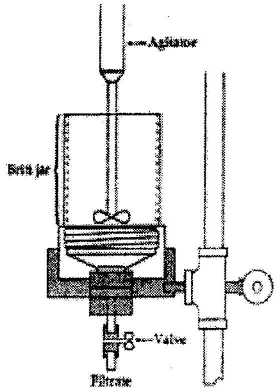


FIG. 4:BRITT DYNAMIC DRAINAGE JAR

The static drainage testers referred previously failed to account for one critical characteristic that sets from the dewatering which takes place on the paper machine– drainage is a dynamic process. The need to have the stock sample in a dynamic, turbulent condition during drainage test led Britt and Unbehend to develop the dynamic drainage jar (**Fig. 4**) in 1976. The Britt jar uses an impeller rotating at a high speed (500-1200 rpm) to impart turbulence in the stock as it drains through a fine mesh screen of precise hole size and most popular device to measure dynamic drainage.

Because of the ease and simplicity of its operation, many variations on dynamic drainage jar had been employed in attempts to quantify the drainage of stock under dynamic conditions.

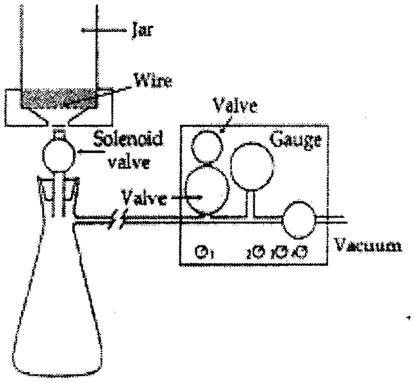


FIG. 5:MODIFIED BRITT DYNAMIC DRAINAGE JAR

In order to develop a test device in which the consistency, volume, basis weight, drain time, vacuum and level of turbulence are similar to actual papermaking conditions in mills, Britt et. al. modified the original dynamic drainage jar, by connecting it to a vacuum (**Fig. 5**)

The instrument was developed aiming to work over a considerable range of the parameters (simulating paper machine conditions) as under.

- Consistency– 0.3% to 1.0%
- Volume–50 ml (<1 cm depth) to 200 ml (~4 cm depth)
- Basis weight–20 g/m^2 (tissue) to 300g/m^2 (light board)
- Vacuum–5 to 30 cm of water
- Turbulence–0 to 500 rpm (0-100 m/min avg. rate of travel rod)
- Test time–3 to 12 s (relating the time of stock in forming zone)

The centre of reservoir cylinder is blocked out with a plastic cylinder 3.2 cm in diameter leaving an annulus of 3.5 cm in width.

ii.Hercules dynamic drainage jar

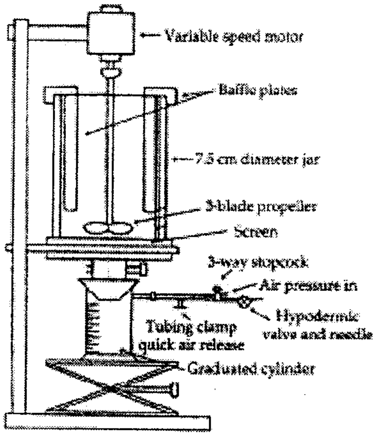


FIG. 6:HERCULES DYNAMIC DRAINAGE JAR

It was first used by Davidson et. al. in an attempt to examine the mechanism of fine particle retention in condition simulating actual fourdrinier paper machine. Compared to Britt jar, the Hercules dynamic drainage jar is considered to give more reliable results as unlike the former, latter one is able to utilize fibre mat formation during rapid drainage approach, a mechanism generally believed to take place in paper machine.

The schematic of this tester is shown in **Fig. 6**. A variable-speed stirrer with a 3-blade propeller located 6 mm above a screen in the bottom of the jar provides high shear turbulence in the stock sample. Two baffle plates break up the stirring vortex. A small positive air flow is applied underneath the screen to give an upward percolating effect. Rapid drainage and subsequent mat formation are produced by opening air release valve. The instrument also provides for slow drainage with continuous stirring but no mat formation. However, this was too modified as shown below.

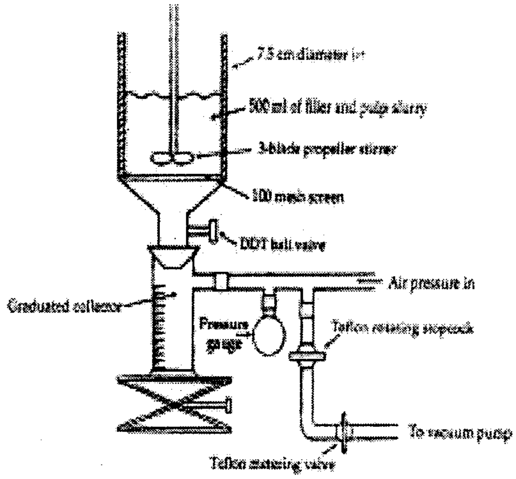


FIG. 7:MODIFIED HERCULES DYNAMIC DRAINAGE JAR

iii.Others

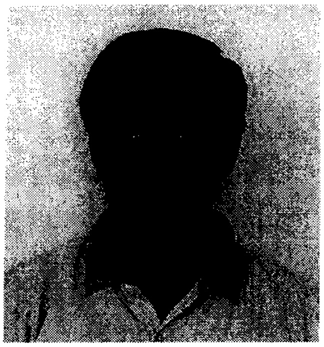
Other dynamic drainage testers include vacuum dewatering apparatus developed by Litchfield, Springer-Wegner vacuum drainage tester developed by Springer *et al.*, turbulent pulse sheet former, G/W drainage system (by Gess), Dynamic drainage Analyser, FRET retention tester, BPDD drainage tester etc. But those are not in much practice and hence not discussed here.

7. CONCLUSION

Drainage of water during papermaking is a complex phenomenon where the flow is subjected to pressure pulses, vibrations, induced turbulence etc., which affect the rheology of fibre suspension and influence the effects of different chemicals incorporated in the wet end. Therefore it is of utmost importance for the papermaker to know in advance how the pulp behaves during drainage i.e. how drainage influences the addition of wet end chemicals and also to observe the effect of drainage aids etc. incorporated for controlling drainage process.

Drainage can take place by different mechanisms viz. filtration, thickening, compaction etc. However in laboratory, drainage of a particular furnish can be monitored and evaluated using static devices e.g. CSF tester and SR tester, which are tremendously popular and also using dynamic instruments like Britt dynamic drainage jar, Hercules dynamic drainage jar etc.

LABORATORY BEATERS OF PULPS FOR EVALUATION



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LABORATORY BEATERS OF PULPS FOR EVALUATION

S. TYAGI

1 INTRODUCTION

The general objective of a quality control of any industrial process is to keep the quality of product as constant as possible, despite of varying process conditions. For any grade of paper, pulp quality is the major contributing factor. The idea of traditional laboratory testing of pulp is to stimulate the papermaking in the laboratory in standardized fashion. In practice, this means testing the paper properties from laboratory sheets instead of measuring single fiber properties.

The different phases of laboratory testing try to stimulate sub processes of the typical papermaking process. Since the papermaking process consists of numerous sub processes and many of these have substantial influence on paper properties, the test results should provide a sufficient basis to estimate the quality and usability of pulp. Pulp quality has no general definition. It always depends on the end purpose, e.g., paper grade.

The main parts of traditional chemical pulp testing are the following:

- Pulp disintegrated in water
- Pulp beaten in laboratory beater
- Pulp properties tested, e.g., drainage properties & fiber length
- Laboratory sheets prepared
- Sheets conditioned at standardized temperature & humidity
- Physical properties tested from the sheets, e.g., strength properties, structural properties & optical properties.

2. PULP EVALUATION METHODS

The importance of pulp evaluation become more important when a paper mill is using purchased pulp and paying substantial amount for it. Basically, there are two broad objectives for pulp evaluation:

- Testing the quality parameters of specific pulp
- Comparing the papermaking potential of different laboratory beating

In the present topic, we mainly discuss on the laboratory beating of chemical pulps in detail.

These methods have been formulated by different organizations like ISO, SCAN, TAPPI and APITA. All these standards are similar in their main principles, but differ in some details.

3. LABORATORY BEATING OF CHEMICAL PULP

Laboratory beating simulates the industrial refining process to predict the usability of a pulp. Since beating often has a large influence on the pulp properties, beating is the essential part of chemical pulp testing. Selection of the beating procedure has a significant effect on the measured properties.

Beating or refining changes several single fiber properties. The classification of primary structural effects on fibers can be internal fibrillation, external fibrillation, fines formation, fiber cutting, and strengthening of fibers. The primary effect occurs in all cases of pulp refining, but the relative importance varies depending upon the pulp type and the method of refining used. Any change in single fiber properties also changes the corresponding pulp and paper quality, e.g., pulp drainage resistance increases due to an increased surface area (mainly fines formation) during refining. Internal fibrillation increases fiber swelling and flexibility by "loosening" the cell wall structure, and external fibrillation increases the outer surface of the fibers. The structure and bonding of paper depends on fiber properties such as fibrillation. Much is available on primary effect of refining in work by Page (1) and Hietanen and Ebeling (2).

Traditionally, the aim of chemical pulp beating is to obtain a beating curve describing the development of pulp properties as a function of the amount of beating applied as shown in **Fig. 1**. By taking samples after different beating time and assessing the pulp quality by various tests, development of properties as a function of beating time may be measured. In classical laboratory beating procedures, the only command variable used is the beating energy.

4. CLASSICAL LABORATORY BEATERS

There are many different types of laboratory beaters used in pulp testing. The laboratory beaters differ in their construction, shape of the beater body, design of beater roll or rotor which carries out the beating, the means by which pressure applied between roll and bedplate, quantity of pulp required and the consistency at which beater is operated. The most important laboratory beaters and refiners and difference between them are given in **Table -1**

The most common laboratory beater is the PFI Mill. Other common types are the Valley Beater and Jokro Mill.

4.1 PFI mill

The beating elements of the PFI Mill consist of a roll with chiseled bars and a circular, smooth beater house (**Fig. 2**). The roll and the house are independently driven and move in the same direction of rotation, but at different peripheral speeds. The beater house is charged with the pulp, and by rotation of the house the pulp is pressed towards the wall of the beater house thereby forming an endless pulp band. As the roll always runs with a higher peripheral speed than the beater house, the roll bars will cut out sections of the pulp band and transport the pulp into the beating zone. The beating pressure is obtained by the roll being pressed against the beater house. Beating conditions, as pulp concentration, beating pressure, relative speed between the beating elements as well as the distance between them, may be varied within wide limits (3).

The PFI Mill consists of three main parts:

- Beater house mounted in the frame
- Roll mounted on a lever arm which can be pivoted and moved vertically
- Weight lever arrangement for obtaining beating pressure.

The beater house is made in one piece with the shaft suspended in ball bearings and mounted in the solid frame. Through the shaft a channel is drilled which is closed by means of a drain plug secured by a screw nut at the lower end of plug. The beater house is equipped on the outside with a rubber ring in order to keep the cover in a stable position during beating. The beater house is driven by a motor, and the speed of revolution can be varied in three steps by means of a step pulley.

The roll is made in one piece with the shaft is provided with 33-chiselled bars. It is suspended in ball bearing & mounted on a lever arm, which is pivoted 90° clockwise when being in its upper position. The roll lever arm is provided with a steering sleeve or pneumatic arrangement for lowering/rising the roll into the beater house. The cantilever which is mounted on the pillar and rigidly attached to the frame when pillar stands in its lower position, serves as the base by transmission of the load weight to beating pressure between roll and house. The beating pressure may be increased by mounting an additional weight on the weight lever arm. The roll, which is driven by its own motor, has only one speed level and the number of revolutions is recorded by means of a revolution counter **(Figs. 3 & 4)**

If a fixed distance beating clearance, greater than what is caused by the pulp layer, is required between the roll and the house, this may be regulated by an adjusting screen, which is screwed against the stop peg. The scale of the adjusting screw is revolving so that the zero point of the scale can be shifted to always indicate the point where it can be heard that the beating elements are just in contact with each other, when the roll and the beater house are turned slowly around by hand.

I. Technical data

Roll dimensions

Diameter	: 200 mm
Bar height	: 50 mm
Bar thickness	: 5 mm
Number of bars	: 33

Beater house dimensions

Inner diameter : 250 mm

Inner height : 52 mm

The speed of rotation for the roll is around 1460 rev/min. The speed of rotation for the beater house can be regulated in three steps. Corresponding relative velocities between roll and beater house will be around 2.5, 6.0, 8.5 m/sec. These data are valid when the beating elements run unloaded. During beating, the roll speed will be reduced and the beater house velocity increased, so that the mentioned relative velocities will be somewhat lower, depending upon the loading conditions. Normally the speed of rotation for the roll will be reduced with 20-40 rev/min (3).

A revolution counter registers the number of roll revolutions, and the ratio between roll and counter is 10:1.

The beating pressure can be varied by means of weights. The weight lever arm and fixed weight give together a linear beating pressure of 1.8 kgf/cm bar weight. With additional weight a beating pressure of 3.4 kgf/cm bar height is obtained. Beating pressure lower than 1.8 kgf/cm bar and higher than 6 kgf.cm are not recommended. The ratio between the load and the total pressure on the roll is 2.47. The total charge in the beater house, pulp and water, should not exceed 450 ml.

The pulp is then charged into the beater house. If the pulp concentration is 10% or more, the pulp is distributed by hand as evenly as possible against the wall of the house. If the pulp concentration is much lower than 10%, the house should be started after the pulp charging. The suspension will then be slung against the house wall, and the roll is lowered into the beater house while this is in operation.

II. Control and maintenance of the mill

Check the following:

The revolution per minute of the roll and the house

The belt does not slip

The roll bracket moves freely so that the entire applied load is transferred as beating pressure

The distance screw is out of action during beating

III. Calibration of PFI Mill

The calibration of PFI mill is carried out according to International standard ISO 5264/2- 1979 (E). The following steps are followed.

- Reverse the direction of rotation of the motor driving the beating housing.

- Set the distance screw to bring about a gap of about 0.5mm between the beating elements.
- Charge the beater housing with 15 gm of silicon carbide powder to pass 90 µm apertures suspended in 50 ml of soluble cutting oil diluted with 50 ml of water.
- Set the beater housing in rotation, so that the powder suspension is flung against the housing. Ensure that the cover is in position in the bracket and the roll in the beater housing.
- Stop the housing, and press the cover into position. Start both beating elements, apply the load, and carefully reduce the gap between the elements by means of the distance screw until the sound of grinding can be heard. Run the mill until this sound has diminished appreciably, and then further reduce the gap, but by an amount not exceeding the equivalent of half a scale division (approximately 0.03). Continue the grinding step by step in this way until the damage has been rectified.
- Clean the beating elements and the cover with soap and water. Ensure that no silicon carbide powder is left.
- After this rough grinding, carry out fine grinding with silicon carbide powder to pass 45 um aperture, as described in (3,4).
- Clean as described in (5).
- Apply a file to remove any rough edges that have appeared on the trailing edges of the bras.
- Thoroughly clean the roll to remove filing.
- Reverse the direction of rotation of the motor driving the beater housing , if ,after grinding with silicon carbide powder to pass 45 um aperture the beating surfaces are too coarse, smooth them to the required level by beating pulp in which approximately 15 gm of silicon carbide powder to pass 45um aperture has been mixed. Set the distance between roll and housing. So that it slightly exceeds that required by the layer of fibres. (When for example bleached coniferous sulphite pulp is used for this purpose a suitable gap could be 2 mm.)
- Thoroughly clean the housing and the roll.
- Disengage the distance screw.
- To stabilize the beating surface a series of beating for periods corresponding to between 50,000 and 100,000 revolutions of the roll is recommended.

4.2Valley Beater

The valley beater consists of a Hollander type machine. Beating occurs between the beating roll and a bedplate with loaded lever arm. (Fig. 5) The bars of the roll and a bedplate are made of stainless steel. The diameter of roll is 168 mm with the 32 fly bars inserted. It is 190–194 mm. The thickness of each fly bar is 4.7 mm and the width of the roll is 152 mm (4).

The bedplate has 7 bars set in load, each 3.2 mm thick and 2.4 mm apart. The grooves between the bars are fitted with stripes of kiln-driven white oak. The bars are rectangular in cross section and bent in the shape of V with the apex of the V pointing in the flow direction of the stock, and the arms of the V forming an angle of 5° with the direction of the roll axis. The width of the bedplate is 159 mm; it is ground to the same curvature as the wall.

The bedplate is pressed against the roll by means of a lever. The ratio between the lever arms is 1.94 to 1, a load of 5500 gs obtained by using suitable weights applied to the longer arm then produces a force of 105 N on the bedplate. The roll is run at a rotational frequency of 8.3±0.2 /s and may conveniently be driven by a motor of approximately 400 W by a belt drive.

The bedplate is balanced so that it is in equilibrium with 23.0 L of water in the beater when the beater is running. An extremely small force will then bring the roll and bedplate into contact.

I. Control and maintenance of Valley Beater

To ensure reproducible beating, the following conditions should be fulfilled:

- The beater roll should run smoothly, without irregular vibrations
- All the roll and bedplate bars should make contact throughout their width
- The threshold formed by the first bar of the bedplate and the depth of grooves between the bedplate bars should be 1.0±0.2 mm
- The loading edge of the fly bars should be rounded to a radius of approximately 0.2 to 0.3 mm.
- All the bars should be clean, undamaged and free from deposits and corrosion.

ii. Calibration of Valley Beater

The calibration of Valley Beater is carried out according to International standard ISO 5264/1- 1979 . The following steps were followed.

- Prepare a standard 360 gm (O.D. basis) beater charge of pulp in the normal way. Operate the beater without load for a few minutes and add 120 gm (appro.80 ml) of the 125µm silicone carbide powder to form a homogeneous mixture.

- With the beater running adjust the setscrews on the lever-arm clamp to bring the bedplate into contact with the beater roll. Continue grinding with occasional adjustment to maintain bedplate contact, until a smooth, vibration free operation is achieved and a shiny ground surface is obtained evenly across the fly bars on the beater roll. With proper alignment, this condition should be attained in approx. 30 min. if the bedplate bars and the beater roll are completely new. If they have already been in use a few minutes should be satisfactory.
- Empty the pulp and abrasive from the beater and retain this mixture for further grinding. Wash the beater thoroughly and dry the area around the bedplate with blotters.
- Take a carbon paper impression of the condition of the bedplate as follows.
- Place a sheet of the carbon paper between two paper sheets to give a total thickness of 0.15 mm. Cut this to 160mm X 250mm. Place the prepared carbon paper between the bedplate and the beater roll and apply pressure to the lever-arm. Hold the paper firmly to prevent slippage and rotate the roll by hand. Repeat the procedure in a few other position of the roll until a complete impression of the bedplate is obtained.
- Continue grinding with the pulp abrasive mixture until the carbon paper impression shows complete contact between the bedplate bars and the beater roll.
- Once complete contact is achieved, empty the beater and retain the pulp abrasive mixture. Remove the bedplate and diaphragm as a unit and repeat the removal of any excess mounting metal from the leading edge of the bedplate bars to a depth of 1.0 +0.2mm.
- Scrape or hone the burred edges of each bedplate bar and clean the area between each bar. Grind or file the raised shoulders at the end of the bedplate and replace the bedplate.
- Pour the pulp abrasive mixture into the beater and grind for appor. 5 min. with the standard 54 N load on the lever-arm to correct any misalignment resulting from the removal of the bedplate assembly.
- Empty the beater dry the bedplate area and check the bedplate contact with a carbon paper impression. If contact is complete remove the splash cover from the beater roll. Rotate the beater roll by hand and check that each fly bar is straight with a precision straight edge. Carefully hone the metal burr from each fly bar trailing edge using the hone and the light machine oil.
- Carefully hone the leading edge of each of each fly bar to a radius of approx. 0.2 to 0.3 mm. At this stage do not attempt to modify the grooved surfaces of the fly bar.

- Check the operation of the beater by beating a reference pulp for a hardwood reference pulp beating for the time normally required to reach about 50 Schopper- Riegler number or 200 "Canadian Standard" freeness number shall give a Schopper- Riegler number about 10% higher or a Canadian Standard Freeness number about 10-% lower than the normal value for that pulp. If checking is made with a softwood reference pulp the drainage values obtained shall be at the level normal for the pulp concerned.

4.3Jokro Mill

The Jokro mill is similar to PFI mill. It comprises a horizontal revolving table in which are mounted 6 cylindrical holders which make a planetary movement around the central shaft. Each chamber can take 16 gm OD pulp. It has no counter bars in the beating housing (5). Beating occurs between bar surfaces and smooth housing. Pulp consistency is 6%.

5. BEATING ACTION IN DIFFERENT LABORATORY BEATERS

The beating action is different in all these three beaters. The main differences are listed in **Table- 2.**

The Valley beater mainly removes the primary wall of the fibers and causes internal and external fibrillation (5, 6). A significant amount of fiber breakage also occurs. In the PFI and Jokro mills, the removal of the outer layer of fibers and fiber breakage is less intense, but the internal and external fibrillation are probably stronger than in the Valley beater. In industrial low consistency refining, the removal of the outer layer and breakage of fibers is probably more important than in any laboratory beating (5).

The advantage of PFI mill is that a single beating uses a small amount of pulp (30 gm OD pulp per beating). For each batch, the beating time can be chosen independently. The necessary amount of pulp can come from a laboratory scale pulp digester with several auto claves each providing sufficient pulp for one beating.

A benefit of the Valley beater is large amount of pulp for beating (360 gm OD) that is probably sufficient to be a representative sample for the pulp (5). This also allows sampling from the beater at selected time intervals to provide a complete curve using a single beating.

In many cases, the PFI mill and Valley beater classify pulps in the same order (7) but in some cases, the results differ (8). The valley beater seems to be a laboratory beater where refining action is closest to that of an industrial refiner, although the beating intensity is significantly lower than for typical production scale refining (especially for softwood pulps). Industrial refining also does not straighten the fibers in the same way as laboratory beating.

Many factors can cause variations in laboratory beating results. The mechanical condition of the beater and especially the bar edges requires careful control. The calibration and maintenance if a valley beater is more difficult than for a PFI mill (9). The quality of the water used has a significant effect on pulp behavior and therefore on the beating results. Important water parameters are pH (10), electrolyte content (10, 11) and temperature. All these influence the swelling of fibers. Lindstrom (12) has reviewed the chemical factors influencing the behavior of fibers.

TABLE - 1
LABORATORY BEATERS AND REFINERS

Proprietary Name	**Capacity of Fiber (grams)**	**Consistency (%)**	**Form of Body and Bedplate**	**Roll Design**	**Fixed Capacity**
Beaters					
Ball Mill	90	4.3	Smooth Cylinder. No Bedplate	Smooth Sphere	No
Valley Beater	360	1.57	Oval Trough. Barred Bedplate. Horizontal Circulation	Barred Cylinder	Yes
PFI Mill	30	10	Smooth Cylinder. No Bedplate	Barred Cylinder	Yes
Lampen Mill	24	3	Smooth Sphere. No Bedplate	Single Large Smooth Sphere	No
Jokro Mill	16	6	Internally Grooved Cylinder	Barred Cylinder	No
Noble and Wood Cycle Beater	900 to 4500	2.5 to 5.0	Oval Trough with Barred Bedplate. Circulation by Propeller	Barred Cylinder	Yes
Banning-Seybold Beater	550 to 900	5 to 6	Oval with two Barred Bedplates in Sides Vertical Circulation	Barred Cylinder	No
Reed Paper Group Medway Beater	125	3.5	Oval with Barred Bedplates in Sides. Vertical Circulation	Barred Cylinder	No
Refiners					
Aylesford Beater	1620	3.0	Internally Barred Conical Cell. Through Circulation	Externally Barred Conical plug Rotor	Yes
Escher-Wyss Refiner R1	500	4.0	Internally Barred Conical Cell. Through Circulation	Externally Barred Conical plug Rotor	Yes

TABLE-2

DIFFERENCE IN BEATING ACTION IN DIFFERENT LABORATORY BEATERS.

Action	**Valley Beater**	**PFI Mill**	**Jokro Mill**
Removal of Primary Walls/outer layer	Main	Less Intense	Less Intense
Internal & External Fibrillation	Little	More	More
Fiber Breakage	Significant	Less	Less

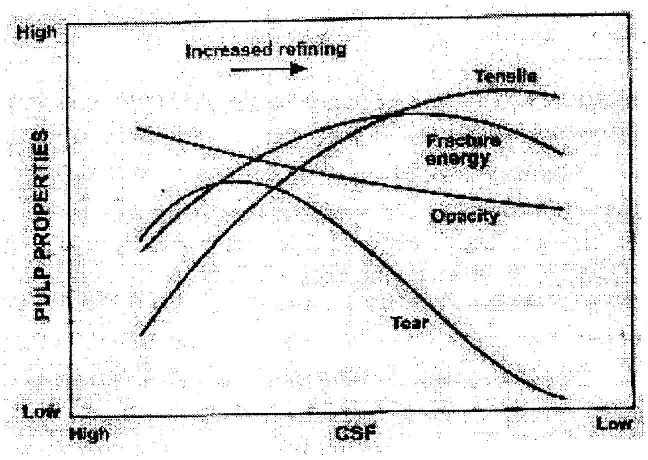


FIG.1: TYPICAL DEVELOPMENT OF CHEMICAL PULP PROPERTIES DURING BEATING

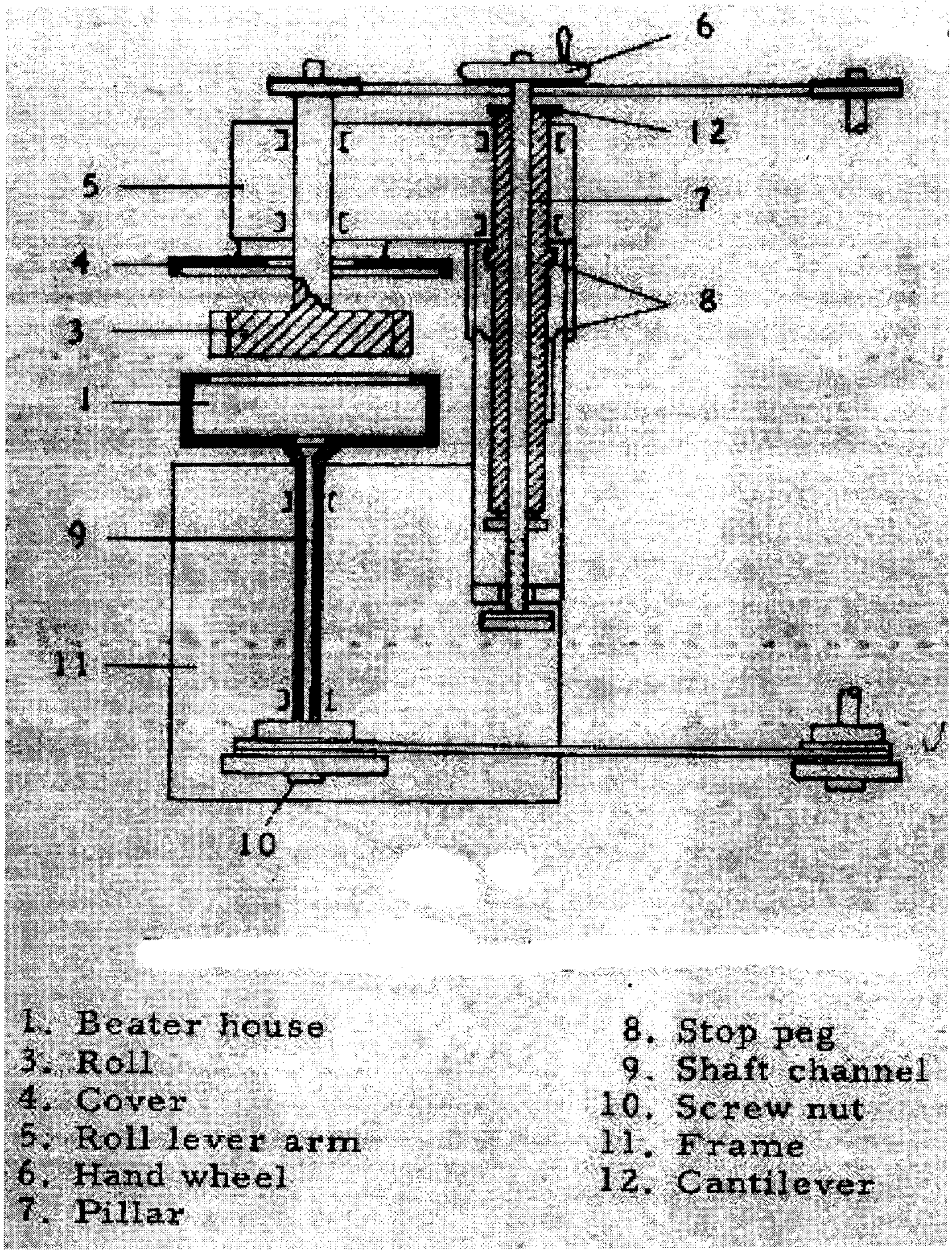
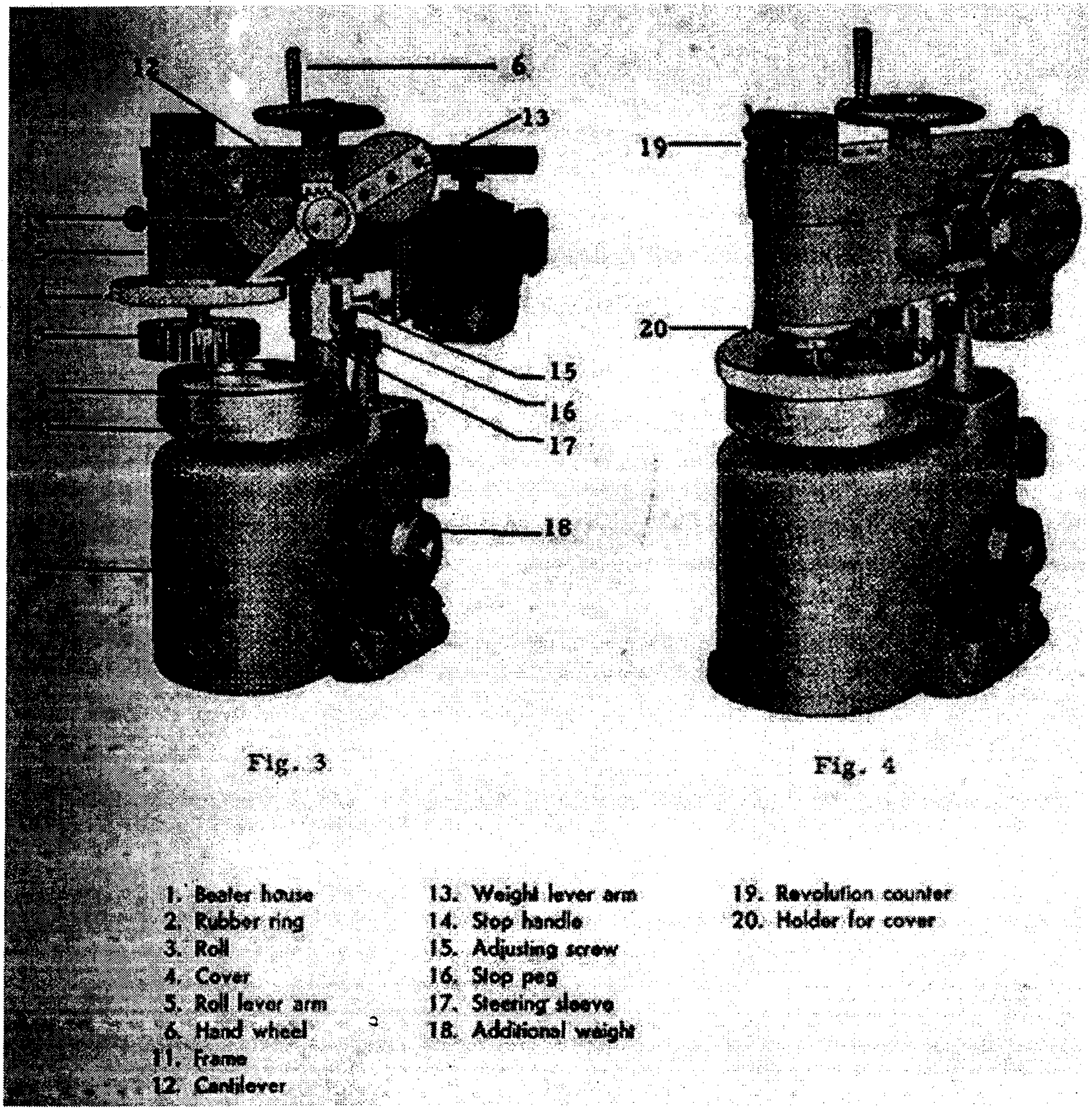


FIG. 2: DIAGRAMMATIC DRAWING OF PFI MILL



FIGS. 3 & 4: PFI MILL

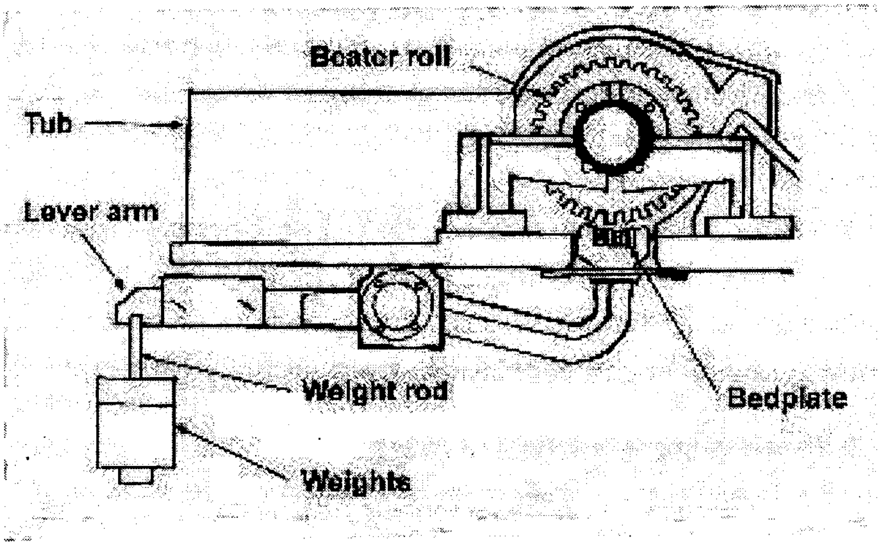


FIG. 5: DIAGRAMMATIC FIGURE OF VALLEY BEATER

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OPTICAL PROPERTIES OF PULP AND PAPER & THEIR MEASURMENT

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OPTICAL PROPERTIES OF PULP AND PAPER AND THEIR MEASURMENT

RENU TYAGI & TRIPTI JOHRI

1. INTRODUCTION

The optical properties of paper are most important for paper manufacturer. As these connote to the appearance of paper and mainly the paper is sold on the basis of its appearance.

The important optical properties of paper are:

- Brightness
- Opacity
- Whiteness
- Shade
- Gloss

The definition of these parameters are:

1.1Brightness

It is the intrinsic reflectance factor measured with a reflectometer equipped with a filter or corresponding function having an effective wavelength of 457 nm and a width at half height of 44 nm, and adjusted so that the UV content of illumination incident upon the test piece corresponds to that of the CIE illuminant C.

1.2Printing opacity

The ratio expressed as a percentage of the single sheet luminous reflectance factor to the intrinsic luminous reflectance factor of the same sample.

1.3Whiteness

It is a combination of the total reflectance of white light and uniformity of the reflectance at all wavelengths.

1.4Shade

Quantitative interpretation of color is done by following three parameters:

- *L**
- *a**
- *b**

+L describes lightness, –*L* describes darkness
+a describes redness, –*a* describes greenness
+b describes yellowness, –*b* describes blueness

1.5Gloss

It is defined as the specular reflectance of a paper sample measured at an angle of 75°.

2. DIFFERENT PULP & PAPER PROCESSES THAT INFLUENCE THE OPTICAL PROPERTIES OF PAPER

2.1 Bleaching

Bleaching of pulp can drastically change the appearance of the paper from the brown of unbleached Kraft pulp to the extremely light of highly bleached pulp.

Unbleached pulp has a high light absorption in the blue part of the spectrum. In bleaching, the light absorption decreases and, to reach the highest brightness values, the light absorption must be pressed down to very low values, **Fig. 1**

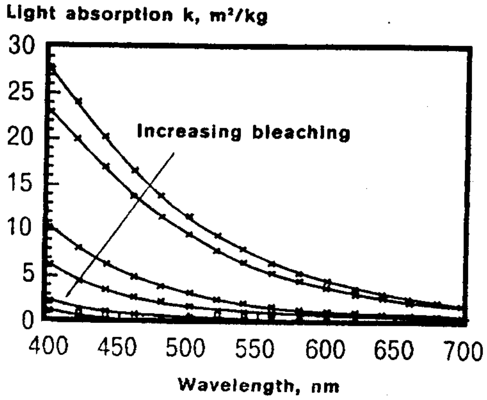


FIG. 1 :THE ABSORPTION FOR SULFATE PULP AT DIFFERENT STATES OF BLEACHING. THE PULP WITH THE LOWEST ABSORPTION HAD A BRIGHTNESS OF 85 %

Unbleached sulphite pulp and mechanical pulp are relatively light compared with the brown unbleached sulphate pulp. The difference in colour is clearly visible in the light absorption values measured with an R457-function **Table 1.**

The light absorption coefficient, which indicates that there are coloured substances in the pulp, is a good measure of the degree of bleaching

TABLE –1

LIGHT ABSORPTION AND BRIGHTNESS FOR DIFFERENT TYPES OF PULP

Pulp type	$K(R_{457})$ m^2/kg	Brightness R_{457} %
Unbleached sulphate	20-70	33-15
Unbleached sulphite	2-15	70-40
Ground wood pulp	5-12	67-55
Aspen CTMP	5-12	55-45
Bleached aspen CTMP	0.1-2	90-70
Bleached sulphate	0.1-2	90-70

3. BEATING OF CHEMICAL PULPS

In beating, the fibre bonds increase, and consequently the free fibre surface decreases. The result is that the tensile strength increases while the light scattering decreases. The light scattering ability of different pulps should therefore be compared at the same tensile index. **Fig. 2** shows that the long-fibre spruce sulphite and pine sulphite pulps have a lower light scattering at a given tensile index than the hardwood pulps. One of the reasons why eucalyptus pulp has had so great a success is that it gives the highest light scattering at a given tensile strength of the chemical pulps.

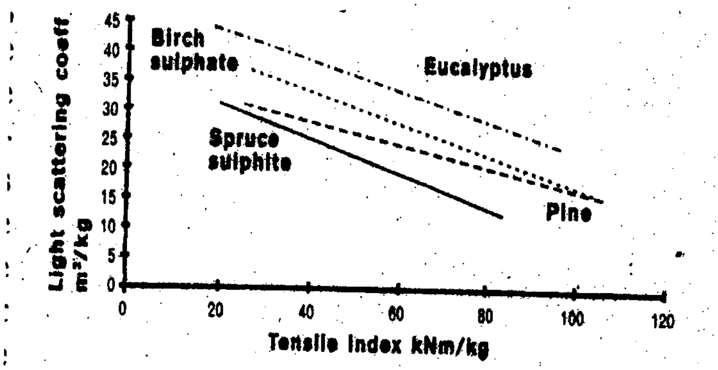


FIG. 2 :THE BEATING ALWAYS REDUCES THE LIGHT SCATTERING FOR CHEMICAL PULP, BUT DIFFERENTLY FOR DIFFERENT PULPS IF THE COMPARISON IS MADE AT THE SAME TENSILE STRENGTH

The explanation for the difference between different types of wood can be found in the structure of the fibre. Important properties are the number of fibres per gram and the surface area / weight ratio. For slender and thin-walled fibres, the surface area / weight ratio is greater than for broad and thick fibres. This gives more surface both for binding and for light scattering see **Table –2.**

TABLE –2

COMPARISON BETWEEN FIBRE DIMENSIONS AND LIGHT SCATTERING ABILITY FOR DIFFERENT TYPE OF WOOD

	Pine	**Birch**	**Eucalyptus**
Length, mm	3.0	1.1	0.9
Width, µm	38	18-22	12-16
Wall Thickness, µm	8	3	2-3
Number of fibres per µ g of pulp	2	8	16
s at a tensile index of 50 kNm/kg	29	34	41

For printing paper, this is important since an in decrease in light scattering gives higher opacity. Hardwood therefore gives more opaque paper than softwood.

4. THE DEPENDENCE OF LIGHT SCATTERING ON THE SPECIFIC SURFACE AREA

The specific surface area is the sum of all the small surfaces of fibres, fibrils, lumen etc. which are not bonded to each other. The specific surface can be determined by measuring how much nitrogen in a unimolceular layer can be adsorbed by the material. The value of the specific surface is expressed in m2/g.

As shown in **Fig. 3** there is often a linear relation- ship between the light scattering and the specific surface area of the sheet. The light scattering follows the changes in the specific surface of the sheet in beating and wet-pressing of the sheet, but the relationship is not generally valid since slightly different relationships are obtained for different types of fibres. One explanation is that the shape of the fibre can be important. This has been shown by studying model fibres which are triangular, circular, rectangular etc., **Fig. 4.**

The different fibre shapes thus give rise to changes in light scattering which do not correspond to the same change in the specific surface area of the material

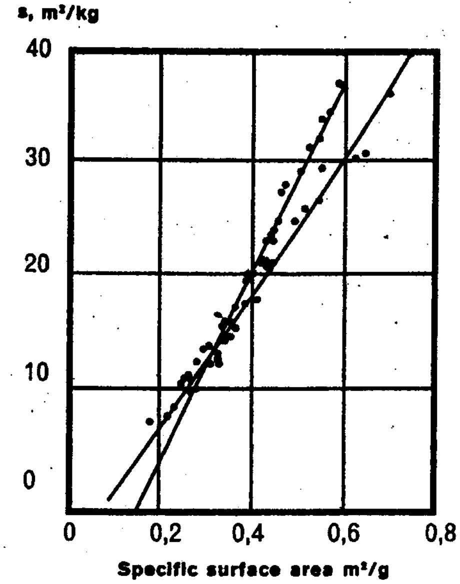


Fig. 3The relationship between light scattering and specific surface of laboratory sheets manufactured from different types of fibre. The point indicate different wet-pressing and beating conditions.

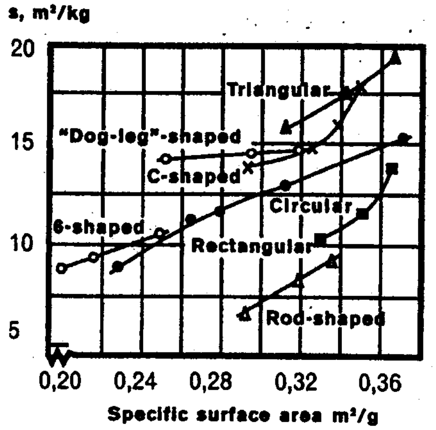


Fig.4The relationship between light scattering and specific surface is influenced by the shape of the fibres. The figure shows the relationship for Model fibres of rayon.

5. COMPARISON BETWEEN CHEMICAL AND MECHANICAL PULPS

The high light scattering associated with mechanical pulps is a consequence of a high content of fines/fine material. The fine material {crushed fibre fragments) also contributes to the bonding strength. This means that the light scattering can actually increase with increasing tensile strength in the case of mechanical pulps, as shown in **Fig. 5**

The difference between chemical and mechanical pulp can be explained by the fact that different types of fines are formed when chemical pulp is beaten and when mechanical pulp is ground and refined. The fines in chemical pulp consist of fibrils and delignified fibres and these are strongly swollen by water. In the drying, these fines are attracted towards the fibre surfaces and contribute to the strength of the sheet. The result is a smaller free surface area and thereby lower light scattering

The higher light scattering of mechanical pulp means that it is suitable for printing papers with low grarnmages, such as newsprint and LWC. Since the light absorption coefficient is also high, an opacity increase is obtained when mechanical pulps are mixed into a paper, see **Fig.6**

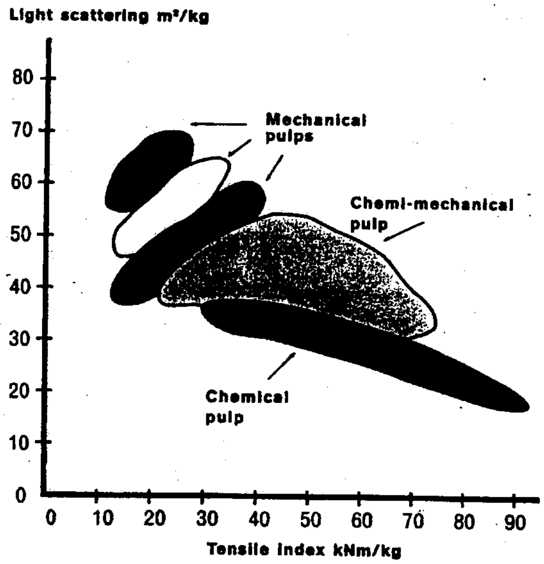


FIG.5: MECHANICAL PULPS GAVE HIGHER LIGHT SCATTERING THAN CHEMICAL PULP

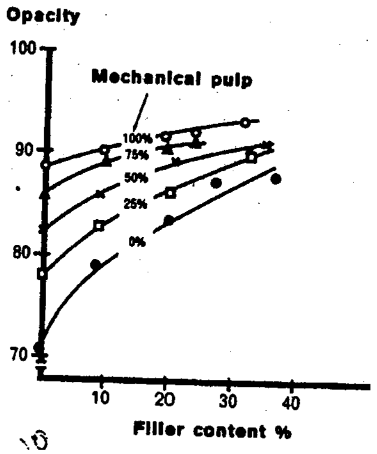


FIG.6: MECHANICAL PULP GIVES HIGHER OPACITY

6. FILLER

When paper contains filler, there are several different factors which influence the optical properties of the sheet. The most important are the refractive index of the pigment, the interaction of the pigment with the fibre, the specific surface of the sheet structure, the pore structure and the light absorption coefficient of the pigment.

7. THE INFLUENCE OF REFRACTIVE INDEX

The refractive index of the pigments should be high since the light scattering arises in the interface between materials with different refractive indices. As shown in **Table-3,** there are two pigments which have a special position, titanium dioxide, $TiO2$, and zinc oxide, ZnO. These also give high light scattering values and are used as opacity-increasing additives, particularly in USA.

$TiO2$ is a very effective pigment. The addition of 1% gives the same opacity-increasing effect as is obtained with the addition of 10-20% of conventional pigments such as clay and calcium carbonate $CaCO3$, see **Fig.7** & **Table-4.**

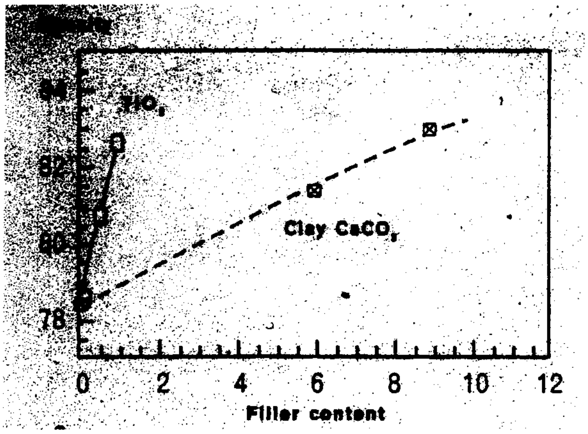


FIG.7: TIO_2 IS AN EFFECTIVE PIGMENT WHICH GIVES OPACITY EVEN WITH SMALL QUANTITIES.

TiO_2 is extremely effective filler due to its high refractive index which gives the pigment good light scattering properties. The pigment also has a low light absorption in the visible range. It thus gives high opacity and high brightness when it is used as filler and as coating pigment.

TABLE -3

REFRACTIVE INDICES OF DIFFERENT MATERIALS

Material	**Refractive index**
Air	1.0
Cellulose	1.53
Clay	1.57
$CaCO_3$	1.61
Calcined clay	1.6
TiO_2	2.6
ZnO	2.0

TABLE -4

APPARENT S VALUE

Filler	**s, m^2/kg**
TiO_2	Ca
Clay and natural Calcium carbonate pigment (CaCO3)	130-150

TiO_2 has a disadvantage, however. It absorbs the UV-radiation very efficiently. Since fluorescent whitening agents are activated by UV-radiation, they function very poorly together with TiO2. This is the explanation of why TiO2 is not used so much in Europe. The prices are of course also important in this context. TiO2 is relatively speaking cheaper in USA than in Europe.

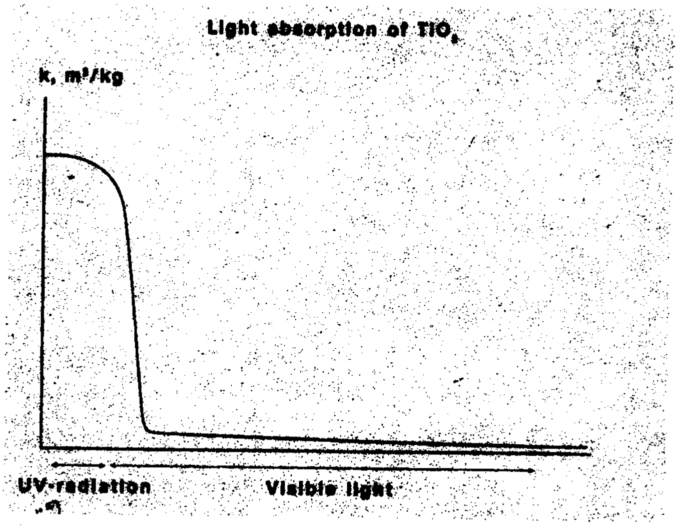


FIG.8: THE HIGH LIGHT ABSORPTION IN UV RANGE MEANS THAT FLUORESCENT WHITENING AGENTS (FWA) FUNCTIONS POORLY TOGETHER WITH TIO2.

8. THE INTERACTION BETWEEN THE FIBRE AND THE FILLER

The most common pigments, such as clay and CaCO3, have approximately the same refractive index as cellulose, and for these it is primarily the interfaces towards air which are important. Small pigment particles which are well scattered on the fibre surfaces give high light scattering.

The papermaker must therefore, find retention agents which give good filler retention without the fillers aggregating together too much, see **Fig.9** . It is not only the pigments which contribute to the light scattering of pigment filled papers, however. The fibre itself makes a contribution, which depends on how large a part of the fibre surface is free, i.e. does not participate in bonds

The addition of pigment means that fibre bonds are broken, so that the internal surface area associated with the fibres increases.

This explains part of the light scattering effect. This so-called debonding effect can in certain cases be the dominant contribution to the light scattering of pigment-filled sheets, but the light scattering gain takes place at the price of a reduction in the strength of the sheet.

The different contributions to the light scattering can be determined by an extensive analysis which includes measurements on sheets where the pulp has been beaten to different degrees, where the sheets have been pressed to different densities, and where the fillers have been dissolved out of the sheets (e.g. CaCO3 by acids)

The light scattering of pigment-filled sheets can be divided into three different components

- Light scattering from non bonded fibers
- Light scattering from pigment particles
- Light scattering from fines and fibrils which in the presence of pigments does not collapse on the surface

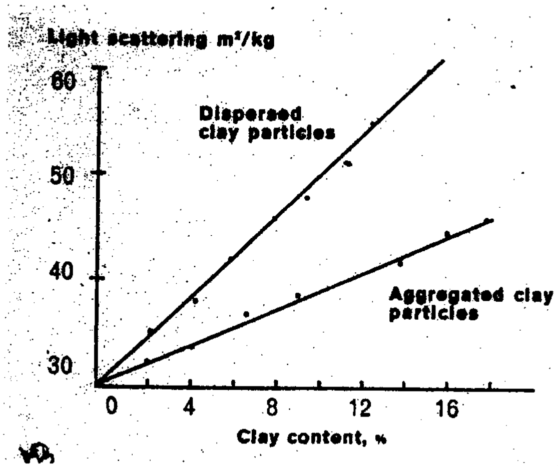


FIG.9: LIGHT SCATTERING FOR LABORATORY SHEETS (BLEACHED UNBEATEN KRAFT PULP) WITH DISPERSED (FINELY DISTRIBUTED) AND AGGREGATED CLAY PARTICLES.

9. CALIBRATION OF OPTICAL PAPER TESTING INSTRUMENTS

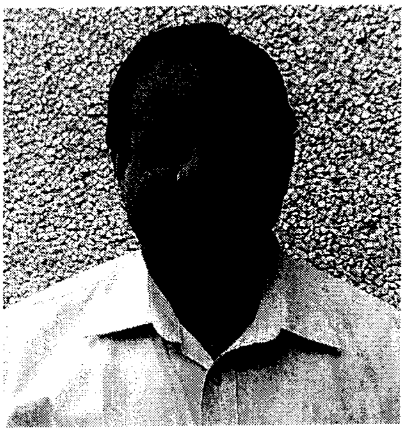
The purpose of calibration is to standardize the measurement procedures in order to make the measurement results comparable.

9.1 Calibration of brightness tester

Following accessories are required for the calibration of Brightness tester:

- Black cavity, having a reflectance factor which does not differ from its nominal value by more than 0.2%, at all wavelengths.
- Non-fluorescent reference standard for photometric calibration
- Fluorescent reference standard for adjusting the UV content of the radiation incident upon the sample, having an ISO brightness value 95±5 % under fluorescence component of ISO Brightness of 10±2 % units.

Methods of Evaluating The Dynamic Surface Characteristics of Paper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METHODS OF EVALUATING THE DYNAMIC SURFACE CHARACTERISTICS OF PAPER

Y.V. SOOD, R. SHARMA & TRIPTI JOHRI

1. INTRODUCTION

Surface Characteristics of paper play key role in assessing the quality of paper especially for printing, surface sizing and coating. Now a days the demand is for increasing web speed in surface sizing, coating and printing. All these require new perspectives on paper characteristics evaluation. Normally quality of the paper is judged by parameter such as grammage, thickness, brightness, opacity, tensile strength, Cobb and smoothness. Unfortunately all these are static parameters, which do not give reliable correlation with the problems experienced in end uses like printing and converting. For good correlation with the problems being experienced in process, it is important that realistic evaluation approach is adopted. At Central Pulp & Paper Research Institute, Saharanpur (India) a part from the latest modern facilities to evaluate various normal characteristics of paper, dynamic surface characteristic evaluation instruments are available which are quite useful in solving production problems in the following areas:

- Printing press
- Fibre rising causing linting or coating problems
- Dimensional stability causing creep, wrap or cockling

2. PRINT QUALITY EVALUATION

Judging the printing quality of paper by normal properties is insufficient & these can better be assessed after carrying out printing. For doing so CPPRI has laboratory **IGT printability tester (Model AIC 2-5)**. Using this instrument the printing tests can be carried out under printing condition similar to letter press, offset and gravure printing process. The following parameters can be evaluated.

- Ink Demand to get a particular print density
- Print through
- Print set off
- Wet picking or wet repellency
- Dry picking velocity
- Non-uniformity in print
- Wet and wet printing (two colours)
- Oil absorbency
- Gravure print missing dots

Such evaluation tests are useful in grading the papers from printing point of view & to carry out the process modification. It solves the problems of taking printing trials on a commercial scale printing press, which is very expensive and even not practicable.

2.1 Linting or fibre rising

This is an important surface parameter, which play an important role in paper quality to be assessed for coating and printing. During the coating, the fibres extending from the base paper are covered to get a smooth surface. With a variation in fibre length distribution, coating will not cover the fibre properly. Normally smoothness tests, like Bekk, Parker print surf, Bendtsen, which measure the air leakage of the clamped surface, are used to assess this property But it is not correct as in such cases it is important to characterize the distribution of fibres sticking out from the surface. Also during offset printing the situation become more complex as surface properties change when the paper is exposed to moisture and heat. The increased amount of long fibre bonded at one end only gets lifted off the paper surface cause linting problem. Also due to increase amount of short end, there may be gloss reduction. Evaluation of all these parameters is possible by using **Fibre Rising Tester (FRT)** available at CPPRI **(Fig.1)**. Using this instrument paper surface can be studied under conditions simulating to printing.

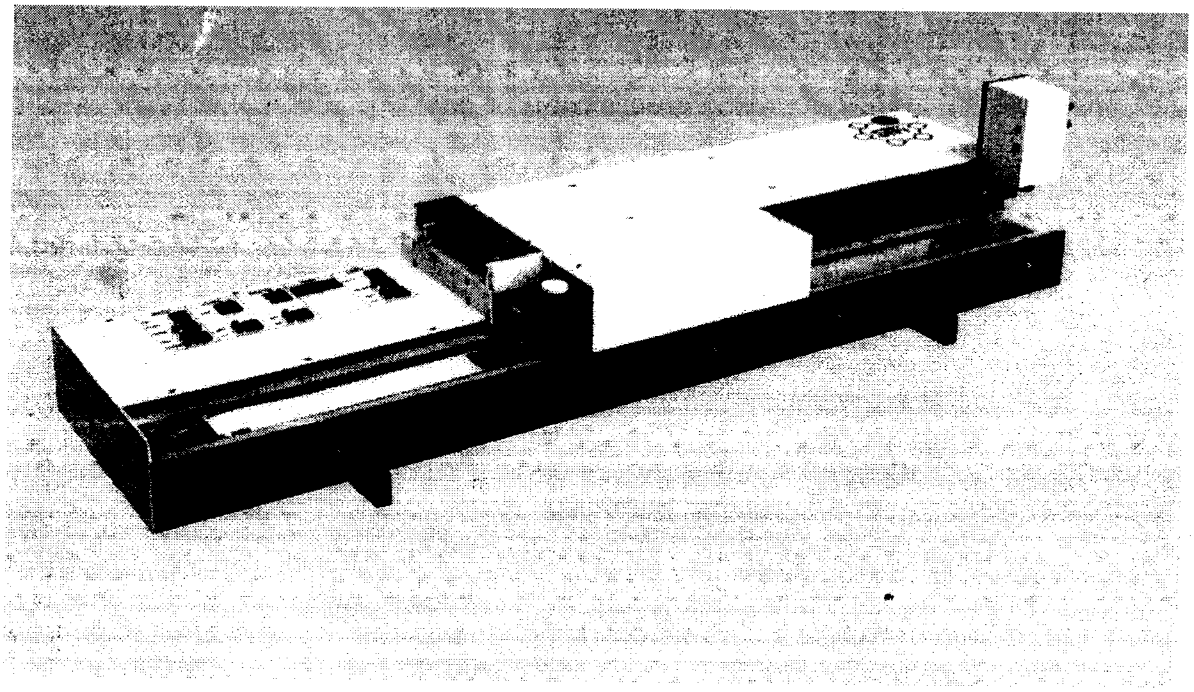


FIG. 1: FIBRE RISING TESTER.

A typical fibre rising observed for newsprint made from 100% virgin and 100% recycled fibres is shown in **Fig. 2**.

100% virgin Pulp

100% recycled fibre

Fig. 2: FIBRE RISING BEHAVIOUR OF NEWSPRINT MADE FROM 100 VIRGIN & 100 % RECYCLED FIBRES

2.2Dynamic dimensional stability

Dimensional changes will occur when the paper structure picks up moisture. This response is very dependent on the dynamic process inside the printing press where press design, web speed, tension and applied moisture and paper grade play significant role. To overcome runnability problems caused by dimensional changes the paper marker must achieve "Uniform Quality" enabling the printing press to be run without need for adjustment. Traditionally dimensional changes hygro expansion are measured using specimen strips cut in CD / MD direction. The specimen strips are exposed to different relative humidity and the expansion is measured.

This test does not really conform to actual behaviour of paper on printing press, the web while running inside the press is not likely to reach equilibrium; question has been raised regarding the relevance of such a test. This static value obtained at equilibrium condition may not correlate all with experienced runnability problem as these problems occur only for dimensional changes appearing while the runs inside the press between the printing nips. The more appropriate is "Dynamic hygro expansion test" the instrument to test this parameter is available at CPPRI. In this test the paper sample is sprinkled with copier toner ink and is kept inside a climate chamber. The climate inside the chamber is cycled between 10%-90% RH. After equilibrium the in place displacement are measured with a system based on "**Electronic speckle photography**". The dynamic hygro expansion caused by water vapour is measured from a sequence of image captured at different interval. The expansion with relative humidity is depicted in graphical manner. A typical dimensional stability curve for an offset paper giving misregister problem (B) and no problem (A) is shown in **Fig. 3**.

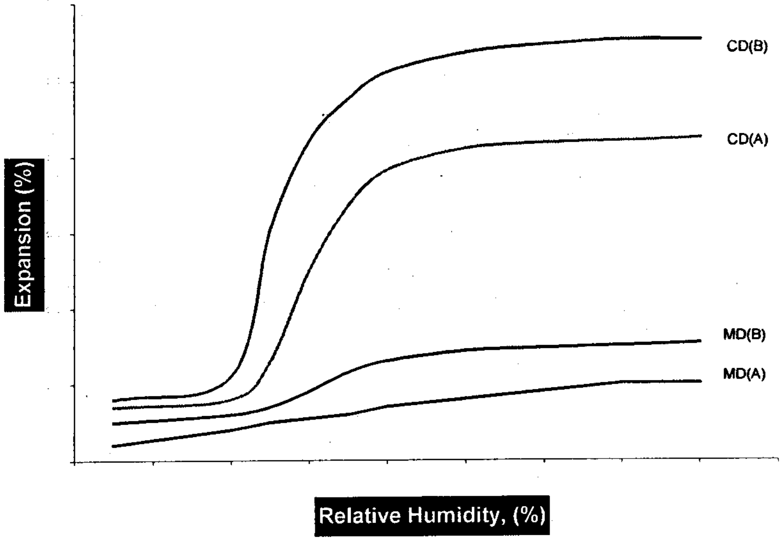


FIG. 3: A TYPICAL DIMENSIONAL STABILITY CURVE FOR AN OFFSET PAPER

2.3Dynamic surface wettability and absorbency of paper using an automated contact angle tester

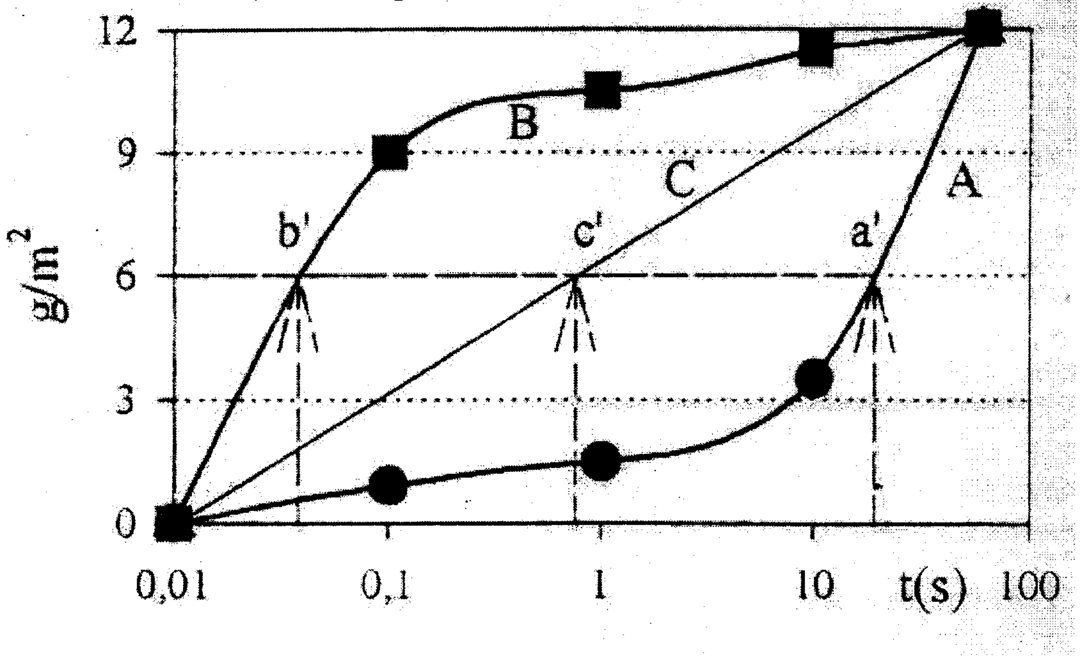


FIG. 4 : DYNAMIC TESTING OF PAPER

The meaning of dynamic testing is explained in **Fig 4.** Here the cobb 60 value of 12 shows that 12 gram of water was absorbed per square metre during a contact time of 60 sec. as the starting value was zero, a linear response of absorption should follow line (a) but this is only one of many possibilities. Curve (b) and (c) indicates two or more possibilities for the dynamic absorption of a surface with a cobb60 = 12. from the dynamic response we can see that 50% of the volume is absorbed at different times (a`-c`) for the curve (a-c). this is important as correlation with many experienced problems in surface sizing, coating as well as printing usually is found during the very first second of contact. this time window excludes all manual procedures because of the critical timing.

The cobb60 method, described above, is one example of a test where a single reading at one time is used for estimation of sorption. Furthermore, the cobb method represent a "macro test" giving an average value for a relatively large area, which limit its use for evaluation of the homogeneity of a surface. for many converting and printing applications this approach is not good enough for characterization of the surface interaction. another approach is the goniometer method (Tappi t458) where a droplet is applied on the specimen surface and the contact angle is manually estimated from a projection device. a correlation with experienced printing problems was, however, often difficult to find as the drop contour was changing too quickly to be tracked manually.

To cope with this and similar demand for testing of short time wetting and sorption, a new method t558pm-95 has been developed. here the droplet is applied on the specimen surface in synchronisation with the video camera used for capturing images of the changing drop. to cope with speed, 50 images of one millisecond exposure time are captured during the first second of contact. the contact angle (wetting), volume (sorption) and base diameter of the drop (spreading) is calculated from each image describing these dynamic properties as a function of time.

Wetting Retardation Time (WRT) can often be used for explanation of problems classified as mottling and adhesion. By definition WRT90 is the time at which contact angle curve falls below 90 degree as displayed in **Fig. 5**. the WRT concept explains how print quality and adhesion can be improved from studies of the interaction between a liquid (e.g. ink or glue) and the surface.

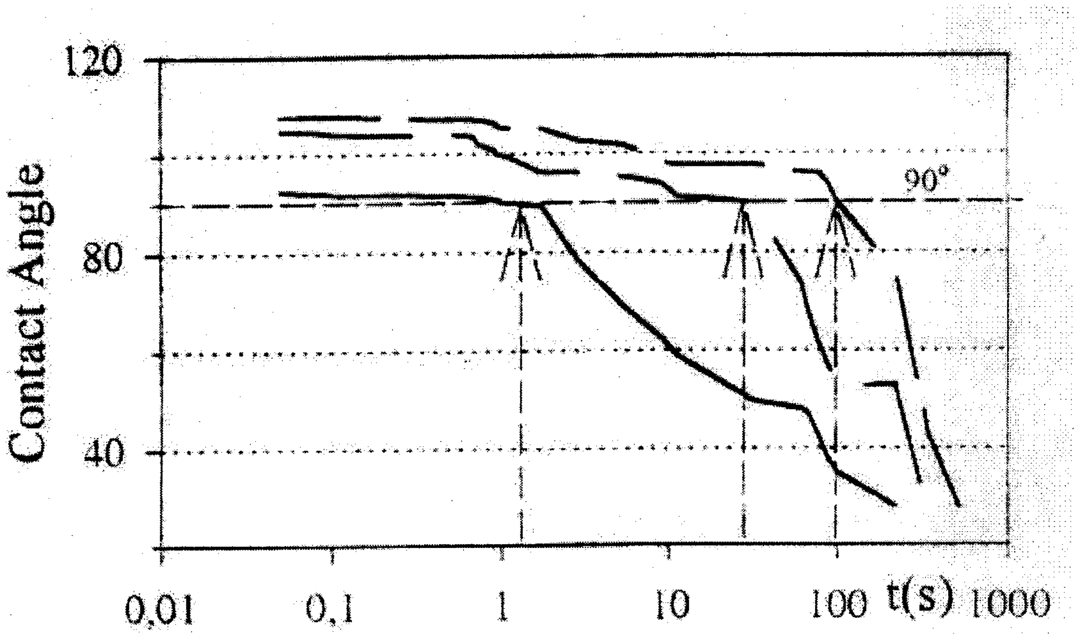
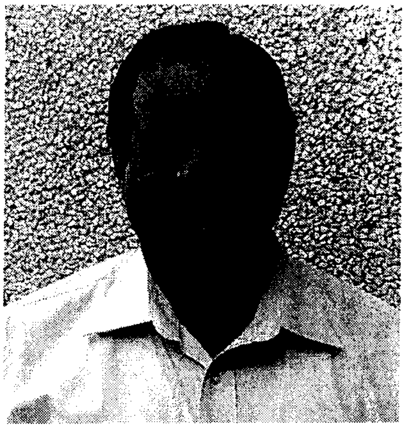


FIG. 5: CONTACT ANGLE CURVE

FRICTION MEASUREMENT OF PAPER



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FRICTION MEASUREMENT OF PAPER

Y.V. SOOD

1. INTRODUCTION

Apart from paper surface properties viz. surface roughness, gloss and surface strength, the friction characteristics also play an important role in various converting operations. As most of the Indian paper mills are switching over from rosin sizing to reactive sizing using AKD / ASA, the evaluation of this parameter becomes utmost important. Generally the paper sized with AKD, ASA and having talc as filler are lower in friction parameter and sometimes give problems in cutting operation and sheet fed printing machines. So the indigenous paper is required to be evaluated for friction characteristics.

Friction is the resisting force that occurs between paper or board surfaces in contact when the surfaces are brought to slide relative to each other. The friction properties of the surfaces are characterized by the coefficient of friction defined as the ratio of friction force to the force acting perpendicular to the two surfaces.

$\mu = F/F_n$

where μ is the coefficient of friction
F, friction force
F_n, normal force

Static friction and the coefficient of static friction describe the friction at the moment when the movement between the two surfaces commences but no actual sliding has yet occurred. Kinetic friction or the coefficient of kinetic friction applies to a situation where the two surfaces are sliding relative to each other at a constant speed.

2. METHODS USED FOR MEASUREMENT OF FRICTION

Several methods are available to measure friction. These fall into two main categories:

- Horizontal plane method
- Inclined plane method

The basic arrangement in both methods is that the test piece is fastened onto a horizontal table and a sled is moved on it. The weight of the sled exerts the normal force, F_n.

2.1 Horizontal plane method

I. Static friction

In this method, a gradually increasing pulling force is applied to the sled until it starts to slide. The friction force at the onset of sliding is measured, and the coefficient of static friction is calculated from the equation given above.

II. Kinetic friction

To measure kinetic friction, the sled is pulled at a constant speed. The average friction force is measure over a specified length of sliding. The coefficient of kinetic friction can be calculated from the average friction force.

2.2 Inclined plane method

I. Static friction

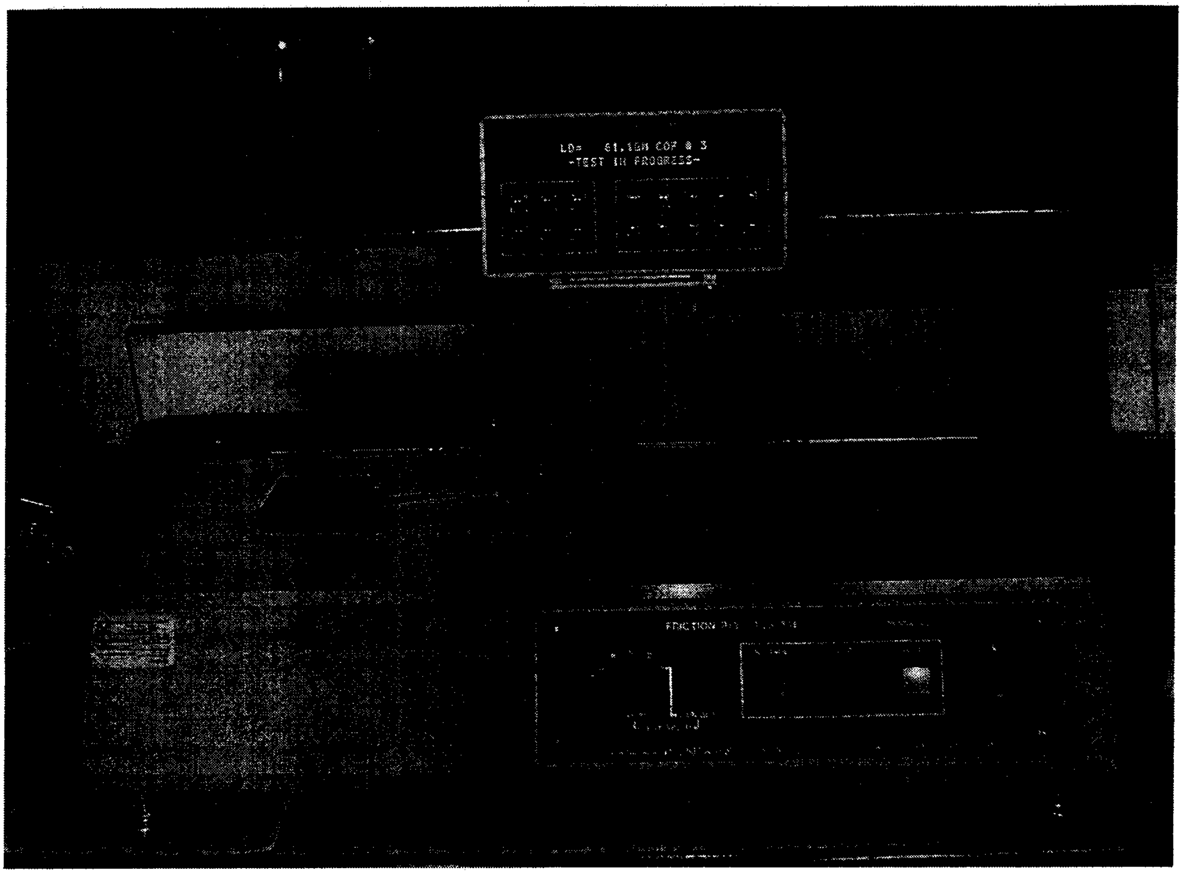
In this method, the sled is placed on the table, and the table is inclined gradually until the sled starts to slide. The coefficient of static friction can be calculated from the angle of inclination at the moment of sliding starts.

$$\mu = \tan(\alpha)$$

where μ is the coefficient of static friction
α is the angle of inclination at the moment of sliding

The inclined plane method has use only for measuring the coefficient of static friction.Many orientations are possible in which the two test pieces can be oriented with respect to each other on the sled and on the table: top side, wire side, cross direction, machine direction, manufacturing direction, and counter manufacturing direction. Since the friction is highly dependent on the surface properties and the topography of the paper, these combinations may provide different test results when testing friction between the surfaces.

CPPRI has recently procured and installed a Friction Tester (employing Horizontal Plane Method) for measuring the friction on paper / board surfaces.



FRICTION TESTER

TEST METHODS FOR PAPER AND PAPER BOARD

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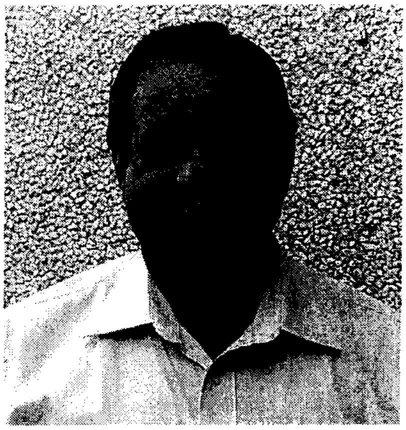

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TEST METHODS FOR PAPER AND PAPER BOARD

Y.V.SOOD, R.TANDON & RENU TYAGI

1. INTRODUCTION

It is relatively easier to measure distance, weight and time because the methods to measure them are quite common, well established & easily accessible to every one, every where. But measuring a specific property of any product may be pulp, paper, paper-board or any thing else is not that simple, because it matters where, how and by whom the measurement is being carried out. That is the reason standardization of testing methods has become a necessity for most of the commodities and paper and paperboard are no exception. Bureau of Indian Standards (BIS) formerly known as India Standard Institution (ISI) established in 1974 bears the responsibility of standards relating to commodities, materials and process and promotion of their general adoption at national and international level, certification of industrial products, assistance in the production of quality goods and circulation of information relating to standardization.

Indian Standards are formulated by large number of technical committees appointed by division council of BIS Standards relating to pulp, paper and paper boards are formulated by two committees, namely paper and its products (excluding packaging materials) sectional committed, CHD 015, and Paper and Pulp Based Packing Sectional Committee CHD 016. Both these committees, functioning under the Chemical Division Council comprise of representatives of the industry, research laboratories, important consumers and government departments. These committees appoint a number of subcommittees and panels comprising of experts in the subjects being dealt with by them.

2. INDIAN STANDARDS ON TEST METHODS

Standard methods for testing paper and paper board have been laid down in IS 1060 (in three parts) **Part-I** covers methods of sampling, preliminary examination of consignment, conditioning and methods of test for the determination of the following properties-

a. Substance or ream weight
b. Thickness
c. Bulk
d. Moisture content
e. PH value

f. Ash
g. Tensile strength and stretch
h. Breaking length
i. Bursting strength
j. Folding endurance
k. Tearing resistance
l. Sizing
m. Resistance of writing papers to feathering
n. Water penetration
o. Water absorbancy
p. Gloss
q. Opacity
r. Oil absorbancy
s. Fibre composition

Part-II prescribes methods of tests for paper for special purposes not covered in IS: 1060 (Part-I). These are

a. Bursting strength, wet
b. Tensile strength,wet
c. Bending test
d. Cupping test
e. Exudation test
f. Bleeding resistance
g. Fastness to light
h. Brightness
i. Water vapour permeability
j. Estimation of alpha cellulose content
k. Estimation of lead and lead compounds
l. Estimation of chlorides (water soluble)
m. Estimation of sulphates (water soluble)
n. Estimation of fatty and/or rosin acid extractable soluble
o. Estimation of matter soluble in ether, and
p. Estimation of benzene soluble in matter

Part-III Prescribes methods of test for paper and board for special purposes not covered in IS (**1060 Part-I and Part-II**), these are-

a. Wax absorptiveness
b. Stiffness
c. Wax pick number
d. Surface pH
e. Water penetration
f. Alkali staining number
g. Alkalinity, and
h. Copper number

Table-1 gives a list of standards available for paper and board testing with different organizations)

3. SALIENT FEATURE OF INDIAN STANDARDS

3.1Standard atmospheric conditions

IS 1060 (Part-I) specifies a relative humidity of 65±2 percent and a temperature of 27±2 °C, provided that in a given series of experiments the temperature does not vary by more than ±1 °C. These conditions are suitable for tropical climate & one among the three set of conditions recommended in ISO 187 i.e.

i. 50±2 % relative humidity and 23±1 °C temperature
ii. 65±2 % relative humidity and 27±1 °C temperature
iii. 65±2 % relative humidity and 20±1 °C temperature

Most of the European countries adopt 23/50 (ii) as preferred atmosphere, however, atmosphere 28/65 (iii) is also widely used

3.2Units

English speaking countries for quite sometime have been using FPS system (foot, pound, second) of measurement. In other countries, metric system (meter, kilogram, seconds) was more common. Now most of the countries are changing to (SI) system international which is based on metric system but differs from it is some ways. In India use of SI units in reporting test results on paper and board is becoming more popular. Some of the important units along with factors needed to convert customary units into recommended SI units are given in **Table-2.**

4. INCREASING ROLE OF ELECTRONICS & COMPUTERIZATION

Earlier instruments used for measuring various properties were mechanical in nature & less precise. With the induction of advanced electronic & computerization in every field, paper testing instruments too have become much more sensitive, accurate & faster. R & D institutions like Central Pulp & Paper Research Institute (CPPRI) & big mills in India too have started using all the latest instruments available elsewhere in the developed countries. Alwetron TH-1 Tensile tester, Data colour 2000, Kajaani fibre length analyzer are some of the examples.

TABLE-1
STANDARDS FOR PAPER AND BOARD TESTING OF DIFFERENT ORGANIZATIONS

	Organisation and number of standards									
Property	**ISO**	**BIS**	**SCAN**	**BS**	**DIN**	**ASTM**	**TAPPI**	**CPPA**	**APPITA**	**OTHERS**
Grammage	536	1060	P 6	3432	53104	D 646	T410	D 3	P 405	-
Thickness	534	-	P 7	3983	53105	D 645	T 441	D 4	P 426	-
Bulking thickness	438	1060	P 47	-	-	-	-	-	P 427	-
Bursting paper	2758	1060	P 24	3137	-	D 774	T 403	D 8	D 403	-
Bursting board	2759	-	P 25	3137	5314	-	T 807	-	D 438	FEFCO 4
Tear	1974	1060	P 11	4468	53128	D 689 D1424 D1922	T 414	D 9	P 400	-
Contact angle	-	-	P 18	-	-	D 724	T458	F 3	-	-
Compression	2872	-	-	-	-	D 642	T 804	-	-	FEFCO 5
CMT	-	-	P 27	-	53143	D2806	T 809	D 24	P 434	-
Tensile	1924	1060	P 38	4415	51221	-	T 404	-	-	-
Bendston Roughness	2494	9894	P 21	4420	53108	-	-	-	-	-
Gurley Air res.	3687	4006	P 53	2952	-	D 726	T 460	D 14	P 420	-
Cobb	535	1060	P 12	2644	53132	D2045	T 441	F 2	P 441	FEFCO 7
Brightness	2469 2470 2471	1060	P 3	4432	5033 53140 6167	-	T 452	E 2 E 1	-	-

ISO- International Standardization Organisation BIS- Bureau of Indian Standards
SCAN- Scandanavian Standard BS- British Standards
ASTM- American Society for Testing Materials DIN- German Standards
TAPPI- Technical Association of Pulp and Paper Industry
CPPA- Canadian Pulp and Paper Association
APPITA- Australian Pulp and Paper Industry Technical Association
FEFCO- Federation Eurppeene des Febriquants du cation ondulc

5. TESTING METHODS OF FEW IMPORTANT PROPERTIES OF PAPER & PAPER BOARD

5.1 Thickness

Thickness of paper is measured with micrometer as the perpendicular distance between two circulars, plane, parallel surface having 16±0.5 mm diameter. The micrometers used in our country employ a pressure of 100 KPa between measuring heads which is the recommended condition of ISO 534 Standard, whereas most micrometer used in U.S.A. use a pressure of 50 KPa. The difference in pressures gives difference in thickness values. If hand operated micrometer is used and the pressure foot is dropped on the paper, the pressure will be much greater than the specified which will give erroneous results. For accurate results, a motor driven micrometer that lowers the pressure foot at a constant rate are used. Different lowering speeds have been recommended in different standards (**Table - 3**).

TABLE-2

RECOMMENDED SI UNITS AND CONVERSION FACTORS

Property	Recommended SI units	Customary unit	Conversion customary to SL
Thickness	**m**	**mil**	**25.4**
Apparent density	g/cm^3	g/cm^3	1.000
Tensile strength	KN/m	lbf/inch	0.17513
		lbf/15 mm	0.29655
Breaking length	Tensile index (Nm/g)	Km	9.804
Tensile energy	J/m^2	ft.lbf/ft^2	14.5839
Tearing resistance	mN	gf.	9.80665
	Tear index	Nil	0.09804
Bursting strength	amN.m^2/g	Points mullen	6.8948
Burst factor	Burst index (ckPa/m^2/g	Nil	0.09804
Static bending Force	N	lbf	4.4482
CMT	N	lbf	4.4482
Ring crush	KN/m	lbf/6 inch	0.02919
Flat crush	Kpa	lbf/in^2	6.8948

TABLE-3

RECOMMENDED CONDITIONS OF INSTRUMENT FOR THICKNESS DETERMINATION IN DIFFERENT STANDARDS

Standard	Pressure (KPa)	Lowering speed mm/s
ISO 534	100	2
SCAN P 7	100	1
TAPPI T 411	50	0.8

In our country the instrument satisfying ISO conditions are being used. For accurate determination of thickness of the paper the measurements should be made 2 cm or more away from the cut edges of the test piece. The thickness is expressed in inches in the English system whereas in SI system it is reported in micrometers.

5.2Tensile strength

There are two basic types of tensile tester. The older type of instruments uses a weighted pendulum to apply the stress at a constant rate of loading. The never instrument uses strain gauge to measure the stress at a constant rate of elongation. The two type of instruments give slightly different results. Presently a more advanced version i.e. horizontal pneumatically controlled microprocessor based tensile testing instrument is available. This is capable of giving tensile strength, stretch, tensile energy absorption & tensile stiffness in single measurement. The precision and accuracy if far better than the earlier instruments.

In making tensile strength determinations, the time during which the load is applied in the test is an important factor. The strip should break within 20±5 seconds. To meet this, motor driven tensile tester are preferred over hand driven models. The other factors which influence the tensile strength are length and width of strip. The recommended standard length in ISO 1924 is 180±2 mm. The strip width of 15mm is in general use in our country as compared to 25mm width specified in Tappi test method. The tensile is reported in lb/inch width in English system, kg/m in metric system and Newton/m in SI units.

5.3Bursting strength

In bursting strength determination the test piece is placed over a circular elastic diaphragm and is rigidly clamped at the periphery but free to bulge with the diaphragm. Hydraulic fluid is pumped at a constant rate, bulging the diaphragm until the test piece ruptures. There are a number of bursting strength testers available namely

- the Cady
- the Mullen
- the Ashcoft, and
- the sehopper Dolen

These differ in size of test area, which affects the bursting strength. In our country most bursting strength measurements are made on the Mullen tester. In older models of bursting strength tester clamping lever is used to clamp the specimen and also pumping of glycerin is hand driven which is not accurate. The present version of bursting strength testers have pneumatic clamping with motor driven pump and electronic pressure transducer which provide a high level of accuracy and excellent reproducibility of measured values. In measuring the bursting strength of paperboard, the tester used require heavier diaphragm because of the high bursting strength being measured; a molded diaphragm is used in these tester to avoid the needed excessive pressure to expand the diaphragm.

The SI units for bursting strength are kilopascal. The factor for conversion of pound per square inch or points mullen to kilopascal is 6.89.

5.4Tearing strength

Tearing strength is the main force required to continue the tearing of an initial cut in a single sheet of paper. In this test different numbers of sheets are torn through a fixed distance and the work done in tearing the test piece is measured.

There are two basic designs of testing instruments.

I. Single Tear Tester

Single tear tester, in which the test pieces are cut to size usually on a guillotine and a single cut is made in the test piece after clamping on the instruments using a pre-set pivoted knife mounted on the instruments and tear test is carried out.

II. Double tear tester

Double tear tester, in which the test piece are cut and two initial cuts made, usually on a guillotine, before the test pieces are clamped in the instruments. International standard (ISO 1974) recommends the use of any one of the above instruments. In our country single tear tester is in use.

The tearing resistance in SI units is expressed in millinewtons.

5.5Folding Strength

For measuring the folding strength the following instruments are used.

- Schopper (German instruments)
- MIT (development by the Massachusetts Institute of Technology)
- Kohler Molin (Swedish instruments)
- Lhamargy (French instruments)

There is no consistent relation between these instruments.The ISO 5626 has recommended the use of any one of the above instruments. In our country the most common instruments used for fold determination are Schopper and Kohler Molin. In ISO method, in contrast with the traditional method of reporting folding endurance as number of double folds before rupture, the reporting of the logarithm (to the base 10) of the number has been recommended.

5.6Porosity

For measuring the air resistance of paper the instruments available are

- Gurley Densometer
- Bendston
- Bekk
- Sheffield

Gurley and Bendtson are common in Indian industry. In Gurley, the time taken for the flow of a standard volume of air through paper is measured whereas Bendtsen measures the amount of air passing per minute through the paper.

5.7Smoothness

Smoothness tests can be classified into five main types.

I. Visual inspection-binocular microscope/photographs under glancing illumination.
II. Pigment application-Microcontour test.
III. Profile recorders-Talysurf.
IV. Air leak-Bendtsen, Bekk, Gurley, Sheffield, Parker.
V. Proof printing-Vandercook, IGT.

For assessing the smoothness of paper for printing quality assessment, the best tests will be those that can be made under pressure akin to those used during printing.

Visual inspection with a binocular microscope and low angle lighting is the simplest and quickest way of obtaining some idea of the surface contours. The angle of lighting must be closely controlled, as a very small change can greatly alter the appearance of surface. Microcontour test is particularly useful for showing up deep pits. It can be misleading, however as there is a tendency for the pigment to be wiped out of shallow depressions. Also the rubbing, necessary to remove rather sticky ink may disrupt the surface of uncoated papers.

Profile recorders produce traces of the contours of the paper surface. These have the disadvantage that they are difficult to interpret, are not obtained under printing pressure and measure only a very small portion (about 2.5 mm) of the surface.

Air–leak instruments are inadequate in that the smoothness values obtained with such instruments are remarkably influenced by the paper porosity. In the Parker instruments, however the porosity effect has largely been eliminated. Smoothness can better be assessed by carrying proof printing under controlled conditions on an IGT tester or Vandercook press with low ink film thickness and a low pressure. It is however, rather time consuming and has the disadvantage that a numerical result is not produced.

5.8Stiffness

Stiffness measuring instruments are usually based on one of the following principles.

I. A measurement of the force required to bend a strip of paper through a given angle-Taber, L&W stiffness testers.
II. A measurement of angle through which a strip of paper is bent when subjected to definite force Gurley tester.

III. A measurement of the angle through which a strip of paper bends under its own weight when held in a horizontal fixed position-Clark stiffness tester.
IV. The length of a strip that is in resonance with a specified vibration frequency-Lhomargy stiffness.

In our country Taber stiffness and L&W stiffness instruments are common. Stiffness are expressed in millinewton.

6. STANDARD PUBLISHED BY BIS

The following list shows the pulp, paper & paper board related standards published by BIS which includes standards for the test methods for paper & paper board as well.

STANDARD NO.	TITLE
IS 1060 (Part 1): 1966	Methods of sampling and test for paper and allied products: Part 1(revised)
IS 1060 (Part 2): 1966	Methods of sampling and test for paper and allied products: Part 2
IS 1060 (Part 3): 1969	Methods of sampling and test for paper and allied products: Part 3
IS 1064: 1980	Paer sizes (second revision)
IS 1397: 1990	Kraft Paper (second revision)
IS 1398: 1982	Packing paper, water-proof, bitumen laminated (second revision)
IS 1763: 1961	Substance of paper & pulp board
IS 1774: 1986	Paper for permanent and semipermanent records (first revision)
IS 1775: 1981	Base paper for sensitized paper (first revision)
IS 1776: 1989	Folding box board, uncoated (first revision)
IS 1848: 1991	Writing and printing paper (third revision)
IS 2483: 1986	Ticket board (first revision)
IS 2617: 1967	Millboard, greyboard and strawboard
IS 2771 (Part 1): 1990	Fibreboard boxes: Part 1 Corrugated fibreboard boxes (second revision)
IS 2771 (Part 2): 1975	Fibreboards boxes: Part 2 Solid fibreboard boxes (first revision)
IS 2991: 1988	Base paper for waxed paper (first revision)
IS 3064: 1986	Handmade drawing paper (first revision)
IS 3302: 1986	Backing sheet for stencil (first revision)
IS 3303: 1997	Match paper for match box-Specification (second revision)
IS 3413: 2004	Base paper for carbon paper-Specification (second revision)
IS 3673: 1986	Alkali resistant paper (first revision)
IS 3962: 1967	Waxed paper for general packaging

IS 4006 (Part 1): 1985	Methods of test for paper and pulp based packaging Materials: Part 1 (First revision)
IS 4006 (Part 2): 1985	Methods of test for paper and pulp based packaging Materials: Part II (First revision)
IS 4006 (Part 3): 1985	Methods of test for paper and pulp based packaging Materials: Part III (First revision)
IS 4261: 2001	Glossary of terms relating to paper and pulp based packaging materials (first revision)
IS 4645: 1993	Code of practice for storage of paper and board (first revision)
IS 4658: 1988	Coated paper and board (art and chromo) (first revision)
IS 4661: 1999	
IS 4664: 1986	Glossary of terms used in paper trade and industry (second revision)
IS 5012: 1987	Pulp board (first revision)
IS 5134: 1977	Cellulose film (first revision)
IS 5195: 1969	Bitumen impregnated paper (first revision)
IS 5285: 1998	Paper stationery items for schools
IS 5457: 1969	Fibre analysis of paper and board- Method of test (first revision)
IS 6211: 1993	Sizes of folders and files
IS 6213 (Part 1): 1971	Code of practice for packaging of paper and board (first revision)
IS 6213 (Part 2): 1971	Method of test for pulp: Part 1 Water solubility of pulp
IS 6213 (Part 3): 1971	Method of test for pulp: Part 2 Determination of freeness of pulp
IS 6213 (Part 4): 1971	Method of test for pulp: Part 3 Determination of alpha, beta and gamma cellulose pulp
IS 6213 (Part 5): 1971	Method of test for pulp: Part 4 Determination of viscosity of pulp
IS 6213 (Part 6): 1971	Method of test for pulp: Part 5 Solubility of pulp in one percent caustic soda solution
IS 6213 (Part 7): 1971	
IS 6213 (Part 8): 1973	Method of test for pulp: Part 6 Copper number of pulp
IS 6213 (Part 9): 1975	Method of test for pulp: Part 7 Ash content in pulp
IS 6213 (Part 10): 1973	Method of test for pulp: Part 8 Beating , sheet making, preparation of hand sheets and testing
IS 6213 (Part 11): 1975	Method of test for pulp: Part 9 Bleach requirements and preparation of hand sheets for optical tests of pulp
IS 6213 (Part 12): 1975	Method of test for pulp: Part 10 Determination of kappa number
IS 6213 (Part 13): 1975	Method of test for pulp: Part 11 Determination of acid insoluble ash
IS 6213 (Part 14): 1975	Method of test for pulp: Part 12 Determination of

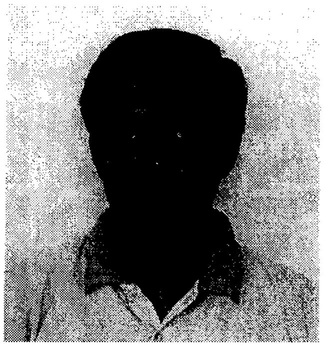
IS 6213 (Part 15): 1975	calcium content
IS 6213 (Part 16): 1978	Method of test for pulp: Part 13 Determination of copper
IS 6213 (Part 17): 1978	Method of test for pulp: Part 14 Determination of iron
IS 6213 (Part 18): 1979	Method of test for pulp: Part 15 Determination of manganese
IS 6213 (Part 19): 1985	Method of test for pulp: Part 16 Dirt count
IS 6213 (Part 20): 1984	Method of test for pulp: Part 17 Determination of saleable mass of pulp
IS 6213 (Part 21): 1984	Method of test for pulp: Part 18 Determination of chlorine consumption (degree of delignification)
IS 6481: 1971	Method of test for pulp: Part 19 Determination of alkali solubility of pulp
IS 6615: 1972	Method of test for pulp: Part 20 Determination of alkali resistance of pulp
IS 6622: 1972 IS 6671: 1987	Method of test for pulp: Part 21 Determination of dry matter content
IS 6715: 1972	Guide for principle uses and styles of fibreboard containers
IS 6956: 2001	General purpose packing/wrapping paper
IS 7063 (Part 1): 1973	Grease proof paper
	Specification for germination paper (first revision)
IS 7063 (Part 2): 1976	Master cartons for export of frozen sea foods and frog legs
IS 7063 (Part 3): 1976	Cover paper-Specification (first revision)
IS 7063 (Part 4): 1976	Methods of test for corrugated fibreboard: Part 1 Thickness of board
	Methods of test for corrugated fibreboard: Part 2 Edgewise crush resistance of board
IS 7149: 1973	Methods of test for corrugated fibreboard: Part 3
IS 7151: 1991	Water resistance of glued bond by immersion
IS 7161: 1973	Methods of test for corrugated fibreboard: Part 4 Determination of substance of the component papers after separation
IS 7162: 1973	Fibreboard boxes for canned sea foods for export
IS 7186: 1973	Corrugated fibreboard boxes for para-dropping of supplies (first revision)
IS 7601: 1983 IS 8113: 1976	Vegetable parchment or grease proof paper/aluminium foil laminate for wrapping butter
IS 8431: 1986	Waxed cartons for packaging of ice cream
IS 8460: 1977 IS 8970: 1991	Glossary of terms relating to paper and flexible packaging
IS 9028: 1976 IS 9032: 1978	Fibreboard drums for general purposes (First revision)
IS 9033: 1978	Primary cartons for packaging butter

IS 9042: 1978	Tracing paper (first revision)
	Wrapping tissue paper
IS 9313: 1979	Aluminium foil laminates for packaging (first
	revision)
IS 9493: 1980	Glossary of terms relating to paper sacks
IS 9588: 1990	Diazosensitized paper
IS 9894: 1981	Reproduction tracing paper
IS 9988: 1981	Method of measurement and expression of the
IS 10066: 1981	dimensions of paper
IS 10176: 1982	Corrugated fibreboard boxes for the export
IS 10177: 1982	packaging of glass jars and bottles filled with
IS 10279: 1982	processed foods
	Cartond for non-soapy detergents
IS 10287: 1982	Kraft liner (first revision)
IS 10328: 1982	Methods of test for smoothness/ roughness of
	paper
IS 10380: 1982	Waxed paper for bread and biscuits
	Corrugated fibreboard boxes for packing cigarettes
IS 10405: 1982	Fibreboard boxes for packing soaps
IS 10528: 1983	Icecream cups and lids
IS 11052: 1984	Overall trimmed sizes of articles of stationary that
	include detachable sheets
IS 11080: 1984	Holes for general filling of paper
IS 11087: 1986	Methods of expression of dimension and direction
	of manufacture of unprocessedwriting and printing
IS 11091: 1984	paper
	Methods of test for printing ink permeation of paper
IS 11227: 1985	(castor oil test)
IS 11324: 1985	Black centered board
IS 11357: 1985	Method of sampling empty paper sacks for testing
IS 11687: 1986	Methods of test for vertical impact drop test on
IS 11688: 1999	paper sacks
IS 11761: 1997	Method for determination of porosity of paper
	Paper for magnetic ink character recognition
IS 11844: 1987	cheque printing (first revision)
	Method of test for degree of curl of paper and
IS 12212: 1987	degree of sizing
	Gelatin for sizing of paper
IS 12490: 1988	Multi- ply paper sacks for carbon black
IS 12715: 1989	Composite containers for dry products
IS 12765: 1989	Base paper for tracing paper
IS 12766: 1997	Newsprint-Specification (first revision)
IS 12780: 1989	Multi-wall paper sacks for cement-Specification
IS 12808: 1989	(first revision)
	Corrugated fibreboard boxes for transport
	packaging of apples

	Corrugated fibreboard boxes for transport packaging of butter packed in primary cartons
	Glassine paper
	Polyvinyl alcohol for sizing of paper
	Map printing paper
	Paper, computer –Specification (first revision)
	Commercial stationary forms/books
	Base paper for one time carbon paper
IS 12810: 1989	Shorthand note book
IS 12999: 1990	Folding box board, coated
IS 13012: 1990	Sack kraft paper and extensible sack kraft paper
IS 13228: 1991	Corrugated fibreboard boxes for packing and transporation
IS 13892: 1999 IS 13975: 1994	Correspondence envelopes-Specification (first revision)
IS 13976: 1994	Axial (end to end) compression strength of composite cans, tubes and cores-Method of test
IS 14319: 1995 IS 14490: 1997	Method of end blow-off pressure test for composite cans
IS 14619: 1998 IS 14661: 1999	Laminated paper board pack for vanaspati-Specification
IS 14915: 2001	Plain copier paper-Specification
IS 15307: 2003	Release base paper-Specification
IS 15576: 2005	Toilet paper- Specification
	Filter paper-Qualitative, handmade-Specification
	Post card Specification
	Multiwall paper sacks for tea for export

It is seen that the Indian Standards are becoming more and more akin to the international ones which will facilitate better cooperation and understanding among the scientist and technologists and healthier trading practices with different countries of the world.

ROLE OF CHARGE MEASUREMENTIN PAPERMAKING-PRACTICAL ASPECTS



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ROLE OF CHARGE MEASUREMENTIN PAPERMAKING–PRACTICAL ASPECTS

S. TYAGI

1. INTRODUCTION

The objective of stock preparation is to prepare the furnish required for the production of paper on paper machine. In order to have the desired characteristics in produced paper, it is necessary that the non-fibrous additives be uniformly distributed, throughout the sheet and that they be associated with the long fiber fraction for maximum efficiency. This goal is largely accomplished by manipulating the surface and colloidal interactions or charge interactions in general, that occur among furnish components during stock preparation i.e. by manipulating the paper machinery chemistry of the system.

2. FURNISH CHARACTERISTICS PERTINENT TO PAPERMAKING CHEMISTRY

Paper making chemistry can be defined as the surface and colloid chemistry of papermaking furnish components. Some characteristics of typical furnish is discussed below.

2.1Resident species of typical papermaking furnish

Resident species are present at various concentrations and combine to make a head box consistency of 0.3% to 3.0% depending upon the grade of paper being manufactured. These include the following

- Dissolved electrolytes
- Suspended fibers
- Suspended fiber fines
- Suspended filler particles
- Water
- Surface active molecules like defoamers, dispersants and weed extractives.
- Dissolved polyelectrolytes like cationic starch, wet/dry strength agents, polyacrylamides
- Sizing molecules

2.2 Interactions among furnish compounds

Furnish compounds interact with one another in a variety of ways. These interactions may be controlled by the papermaker to achieve particular goals such as sheet formation or fines retention. They may be desirable or undesirable. Some are given below

- Aggregation of fibers, fillers and fines
- Adsorption of dissolved polymers onto fibers, fines and fillers.
- Adsorption of sizing molecules onto fibers, fines and fillers.
- Sorption of water by fibers, fines and starches.

2.3 Wet end interaction forces

Chemical interaction between any two entities involves both attractive and repulsive forces. The attractive interaction usually referred as primary and secondary bonding interaction. Primary bonding forces are those, which unite atoms into molecules or chemical compounds involving energies of 20-200 kcal/mol operating at interatomic distance of 2-3^{O}A. Hydrogen bonding is special case of secondary bonding with energy range of 3-5 kcal/mol and bond distance of 3A^{O}. **Table -1** gives some dimensions of furnish components of papermaking.

TABLE-1

SMALLEST DIMENSION OF FURNISH COMPONENTS

Components	Size	Dimension
Fiber	10-20 µm	Width
Filler particle	0.1-10 µm	Diameter
Fiber fine	<1-2 µm	Diameter
Dispersed rosin Size aggregate	<1 µm	Diameter
Dissolved polyacrylamide molecules	<<1µm	Radius of gyration

3. ORIGIN OF CHARGE ON PAPERMAKING FIBERS, FINES, FILLERS AND MINERAL PIGMENTS

The papermaking chemistry is a colloidal chemistry as most of the papermaking furnishes particles has colloidal dimensions and high specific surface areas. In addition, many of the interaction that occurs among these particles include secondary bonding forces. These are the two primary traits of colloidal systems and it is appropriate to define wet end chemistry in terms of surface and colloidal science.

An electrostatic charge develops at the solid/water interface when solids are dispersed in water. In papermaking systems, this charge arise from dissociated surface carbonyls and sulphonic acid group and the adsorption of substances such as hemicelluloses, dissolved lignin, retention aids etc. onto the surface. Mineral fillers like clay, TiO_2 also develop surface charge by ionization of the particle/water interface and by adsorption of other charged substance. The description of charges at solid/liquid interface was first outlined by Helmholtz [1] and was elucidated by Gouy [2], Chapman [3] and Stern [4]. **Fig. 1** depicts the currently accepted representation of the phenomenon [5-6]. Papermaking particles are generally anionic in nature. Therefore , **Fig. 1** is drawn with a negative charge at the surface. Ions of opposite charge to that of the particle surface (counter ions) are held closely adjacent to the surface by electrostatic and Vander Waals forces. The potential energy drops off rapidly in this region. A hydrodynamic slip place exists between the tightly bound counter ions and solvent molecules adjacent to the surface (stern layer) and the rest of the solution. The less ordered, diffuse portion of the system is called Gouy-Chapman region. The potential at the interface plane between the stern layer and Gouy-Chapman region is called the "Zeta Potential". The common methods of measuring surface charge actually gives values for zeta potential rather then surface charge. The common name applied to the entire system is the electrical double layer.

There is no overall net charge in the suspension due to the requirement of the electro neutrality. However, local areas of positive and negative potential exists close to the surface of the particles being observed. The strength of these potentials and distances involved determine the resistance of hydrophobic suspensions to aggregation.

4. PROPERTIES OF SPECIFIC FURNISH COMPONENTS IMPORTANT TO PAPERMAKING CHEMISTRY

The papermaking furnish is make up of fibers, fiber fines, functional additives and chemical processing aids. There properties play a vital role to papermaking chemistry

4.1 Fiber morphology

Fiber length contributes to the tendency of fibers to flocculate when suspended in water. The fiber width to diameter, which lies approximately in the colloidal particle range, contributes toward the specific surface area of fibers. There may be the presence of large percentage of non-fibers elements like vessel segments, ray cells and parenchyma cell. These elements exert greatest effect on paper making chemistry through their behavior as primary fines.

4.2 Fiber chemistry

Wood pulp fibers contain cellulose; hemicelluloses and lignin arranged in complex mixture in the cell wall along with extraction. The cellulose chains existing on the fiber surface contain hydroxyl group that participate in fiber-to-fiber bonding in paper. Cellulose hydroxyl groups on the fiber surface and within the fiber wall also interact strongly with water and are important to the swelling phenomena associated with refining and promotion of fiber-to-fiber bonding. Hemicelluloses contribute greatly to swelling of wood fibers during beating due to their strong interaction with water. They also promote the development of fiber-to-fiber bonding.

Residual lignin in papermaking fibers adversely influenced the ability of fibers to participate in strong inter fiber bonds, provides an inhibiting influences on the swelling of fibers in water and led to the formation of a bulky, rapid draining mat on the paper machine.

4.3 Influence of pulping process on fibers

A mechanical pulping process produces few chemical changes in fibers. The inner cell wall region is inaccessible and consequently, mechanical fibers do not swell much during beatings and refining. Chemical pulping processes produces both chemical and structural changes in fibers. Structurally, the dissolution of lignin and hemicelluloses results in more of the internal fiber cell wall surface becoming accessible to water and other materials in papermaking process. It also results in the chemical modification of cell wall constituents e.g. cellulose may be degraded by hydrolysis and oxidation reaction, resulting in polymer chains having reduced molecular weight and containing carbonyl and carbonyl groups. Hemicelluloses are dissolved, deacetylated and hydrolyzed during chemical pulping. Extractives are normally removed by chemical pulping.

4.4 Fiber fines

Fiber fines may be defined to be that part of furnishes which passes through a 200-mesh screen [7-8]. They vary widely in morphology may be rod shaped semi crystalline, fibrillar material having width of 0.1-0.5 μm and length of several micrometers. They may be divided into three categories [9].

I. Primary Fines

Fines that occurs naturally in the wood pulp sample. These include parenchyma and ray cell and some hardwood vessel cell.

II. Secondary Fines

Fines generated by refining.

III. Tertiary Fines

Fines are generated during the flow of un retained stock around the paper machine white water system. This material may arises as the result of the action of pump impeller, tank agitators etc.

Fines have major influences on paper machine drainage performance due to their high water sorption capacity (2 - 3 times that of fibers), which causes them to behave much like a gel.

5. SPECIFIC SURFACE AND COLLOID INTERACTIONS OCCURRING IN STOCK PREPARATION

5.1 Flocculation and coagulation Interactions

The compounds that participates in these types of interactions are the fibers, fines, fillers, size particles and other colloidaly dimensioned particles suspended in the furnish. Fiber-to-fiber flocculation occurs spontaneously at the consistencies found in the stock preparation due to both mechanical entanglement and colloidal interactions. If flocs persist and get incorporated in paper web they become undesirable. Flocs formation is generally prevented by the application of shear and turbulence at various points in the head box and on the forming table.

The formation of fiber- fines aggregates and the deposition of fillers; sizes and other additives on fibers are desired in order to minimize chemical additive use efficiency. The mechanisms involved in the flocculation and coagulation of fibers and fines materials include bridging, charge neutralization and patch formation. Flocculation and coagulation mechanism are also important in the formation of deposits in stock preparation and papermaking systems.

5.2 Adsorption of polyelectrolytes

A number of different furnish additives like retention aids, drainage aids, dry/wet strength agents, dissolved hemicelluloses occur as polyelectrolytes in papermaking systems. These materials all adsorb on to the fiber and colloidal particles present in the system. The chemical interactions occur in the adsorption of these particles include ionic attraction between charged groups on polyelectrolyte and on the particles (e.g. cationic groups on cationic starch and ionized carboxyl groups on fiber or fiber fines) and van der Waals attractions between polyelectrolytes and particle surface (e.g hydrogen bonding between the surface cellulose molecules on fiber and acrylamide groups in a polyacrylamide).

5.3 Ionic reactions between dissolved and suspended components

This generally includes the reactions like hydrolysis of alum to produce protons and lower the pH of papermaking system like the reaction between alum and rosin to produce a rosin size precipitate and the reaction between Ca^{2+} and SO_4^{-2}, Ca^{2+} and SO_4^{-2} etc.

6.0 CHARGE MEASUREMENT PRACTICES IN THE PAPER INDUSTRY

6.1Source and nature of surface charge on papermaking fibers and fiber fines

In kraft pulp fibers, carboxyl group located on hemicelluloses represents the largest source of charge. A typical range for carboxyl content in wood pulp is 5 -10-meq/100gwoods. Unbleached pulps also contain lignin phenolic groups. Mechanical pulps, such as CTMP contain sulphonic acid groups that originate from sodium sulfite treatment during pulping. With the exception of sulphuric acid groups, which are completely ionized at all pH values, the charged groups on fibers and fillers are mostly weak acids and their extent of ionization depends upon the pH of the system.

6.2 Surface charge on mineral pigments and fillers

In some mineral pigments like clay, isomorphous substitution of ions in the crystal lattices leads to charge imbalance and net particle charge. Clay exhibits both anionic and cationic characteristics. The ratio of two types of sites depends on the pH and chemical environment. Under acidic conditions, the edges are positive charged, while the faces are negatively charged. In some pigment, ion adsorption produces a surface charge.

6.3 Fundamentals of charge measurements

Electrokinetic measurements

In principle, there are several options of determining the surface potential of colloids. All these methods rely on one of the three electrokinetic phenomena given below. However, a potential cannot be measured directly. Instead, the particles have to be exposed to forces of some kind, such as to an electric field. (Electrophoresis). With in this electric field, the particle move all the faster the higher their surface charge is. From the migration rate of the particles the zeta potential can be theoretically calculated. Three electro kinetic methods are employed by papermakers to measure the zeta potential of paper making particles are

- Micro electrophoresis
- Streaming Potential
- AC streaming current

Of the three, micro electrophoresis was the first method developed and has been most commonly used. More recently, AC streaming current has been widely adopted for end point detection in dissolved charge determination. Also, an online streaming potential sensor is now available and is in use worldwide.

(a) Micro electrophoresis

In micro electrophoresis, particle dispersion is placed in a specially designed cell, an electric field is placed across the cell and the migration velocity of the dispersed, charged particle is measured. The quantity determined by the measurement is called the electrophoretic mobility (μm/s/v/cm). A schematic drawing of the electrophoresis cell is presented in **Fig .2.**

The electrophoretic mobilities can be converted into zeta potential by following equation[10]

$$\xi = \frac{f\mu\eta}{\varepsilon}$$ (A form of Helmholtz-Smoulchouski Equation)

where, ξ=zeta potential

μ =electrophoretic mobility

ε =di-electric constant of medium

η= viscosity of He medium

f = a constant

ε = di-electric constant of medium

η = viscosity of He medium

f = a constant

Typical values of electrophoretic mobility in papermaking systems range from -3 to +3, while zeta potentials typically range from -40 mV to +40 mV.

Practical considerations in microelctrophoresis measurement

- Micro electrophoresis measurement is made on the particles that lie within stationary layer. The liquid flow velocity affects the migration velocity of the particles in the electric field.
- Micro electrophoresis measurement requires that the particles lie completely in the stationary layer and they do not settle in the cell. These requirements mean that the measurements can't be made on papermaking fibers, but only on the fines fraction of a sample.
- Temperature gradients must be avoided in a cell or conventional currents in the sample slurry will invalidate the measurements.

- Measurements should be made as rapidly as possible and the polarity of the electric field alternated often. High conductivities and long measurement time may violate the results.
- The sample is sufficiently diluted so that individual particles don't interfere with one another when they migrate in the cell. Since zeta potential is a function of ionic environment, dilution with distilled or deioniozed water will alter the ionic conditions and change the zeta potential.

(b) Streaming Potential

In streaming potential measurement (Fig. 6.3) liquid is forced under a pressure gradient, through a plug formed from fibers, fines and other furnish components. Electrodes are placed on either side of the plug to measure the streaming potential established across the plug by the flowing liquid. The zeta potential can be calculated from the following equation [11]

$$\xi = (SP)(C)\ \eta/\varepsilon\Delta P$$

where SP = Streaming Potential;
η = viscosity of flowing liquid;
ε = dielectric constant;
C = Conductivity of liquid;
ΔP = Liquid pressure drop across the pad

Practical considerations regarding streaming potential measurement:

- Streaming potential measurement is made on a formed pad. All the components -including fines are contained in the measurement. This has advantages for on line instrumentation because no stock filtration is required prior to making a measurement.
- This measurement is sensitive to anything that affects the pressure drop across the formed pad. So precautions are taken to prevent such occurrence when setting the equipment.
- Entrained air or foam can interfere with streaming potential measurements. Air bubbles in the formed pad tend to impede the flow of liquid during the measurement and leads to erratic results.
- Stock temperature may also affect the streaming potential measurements due to its effects on the conductivity and viscosity of the liquid forced through the plug.

(c) AC Streaming Current Measurement

In this method, the streaming potential is measured over a thin slit rather than a packed plug of fibers. It utilizes a piston to force the liquid back and forth through a small clearance between the piston and enclosing cylinder. The pressure created by the piston motion produces an A.C current that can be detected by electrode imbedded in the sides of the cylinder. **Fig. 4** shows a schematic drawing of such a device.

One major consideration when using streaming current instrument is the absolute requirement for cleanliness.

The streaming current measurement does not necessarily reflect the exact zeta potential of suspended particles, but rather indicates whether a system is cationic or anionic.

Colloid titration

The different approaches adopted for the determination of level of interfering substance are based on a colloid titration method. This method is based on the fact that in many instances, oppositely charged; dissolved polyelectrolytes will form one to one charge complexes **(Fig. 5).** Experimentally, colloid titration usually involves titrating a sample with a standard cationic or anionic polymer that will react with its oppositely charged counterpart in the sample to form a one-to-one charge complex. An end point detection method is employed that indicates the point at which the first bit of titrant is added. The amount of titrated charge up to that point is equal to the amount of oppositely charged dissolved charge in the sample.

The colloid titration procedure can be performed either with indicator dye or use of the micro electrophoresis end point detectti0on or A.C streaming current end point detection.

The colloid titration with indicator dye end point has some disadvantages mainly as

- 'Vague' end point (pink/purple)-to-blue
- Paper mill dyes may interfere
- Manual test
- Tedious, time consuming titrations required.

These disadvantages lead to a search for end point detection methods other than dye color change. Two approaches have been employed frequently. First approach was to combine colloid titration with micro electrophoresis zeta potential end point determination and second is the use of A.C Streaming current end point detection.

Use of A.C Streaming current end point detection

During past few years it has become common practices to employ an A.C Streaming current detector zero point reading as the end point in colloid titration measurement. When this technique is combined with an automatic titrator, a completely automated colloid titration procedure can be carried out. The apparent ease of carrying out this determination has lead to its wide adoption by papermakers and chemical suppliers.

6. APPLICATION OF CHARGE MEASUREMENT IN CONTROLLING THE ANIONIC TRASH' IN PAPERMAKING

As mills are closing up their white water systems and using high level of recycled fibers, the level of dissolved and suspended interfering substance increases. These substances are collectively called as 'anionic trash'. These detrimental materials when increases beyond limit, they disturb the manufacturing process. They influence the process of machine runnability by decreased fiber swelling and delayed beating rate. This leads to higher energy costs. In addition, there is a loss of retention and dewatering which affect the paper quality.

The anionic trash, accumulated in the water circuit, results in reduced hydrogen bonding and as a consequence, results in lower fiber strength as well as reduced brightness. They also leave agglomerates in the water circuit, if i.e. the concentration is over the limit of their solubility, they may form complexes, with other components, like cationic additives. During sheet forming, these deposits will remain mainly on the wire and on pipelines, refiners, chests etc. The adverse effect includes the appearance of dark spots and holes as well as an increase in breaking frequency in the paper.

'Anionic trash' gives a negative effect on the efficiency of cationic additives like retention aids, starch, etc due to the neutralization reaction of anionic trashes with these components.

There are many ways to rseduce the amount of 'anionic trashes ' concentration as summarized below

- Separation of water circuit between wood pulp production and paper machine.
- Short storage time of the fiber stuff.
- Avoidance of erratic change in material parameters
- Selection of adapted points of dosage
- Optimizing of retention for water circuit release
- Timely avoidance of anionic trash by measuring the quantitative amount of charge in the white water.

All above parameters have their limitations. The best way to reduce the amount of anionic trash is their timely avoidance- by measuring the quantitative amount with any charge measuring technique and neutralizing these substance with the addition of an adsorbent like bentonite or a cationic fixing agents like alum, poly dadmac, hyamin, methylglycolchitosan (MGCH) etc.

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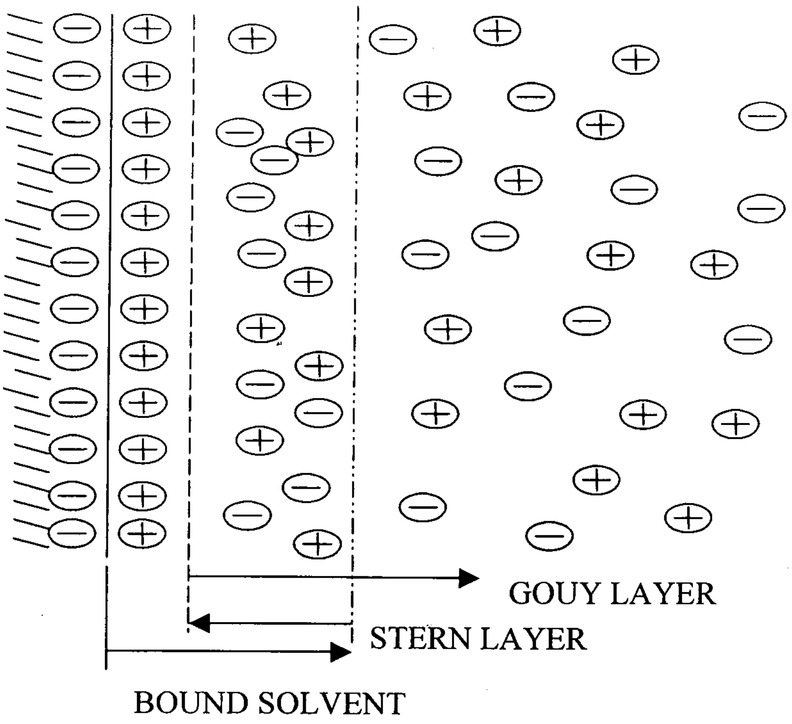


FIG. 1: REPRESENTATION OF ELECTRICAL DOUBLE LAYER EXISTING AT THE SURFACE OF HYDROPHOBIC PARTICLE SUSPENDED IN

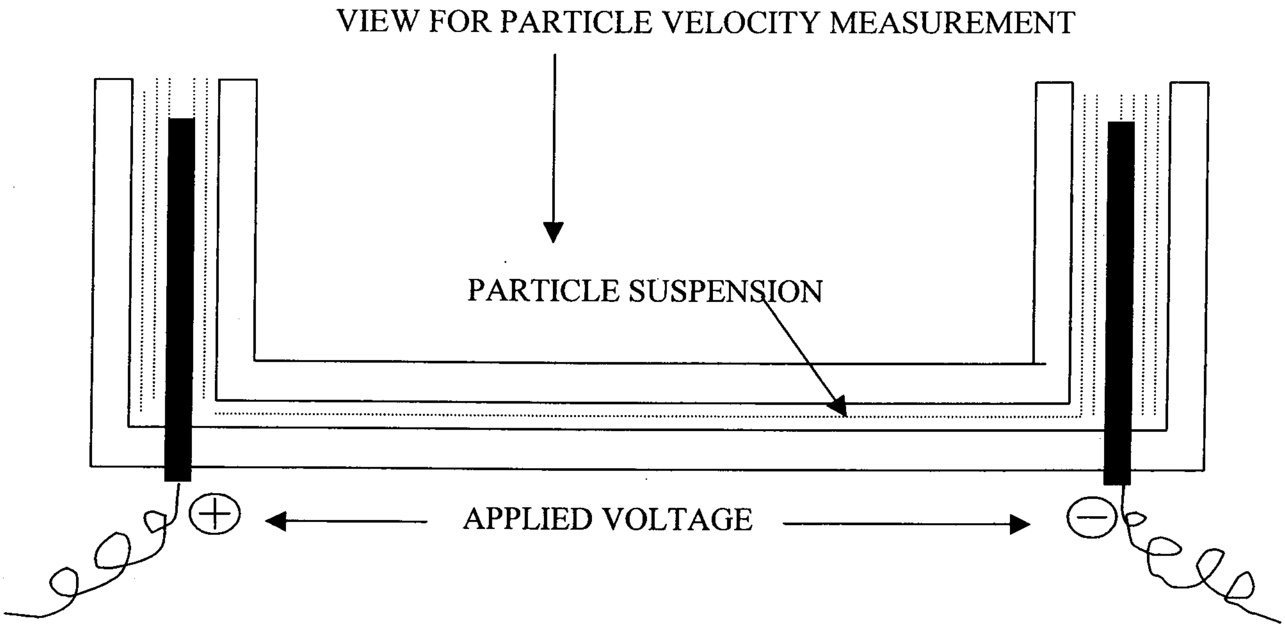


FIG. 2: ELECTROPHORESIS CELL

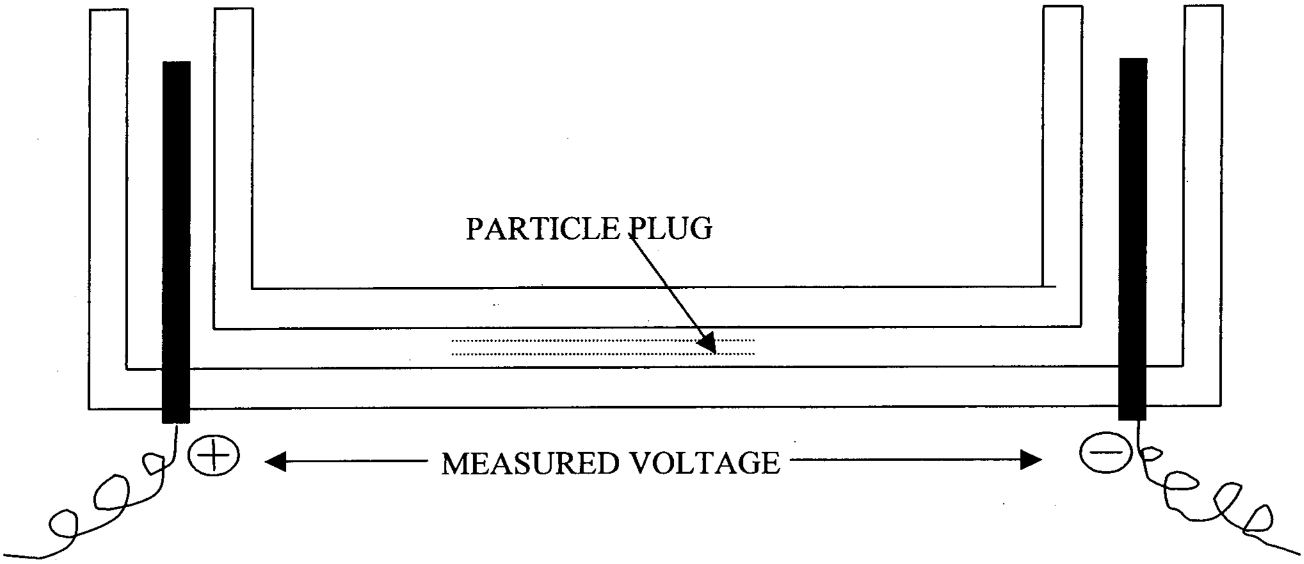


FIG. 3: STREAMING POTENTIAL MEASURING CELL

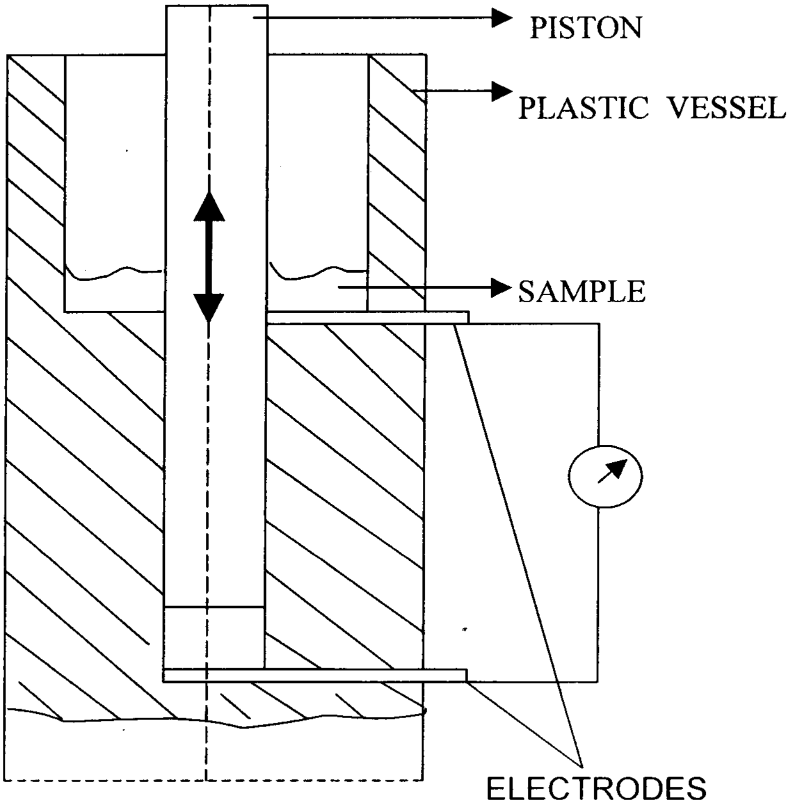
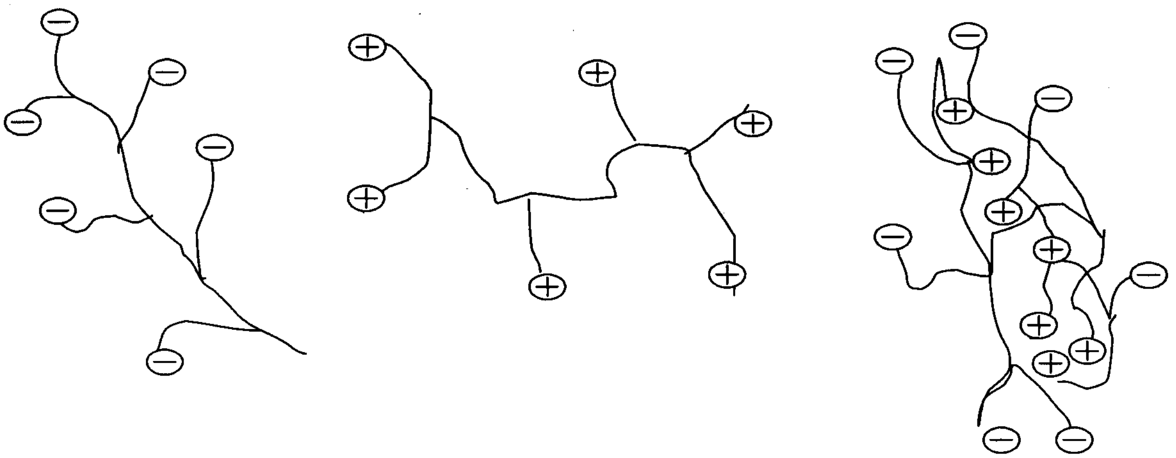


FIG. 4: AC STREAMING CURRENT DETECTOR



ANIONIC POLYMER

CATIONIC POLYMER

POLYMER COMPLEX

FIG .5: ONE-TO-ONE CHARGE COMPLEX FORMATION BETWEEN DISSOLVED ANIONIC AND CATIONIC POLYELECTROLYTES

FIBRE FURNISH ANALYSIS

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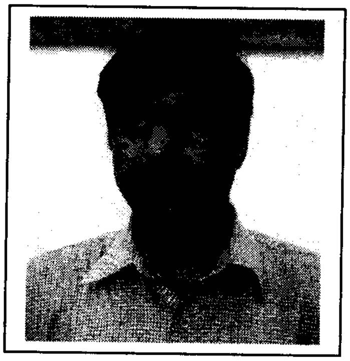


ABOUT THE AUTHOR

Dr.R.D. Godiyal holds Master degree in Physics & Ph.D. in the area of Nuclear Physics. He joined Central Pulp & Paper Research Institute in the year 1991. He has 14 years experience in the field of pulp & paper particularly pertaining to fibre morphology , fibre furnish, refining of different raw materials. Has wide experience in handling sophisticated instruments. Has been trained at PAPRICAN, Canada in the area of "New techniques developed for the study of internal & cross sectional structure of pulp fibre in SEM & CLSM and Zero Span Tensile Strength", Has also been trained at CTP, France in the area of "Understanding & control of pitch & deposits in papermaking process ". Has 50 publications to his credit.

FIBRE FURNISH ANALYSIS

R.D.GODIYAL

1. THE FIBER COMPOSITION OF PAPER

Fibers constitute the basic component of a paper sheet, and determination of fiber composition is essential in characterization of the paper. The methods of fiber analysis make possible the identification of the kinds of fibers present and determination of their relative amounts.

The identification of fibers rests principally on the application of stains, which produce a variety of colors depending on the source and previous history of the material. In addition, observation of the morphology of- the fiber is often helpful and sometimes necessary in identification.

Considerable training and experience are necessary to obtain the most reliable and accurate results. The behavior of fibers in mixtures of known composition should be observed from time to time to maintain the confidence of the analyst in his procedures Find judgments. A large collection of authentic fiber samples, known as the TAPPI library.

Procedures for fiber analysis are given in TAPPI Standard T401 and in ASTM D1030. The subject is treated in more detail in Graff and Isenberg's Pulp and Paper Microscopy Paper Microscopy 15). An atlas illustrating the colors produced upon staining many types of fibers has been published [1) . The procedures given in Sections II-X are based on the TAPPI Standard.

2. APPARATUS

I. Microscope: compound, preferably of the binocular type, which should be equipped with a mechanical stage and Abbe condenser. A magnification of 100 diameters is satisfactory for observation of stained fibers, but higher magnification is desirable in the study of fiber morphology. The eyepiece should be provided with a cross hair or pointer for aid in counting fibers.
II. Glass slides: 25 x 75 mm (I x 3 in.) ind No. 2 cover, glasses 25 x 25 mm. It is desirable to store the slides and cover glasses in ethyl alcohol-water (1:1).
III. Dropper: about 10-cm long and 8-mm inner diameter, fitted with a rubber bulb and graduated to deliver 0. 5 mi.
IV. Warming plate: electric, with level top having a black mat finish and with a control to keep the temperature of the surface at 50 to 60'C.
V. Dissecting needles: preferably of platinum-iridium alloy or stain- less steel.

3. PREPARATION OF THE PAPER SAMPLE

Portions (total about 0. 2 g) are torn from the paper or paperboard to provide a representative sample. The sample is defibered by one of the fol- lowing procedures.

3.1Procedures

- Ordinary papers

The sample is torn into small pieces, placed in small beaker, and covered with distilled water. The water is brought to a boil on a hot plate, the water is removed by, decantation, and the pieces are rolled into small pellets between the fingers4 The pellets are placed in a large test tube with a little water and shaken vigorously, with addition of small quantities of water from time to time, until the paper Is thoroughly defibered. Class beads may be added to the test tube to aid in the disintegration. The suspension is diluted to a consistency of about 0. 05%.

- Resistant papers

If the paper is not defibered by procedure 1, it is placed in a beaker, 1% sodium hydroxide solution is added, and the solution is brought to boiling temperature. The alkaline solution is removed and the paper is washed twice with water by decantation. The paper is covered with 0. 05 N hydrochloric acid and after several minutes the acid solution is removed and the paper is washed several times with water by decantation. The paper is rolled into pellets and defibered according to procedure 1. (Note: Any wool fibers in the paper will be dissolved by the treatment with

- Asphalt-treated papers

Asphalt, tar, and similar bituminous materials can be removed by extraction with chloroform, dioxane, trichloro-ethylene, or carbon tetrachloride in a Sox let or other extractor. Extraction can also be performed by soaking the paper in the solvent and repeat the extraction with fresh portions of solvent until the extract is colorless. In another method the paper is treated with hot kerosene (at the temperature of a steam bath) for 1 hr, pressed between blotters, treated again with hot kerosene and pressed, and then extracted with Portions of cold benzene until the solvent is colorless. The extracted paper is then defibered according to procedure 1or 2.

- Rubber-containing papers

The paper is extracted for 6 hr in a Sox let extractor with isopropyl benzene (cumene), dried, and then boiled in water to which a little wetting agent has been added. The paper can then usually be defibered in water, but sometimes the treatment with 1% sodium hydroxide solution is necessary.

- Parchment papers

The paper is placed in diluted sulfuric acid (25 ml H20 + 25 ml cone. H2SO4) cooled to 50 to 60'C. After the paper begins to disintegrate, the mixture is stirred and poured into Nvater (about 700 ml). In an alternative method, the paper is soaked for 5 min in concentrated hydrochloric acid, washed with water, and boiled in a 0. 5% solution of sodium hydroxide. The sequence is repeated if necessary. This paper is washed, treated with dilute hydrochloric acid, washed, and boiled in water containing a little wetting agent. The following method for disintegration of vegetable parchment has been. described. Paper (0. 5 g) is treated with I 00 ml of KMnO4 solution (20 g in 300 ml) at 27'C for 15 min. The solution is poured off, the paper is washed and placed in a 3% solution of oxalic acid for 90 see. The paper is again washed, heated in a 0. 1% NaOH solution for 20 min at 60'C, washed, treated with 0. 05 N HCl for 5 min, and again washed. The fibers can then be pressed into pellets for dispersion in the usual manner. A similar procedure is described in the SCAN method.

- Wet-strength papers

The paper is boiled for 10 to 20 min with a 5% aluminum sulfate solution washed with water, and defibered according to procedure 2. Alkaline-cured resins may be repulped with NAOH at 38oC and a pH of 10, or 0. 1% NACIO (on weight of fiber).

- Colored papers

The treatment necessary for removal of dyes depends on the type of dye present: (a) the paper is extracted with ethyl alcohol, ammonia, acetic acid, or hydrochloric acid; (b) the dye is oxidized by treatment with nitric acid or hypochlorite bleach liquor; or (e) the dye is reduced with sodium hydrosulfite, zinc hydrosulfite, stannous chloride, or zinc and hydrochloric acid.

- Other papers

Papers may contain sizing, coating, impregnating, and laminating materials, which are not removed by any of the above treatments. Other solvents may be suggested by reference to the characteristics of additives described in Chapters 19, 20, and 21.

4. PREPARATION OF SLIDES

A glass slide is dried and polished. Lines are drawn I in. from each end of the, slide with a glass-marking pencil or with an aluminum stearate solution (a 1. 5% solution of aluminum stearate in benzene). The lines retain the fiber suspension within the 1-in. squares at each end of the slide. Any dust or lint is removed from the slide with a small camel's hair brush, and the slide is placed on the warming plate. The test tube containing the defibered sample is shaken; a portion is withdrawn immediately with the dropper, and 0. 5 ml of the suspension is deposited on one end of the slide. A similar portion is deposited on the other end of the slide. The water on the slide is allowed to evaporate until the fibers are barely suspended; then the fibers are distributed evenly with a dissecting needle. The slides are left on the warming plate until they are completely dry.

5. STAINING

- The C stain is suggested for general analysis.
- The stain (3 drops) is applied to the fiber field on the slide and the cover glass is placed over it, with care to avoid inclusion of air bubbles.
- The slide is allowed to stand for 1 to 2 min, and the surplus stain is drain off by tilting the long edge of the slide into contact with a blotter.

6. EXAMINATION OF THE SLIDE

6.1Identification

The stained slide is placed in position on the stage of the microscope. For illumination, a 15-W "daylight" fluorescent tube, placed 10 to 12 in. from the mirror of the microscope, is recommended. The slide is examined for the different fibers, with attention given also to morphological characteristics. It is often desirable to prepare slides of authentic fibers for comparison with the sample.

6.2Determination

The field is moved with the mechanical stage so that the pointer is 2 to 3 mm from a top corner of the cover glass. Then the field is slowly moved horizontally and the fibers of each kind are counted as they pass the pointer. A single tally counter may be used to record the number of each kind of fiber, and repeated passes may be made to count additional kinds until all are accounted for. With a multiple tally counter, fibers of the various kinds can be counted from a single pass.

When all the fibers in one line have been counted, the stage is moved 5 mm vertically and the fibers are counted similarly in the second line. The fibers in a total of five separate lines, each 5 mm apart, are counted. If the slide has been properly prepared, the total fiber count will be between 200 and 300. If a single fiber passes the center of the pointer more than once, one count is made for each pass. If the fiber follows the center for some distance, it is counted only once. Each fiber in a fiber bundle (e.g., as in ground wood) is counted. Very fine fragments are ignored, but large fragments are mentally added together to give the equivalent of a whole fiber. The number count of each kind of fiber is multiplied by its respective weight factor to obtain the weighted count. The fiber composition for each kind of fiber is found from

$$\text{Fiber composition (\%)} = \frac{\text{Weighted count}}{\text{Total weighted count}} \times 100$$

Both fields on the slide are counted. If the results for the two fields vary for any type of fiber by more than the tolerances given below, one or more additional fields should be prepared and the average of results from all fields reported.

Percentage of Total	Tolerance, %
<20 and >80	±2
20 - 30 and 70 -80	±3
30 - 40 and 60 -70	±4
40 - 60	±5

The results are usually reported to the nearest 1%. A percentage of less than 2% is reported as a "trace.

7. WEIGHT FACTORS

Fibers from different sources differ markedly in average weight per fiber, and use of a factor is necessary in calculating the percentage by weight of each kind of fiber. Whenever possible, the factor should be determined for

the actual pulps used in the sample being analyzed. If this is not possible, the analyst should determine the factors for each type of pulp he is likely to encounter. The factors given in Table 5-1 were determined by Graff (21 and should be used only as a guide when better factors are not available.

8. COLORS PRODUCED BY 'C' STAIN

8.1Ground wood: vivid yellowish orange

8.2Softwood pulps

- Sulfite
  Raw: vivid yellow
  Medium cooked: light greenish yellow
  Well cooked: pinkish gray
  Bleached: light purplish gray to weak red-purple
  High alpha
  Unbleached: very pale brown to brownish gray
  Bleached: moderate reddish orange to dusky red

- Sulfate
  Raw: weak greenish yellow
  Medium and well cooked: strong yellowish brown to moderate yellowish green and dark greenish gray
  Bleached: dark bluish gray to dusky purple

8.3Hardwood pulps

- Sulfite
  Unbleached: pale yellow-green
  Bleached: weak purplish blue to light purplish gray
  High alpha
  Bleached: moderate reddish orange to dusky red
  Soda, sulfate, and neutral sulfite
  Unbleached: weak blue-green to dusky blue-green and reddish gray
  Bleached: dusky blue to dusky purple

8.4Non conventional fibres

- Rag: moderate reddish orange

- Abaca (manila fiber)
  Raw: light greenish yellow
  Unbleached and bleached: yellowish gray to weak blue and medium gray

- Jute
  Unbleached: vivid yellowish orange
  Bleached: light yellow-green

- Straw, bamboo, bagasse, flax and esparto
  Raw: light yellow to weak greenish yellow
  Unbleached and bleached: light greenish gray to dark bluish gray and medium purplish gray

- Japanese fibers
  Gampi and mitsumata: light greenish yellow to light bluish
  Kozo: pinkish gray

9. PREPARATION OF 'C' STAIN

9.1 Solutions

I. Aluminum chloride solution: AIC13- 6H20 (about 40 g) is dissolved in water (100 ml) to make a solution of 1. 15 specific gravity at 28'C.

II. Calcium chloride solution: CaCl2 (about 100 g) is dissolved in water (150 ml) to make a solution of 1.36 specific gravity at 28'C.

III. Zinc chloride solution: Water (about 25 ml) is added to dry ZnCL2 (50 g) to make a solution of 1. 80 specific gravity it 28'C. (Fused reagent-grade sticks in sealed bottles or crystals should be used; ZnCl2 from a previously opened bottle should not be used.)

IV. Iodide-iodine solution: Dry potassium iodide (0. 90 g) and dry iodine (0.65 g) are dissolved in water (50 ml). The reagents are mixed and crushed together with a little water, and water is added slowly with stirring until the solution is complete.

Solution 1 (20 ml), solution 2 (10 ml), and solution 3 (10 ml) are mixed thoroughly; then solution 4 (12.5 ml) is added and mixed well. The mixture is poured into a tall, narrow vessel and placed in the dark. After 12 to 24 hr, when the precipitate has settled, the clear portion of the solution is pipette off into a dark bottle and a leaf of iodine is added.

NOTE:

The solution should be kept in the dark when not in use. Fresh stain should be made every 2 or 3 months. The C stain is very sensitive to slight differences in composition, and care should be taken in its preparation and use. The solutions should be of the exact specific gravity specified and measured accurately with graduated pipettes. Dark-colored, glass-stopper dropping/bottles, preferably wrapped with black paper, should be used as containers.

TABLE – 1

WEIGHT FACTORS OF WOOD AND NONWOOD FIBRES

Sl.No	Fibre type	IS:5285-1969	CPPRI	TAPPI	SCAN G 4:90	BROWNING
1.	Rag	1.00				1.00
2.	Rag (cotton, flax & hemp)				1.00	
3.	Cotton linters			1.25	1.25	1.25
4.	Bamboo, ubld & ble sulfate, sulfite &soda	0.78				
5.	Bamboo pulp				0.55	
6.	Sabai grass, ubld & bld soda	0.48				
7.	Bagasse, unbld soda	1.20				
8.	Bagasse, unbld for boards		0.75	0.90		0.90
9.	Bagasse ubld & bld for papers			0.80	0.75	0.80
10.	Straw for board			0.65	0.60	0.65
11.	Straw bleached			0.35	0.35	0.35
12.	Wheat straw unbleached		0.69			
13.	Wheat straw bleached		0.55			
14.	Rice straw unbleached		0.47			
15.	Rice straw bleached		0.43			
16.	Khar grass bleached		0.43			
17.	Khai grass bleached		0.44			
18.	Flax and Ramie bleached			0.50		0.50
19.	Flax pulp				0.80	
20.	Flax shives				0.40	
21.	Hemp unbleached soda	1.13				
22.	Esparto			0.50	0.50	0.50
23.	Abacca and Jute			0.55	0.55	0.55
24.	Jute unbleahced		0.40			
25.	Jute stic unbleached sulfate	0.40				
26.	Sisal			0.60	0.60	0.60
27.	Indian spruce mechanical	1.40				
28.	Imported coniferous ubld & bld sulfate	1.04				
29.	Softwood ubld & bld sulfite & kraft			0.90		0.90
30.	Western hemlock			1.20		1.20
31.	Douglas fir			1.50		1.50
32.	Southern pine			1.55		1.55
33.	Alfa northern			0.70		0.70
34.	Alfa southern			1.50		1.50
35.	Softwood chemical pulps bleached				0.90	
36.	Softwood chemical pulps unbleached				1.00	
37.	Douglas fir inland variety				0.90	

38.	Douglas fir coastal variety				1.40	
39.	Southern yellow pine				1.40	
40.	Radiata pine				1.20	
41.	Softwood dissolving grade pulps				0.85	
42.	Softwood semichemical sulfite pulps				1.40	
43.	Softwood TMP				1.70	
44.	Softwood CTMP				2.00	
45.	Ground wood depending on its fineness			1.30	1.30	1.30
46.	Eucalyptus teretecornis unbleached		0.30			
47.	Eucalyptus teretecornis bleached		0.28			
48.	Salai unbld & bld sulfate	1.16				
49.	Salai ubld & bld mechanical	1.40				
50.	Hardwood soda, sulfate or sulfite			0.60		0.60
51.	Gum			1.00		1.00
52.	Alfa northern			0.50		0.55
53.	Birch, Aspen, Poplar, Beech				0.50	
54.	Maple, Willow, Hickory				0.40	
55.	Sweet gum, Black tupelo, Tulip, Poplar				0.80	
56.	Eucalyptus, Oak				0.45	
57.	Hardwood semichemical Birch				0.90	
58.	Hardwood semical gums				1.30	
59.	Hardwood mechanical pulps				0.90	
60.	Wool				3.10	

TABLE-2

DIAGNOSTIC FEATURE OF DIFFERENT PULPS (CLAUSE 12.5)- ALL FIGURES IN MICRON (μ)

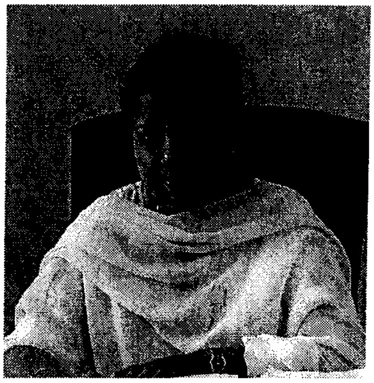
Sl No	Pulp	Fibres		Parenchyma		Vessels		Epidermal cells	
		Length	Width	Length	Width	Length	Width	Length	Width
I	II	III	IV	V	VI	VII	VIII	IX	X
1	Rag	2900 (780-8000)	24 (10-34)	-	-	-	-	-	-
2.	Imported coniferous (chemical)	3000 (800-6500)	45 (20-80)	-	-	-	-	-	-
3.	Spruce (Mechanical)	Incomplete	40 (30-60)	-	-	-	-	-	-
4.	Jute bast (Unbleached)	2000 (1500-5000)	20 (10-25)	-	-	-	-	-	-
5.	Hemp (Rope Cutting)	6000 (3000-21000)	20 (10-30)	-	-	-	-	-	-
6.	Salai (Chemical)	850 (350-1300)	25 (15-45)	120 (40-320)	35 (20-70)	470 (250-830)	170 (70-280)	-	-
7.	Salai (Mechanical)	Incomplete	25 (15-45)	-	-	-	-	-	-
8.	Eucalyptus tereticornis	900 (400-1600)	12 (6-30)	69 (30-160)	23 (15-40)	360 (200-500)	140 (70-200)	Absent	Absent
9.	Bamboo	2100 (400-5000)	15 (5-40)	110 (30-500)	35 (10-60)	650 (150-1200)	120 (40-250)	-	-
10.	Bagasse	1750 (250-4000)	23 (10-60)	375 (100-900)	100 (30-180)	600 (180-1600)	100 (30-220)	-	-
11.	Wheat Straw	1300 (600-4520)	13 (8-25)	237 (83-300)	50 (20-100)	240 (188-292)	61 (21-100)	116 (30-250)	18 (6-30)
12.	Rice Straw	1100 (600-4200)	7 (4-15)	123 (30-200)	18 (8-30)	51 (17-170)	50 (30-180)	135 (30-300)	10 (6-20)
13.	Sabai Grass	1500 (600-4000)	9 (5-15)	70 (30-220)	15 (10-50)	-	30 (15-45)	-	-
14.	Khar Grass	1500 (680-4600)	9 (4-20)	100 (40-420)	22 (10-50)	149 (60-230)	49 (30-75)	73 (58-100)	16 (10-20)
15.	Khai Grass	1500 (700-4700)	8 (4-15)	143 (30-380)	32 (10-73)	200 (52-560)	65 (30-100)	123 (25-210)	11 (6-20)
16.	Jute Stick. Core Wood	700 (300-1250)	30 (15-50)	90 (30-220)	30 (13-52)	400 (150-800)	80 (30-140)	-	-

10. REFERENCES

IS 12810: 1989	Shorthand note book
IS 12999: 1990	Folding box board, coated
IS 13012: 1990	Sack kraft paper and extensible sack kraft paper
IS 13228: 1991	Corrugated fibreboard boxes for packing and transporation
IS 13892: 1999	Correspondence envelopes-Specification (first revision)
IS 13975: 1994	Axial (end to end) compression strength of composite cans, tubes and cores-Method of test
IS 13976: 1994	Method of end blow-off pressure test for composite cans
IS 14319: 1995	Laminated paper board pack for vanaspati-Specification
IS 14490: 1997	Plain copier paper-Specification
IS 14619: 1998	Release base paper-Specification
IS 14661: 1999	Toilet paper- Specification
IS 14915: 2001	Filter paper-Qualitative, handmade-Specification
IS 15307: 2003	Post card Specification
IS 15576: 2005	Multiwall paper sacks for tea for export

It is seen that the Indian Standards are becoming more and more akin to the international ones which will facilitate better cooperation and understanding among the scientist and technologists and healthier trading practices with different countries of the world.

PROCESSING AND QUALITY CONTROL OF RCF PULP



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About The Author

Presently working as a senior scientist in CPPRI, the author has a Master's Degree in Chemistry with specialization in Organic Chemistry. The author with a brilliant academic record has dedicated almost 19 years of service to the Institute working in the area: Paper testing, Papermaking, Black liquor characterization, Chemical Recovery, Secondary fibre processing, Energy & Environmental Management.

She has a wide experience in the area of chemical recovery and secondary fibre processing. Has completed two projects on waste paper namely **"Technological Treatment Of Waste Paper"**, sponsored by AISPMA and **"Availability And Utilization Of Waste Paper"** sponsored by IPMA under which **an indigenous gradation system has been evolved** and proposed for implementation. Currently working on project **"Identification, Characterization And Removal Of Contaminants From Recycled Fibre"** under a plan scheme.

She was associated with development of Desilication technology also and currently working on development of color removal technology for liquid effluents.

She has undergone two months extensive training on "Energy Management in Pulp & Paper Industry" in Cia Suzano Mill in Brazil under UNIDO fellowship training programme. **She has around 30 publications to her credit and author/co-author of number of R&D reports, training & course manual.**

PROCESSING AND QUALITY CONTROL OF RCF PULP

RITA TANDON & A. SENGUPTA

1. BACKGROUND

Presently 38% of total paper production in India is contributed by recycled fiber (RCF) / waste paper. Over the years the use of recovered paper in the paper industry has increased and will continue to remain as a mainstay raw material for paper making in the years to come. The current recovery rate of indigenous waste, paper in India is very low which is mainly due to unorganized collection system prevailing in our country and may be increased to a level of 30% if concerted efforts are made to improve the collection mechanism. Nevertheless the industry will have to rely on imported grades of waste paper to meet the growing demand of waste paper for paper production. Presently about 80% of the waste paper requirement is met through imports from developed countries and remaining 20% is met through indigenously recovered papers. Due to cost reason the quality of imported fiber being imported is inferior and the outthrows and prohibitive percentage is high due to presence of contaminants and contraries in the recovered stock. The wastepaper use introduces many contaminants to the papermaking system as recycled fiber often contains a wide variety of contaminants such as pressure-sensitive adhesives, hot melts, waxes, inks, seam bindings, latexes, wet-strength resins etc. These contaminants can be classified as external or internal, as shown in **Table - 1**.

TABLE – 1

CONTAMINANTS IN RECOVERED PAPERS

External	**Internal**		
	Soluble	**Insoluble**	
		Nonstickies	**Stickies**
Sand	Starch	Plastics	Hot melts
Glass	Alum	Fillers	Wax
Wires	Soluble glue	Wet strength	PSA
Golf balls	Sizing agent	Ink	Latex
Sneakers	Notes: Notes: 1. PSA= Pressure- sensitive adhesives 2. Wax may or may not be sticky, depending on temperature. 3.Certain components of inks may be water soluble components may be sticky or nonstickies.		
Wooden boards			
Watches			
Engine blocks			
Kitchen sinks			
Styrofoam			
Cans			

As on today, the mills are using 100% of waste paper for the production of packaging grade papers and newsprints and a shift is also witnessed in production of Creamwove and Maplitho varieties using high quality imported waste papers. One of the important basics of effective processing of waste paper is the efficiency of contaminant removal, which depends mainly on selection of appropriate process configuration equipments and optimum process parameters for the particular waste paper/raw material furnish. In the past few years CPPRI has done extensive work in the area of contaminant control & effective processing of waste paper, which included repulping of wet strength paper, quantification and control of stickies and hot melts contaminants, development of new cost effective deinking technologies which have been successfully conducted on lab scale.

Production of clean stock from waste paper involves a number of process steps including pulping, screening, flotation deinking, washing etc. The primary task of recycled fiber processing is the removal of contaminants and elimination of their effects or both, as much as necessary to meet quality requirements. Removal of contaminants makes recycled fiber processing systems significantly more complex than systems for virgin fibers. Keeping this in view, CPPRI uses various methodologies to quantify these contaminants which are detailed below. The problems caused by these contaminants and the control measures to be adopted in dealing with them are also discussed.

2. METHODOLOGIES AND CONTROL MEASURES ADOPTED FOR DIFFERENT CONTAMINANTS

2.1 Fillers

Finely divided white mineral fillers are added to the papermaking furnish to improve the optical and physical properties of the sheet. These particles fill the spaces between the fibers to produce a denser, brighter, smoother and more opaque sheet. In some cases, the paper can also be made more cheaply because most fillers are less expensive than fiber. It is important to remove the fillers from RCF stock as the presence of fillers may limit the use of RCF for production of high grade papers such as tissue and Wr/Pr grade papers.

I. Quantification

The most important filler component used in paper production is china clay and calcium carbonate. The determination of filler component is evaluated through ash content in pulp or paper. To determine the ash, the pulp suspension should be dewatered with an ash free filter paper, dried and subsequently combusted. According to TAPPI T 211 an ignition temperature of 525^0C is recommended and according to TAPPI T 413 an ignition temperature of 900^0C is recommended. If ignition is carried out at 900^0C and 525^0C, calculating the ash of calcium carbonate from the difference between the ash at both combustion temperatures is possible. This considers the proportion (44%) corresponding to the molar masses that is lost as CO_2 at 900^0C. the calculation is shown below.

$\text{Ash}_{525°C} - \text{Ash}_{900°C} = CO_2$ volatile proportion
$\text{Filler Content}_{CaCO_3} = CO_2$ volatile proportion x (100/44)

The ash of china clay is the difference between the total ash at 525^0C and the filler content of the $CaCO_3$.
$\text{Ash}_{Clay} = \text{Ash}_{525°C} - \text{Filler Content}_{CaCO_3}$

The ash factor of clay is 1.13. This allows the filler content of clay:

$\text{Filler Content}_{Clay} = 1.13 \times \text{Ash}_{Clay}$

The total filler in paper or pulp is always higher than the ash due to ignition losses. It uses the following calculation

$\text{Filler Content}_{Total} = \text{Filler Content}_{Clay} + \text{Filler Content}_{CaCO_3}$

When performing combustion at two different temperatures, the dewatered and dried paper / pulp sample can first be weighed in platinum crucibles followed by ignition at 525^0C. After cooling the crucibles in a desicator, weighing is done again. Subsequently, ignition can be done again at 900^0C followed by cooling and weighing.

II. Control measures

The washing process can be applied very effectively to separate filler and other fine contaminants. Ash levels can be reduced to 1% in the final pulp by high-efficiency washers. Conventional washers and also flotation to a certain extent will also reduce the ash content of the pulp. The most common types of washers are gravity deckers, side hill screens and dewatering screens. **Table - 2** shows the commercial scale washer performance of different washing devices.

TABLE – 2

TYPICAL COMMERCIAL SCALE WASHER PERFORMANCE

Equipment	Inlet cons. %	Discharge cons. %	Effluent cons. %	1st stage Ash removal (%)
Side Hill Screen	0.6 - 1.0	3 - 4	0.15 - 0.35	55 - 60
Gravity Decker	0.6 - 1.0	4 - 6	0.04 - 0.18	50 - 55
High Speed Belt	0.6 - 3.0	8 - 15	0.18 - 0.38	65 - 85
Screw Extractor	2.0 - 4.0	8 - 12	0.25 - 0.40	40 - 45
Screw Press	2.0 - 4.5	18 - 28	0.15 - 0.28	35 - 40

2.2Ink / fluorescent particles

Most inks are two-component systems comprising a pigment for color and a vehicle that attaches the pigment to the substrate. The simplest form of ink is a carbon black pigment that is ground with linseed oil. Resins and other additives may be used to give the ink special properties.

I. Quantification

One of the most important unit operations for recycled fiber is deinking. The purpose of deinking is to remove printing inks and other substances that might affect the papermaking process or final properties of paper. The parameters most commonly used to assess deinking performance is the brightness of the pulp i.e. the optical property which includes:

- ISO Brightness
- Whiteness
- Deinkability Factor
- Dirt Count
- Residual Ink Concentration

ISO brightness, R_{457}

ISO brightness is an intrinsic reflectance factor determined with a brightness whose sensitivity to light agrees with a brightness meter whose sensitivity to light agrees with ISO Standard 2470. The peak of the sensitivity spectrum corresponds to a wavelength of 457 nm. This is the origin of the abbreviation R_{457}. ISO brightness measurement is most concentrated at the blue end of the spectrum. This means the determination is sensitive to very small changes in brightness. ISO 2470 also states that the UV radiation of the illumination must correspond to the relative amount of UV in the standard illuminant when measuring fluorescent objects. CPPRI uses Elrepho Brightness Tester based on the above principle for all brightness measurements.

Whiteness index

Whiteness is a colorimetric property. This is an important parameter for waste paper furnishes containing fluorescent matter. The impression of whiteness consists of the perceived lightness and the hue, i.e. how much the hue deviates from neutral. Normally the CIE whiteness is adopted measured under condition $D_{65}/10^0$, which is suitable for all white papers and paperboards. A significant portion of the method is the so-called UV calibration. This is a procedure to adjust the proportion of UV in the light source to correspond to the relative proportion of UV radiation in D_{65} light. CIE officially recommended the color spaces L, a, b or CIELAB system.

L A measure of perceived lightness. The scale is 0 to 100. Values near 0 reflect grayness and near 100 reflect whiteness.
a A measure of the hue on the red/green axis. A positive value for **a** means read and a negative value means green.
b A measure of the hue on the yellow/blue axis. A positive value for **b** means yellow and a negative value means blue

Whiteness Index is usually calculated from L, a, b values using the formulas, $L - 3b$, $L + 3a - 3b$ according to TAPPI 1216 sp-98. The L, a, b values in CPPRI are also measured using the Elrepho Brightness Tester.

Deinkability factor

Deinkability Factor is calculated from the ISO Brightness (R_{457}) of the unprinted deinked pulp (US) (i.e., made from unprinted paper subjected to the deinking process), the printed undeinked paper (BS) and the deinked pulp (DS) can characterize deinkability of a printed material. The formula is:

$$\text{Deinkability Factor} = \frac{\text{Brightness (DS)} - \text{Brightness (BS)}}{\text{Brightness (US)} - \text{Brightness (BS)}} \times 100\%$$

Dirt count

TAPPI method "Equivalent Black Area (EBA) and count of visible dirt in pulp paper and paperboard by image analysis" was developed for calibration of scanner-based image analysis systems. It uses calibration plates each having 16 dirt particles of defined size and optical density. This provides different brightness levels or contrast between background and dirt particles. The scanner based image analysis system for which the standard also includes several specifications requires calibration with these plates. The process is suitable for dirt particles of 0.02 – 3.0 mm^2. The smallest detectable particle diameter is 225 µm. CPPRI uses Paprican Micro Scanner based on the same principle for dirt count in pulp, paper and paperboard by image analysis. Sheets with a basis weight of 60 g/m^2 are usually suitable for this purpose.

Residual ink concentration

Deinked pulp always contains many ink particles with a diameter less than 225 µm and cannot be detected through Paprican Micro Scanner. Although these small particles are also part of the evaluation of optical characteristics such as brightness measurement, their determination can be very interesting for certain applications. For example, floatation is effective for particles with diameter >20 µm, but particles should be <20 µm for best washing results. To determine residual ink particles <50 µm, light microscopes used with image analysis evaluation programs with a low resolution limit of about 3 µm are necessary. The optical evaluation in the near-infrared area is suitable for determination of residual ink particles in deinked pulp. The light absorption there depends exclusively on the black ink content. CPPRI possess Paprican Ink Scanner based on the above principle and uses it for measurement of residual ink concentration. The instrument measures the ink particles of sizes as low as 8 µm while the upper limit is 2000 µm. Sheets with a basis weight of 60 g/m^2 are usually suitable for this purpose.

II. Control measures

The presence of specks in the finished paper is due to presence of ink particles, which have not been efficiently removed. Ink can be removed by washing, flotation technology, oxygen treatment or a combination of all. In certain cases a post bleaching step becomes essential for color stripping.

Wash deinking

In wash deinking, the ink is detached from the waste papers by using deinking chemicals, i.e. wetting agent and surfactant. The detached ink particles are generally less than 10 µm and well dispersed in the pulp slurry. These dispersed inks are removed from the pulp through repeated dilution and thickening actions. This washing technique is suitable for handling waste papers with never dried inks, i.e. ONP or old waste newspapers, which do not form visible specks in the pulping step.

Flotation deinking

The flotation deinking process is based on ink agglomeration chemistry. After the ink is detached from waste paper in the pulping step, the ink particles are agglomerated by using a suitable collector, e.g. fatty acid soap or surfactant. These agglomerates generally range from 20 to 100 µm in size and are hydrophilic or water loving nature. They would remain a stable suspension in the pulp slurry before the flotation cell operation. However, upon entering the flotation cell, the agglomerates are modified to become hydrophobic or water repellant with the addition of lime. In the flotation cells, these hydrophobic ink agglomerates readily attach to the air bubbles and are discharged as foam sludge. The flotation deinking technique can handle both ONP and coated waste papers, i.e. magazines, which form bigger ink particles in the pulping step.

Oxygen treatment

CPPRI initiated the study on using oxygen in combination with peroxide simulating the conditions maintained during EOP stage of virgin pulp bleaching sequence which finally resulted into significant improvement in the final brightness and dirt count in addition to improved strength properties. One of significant achievement was that, it could be accomplished without floatation deinking of the pulp stock. The entire process has been accomplished in four process steps viz, pulping, screening, O_2 treatment and washing.

Post bleaching

The post bleaching sequence for color stripping in recycled fiber pulp is mainly done through reductive bleaching agents, which modifies the chemical structure of dyes present in the pulp, causing them to appear colorless. The most common reductive bleaching agents available are Sodium Hydrosulfite ($Na_2S_2O_4$) and Formamidine Sulfinic Acid (FAS).

(a) Sodium Hydrosulfite ($Na_2S_2O_4$)

It is an excellent bleaching agent for color-stripping many dyes. As a reductive bleaching process,. there is no loss of yield in the bleaching stage. It offers a 10 to 15 –point brightness gain in a tower application. The pH is closer to neutral, making it compatible with other bleaching stages.

(b) Formamidine Sulfinic Acid (FAS)

FAS is an effective reductive bleaching agent, finding many applications in the secondary fiber mill. FAS has been found to be a better color stripper than hydrosulfite for many dyes. The FAS reaction product, urea, is also beneficial to water treatment systems.

2.3Wet strength resins

Wet strength resins are added during papermaking to impart wet strength to the paper. Due to presence of wet strength resins the paper causes processing problems when recycled leading to high energy consumption, lower yields and high reject rates. The wet strength paper keeps its integrity because of its chemical characteristic, which also defends it in the pulper unless it is attacked with the right combinations of chemical, thermal and mechanical energy. The wet strength resin in paper forms a cross-linked insoluble, covalently bond network of organic polymer and hemicelluloses, between and/ or around the fiber contact. Depending on the chemical nature of the resin, the network may become insolublised by resin-resin cross-linking and or by cross-linking of cellulose or hemicellulose through resin molecule.

I. Quantification

It is important to identify the resin type in the paper before it is subjected to repulping since different resins would require different chemical treatment based on their chemistry. Following tests conducted in CPPRI have been found very effective in identifying the resin type on the paper, which subsequently facilitated in formulating the treatment method for repulping as shown in **Table – 3**.

TABLE – 3

IDENTIFICATION METHOD FOR RESIN TYPE

	Test conducted	Inference
Test (I)	Determination of wet strength as percentage of dry strength (Tappi method T- 456-OS-68)	Differentiates temporary and permanent resin
Test (II)	Extract pH	Differentiates acid curing and/ alkaline curing resin
Test (III)	Solvent extraction (Al/Ben.)	Quantifies resin content

In CPPRI the measurement of wet strength of papers as percentage of dry strength is done using Wet Web Strength Tester.

II. Control measures

The process is a combination of chemical and mechanical treatment since process chemistry plays a vital role in repulping of wet strength paper.

Chemical treatment

Extensive studies conducted at CPPRI reveal that Selection of appropriate chemical is essential since different resins will require different pH conditions during repulping and each chemical added has a specific role on repulping chemistry. The different chemicals are:

(a) NaOH

In unbleached grades, treatment with strong alkali (pH >11.0) at 70°C may work by saponifying the ester cross-links between resins and hemicelluloses and is more effective in the range of 60-80 °C.

(b) H_2O_2

The secondary amine and tertiary amines are susceptible to oxidation, which predominates at low pH when strong oxidant is used. With the use of strong oxidant two pH process is more preferable to single pH process.

Process variables

The important process variables affecting the repulping efficiency are pH, Temperature, Consistency, time and Shear rate. Besides high shear rate, pH temperature and consistency have been found to have profound influence on repulping efficiency. Highly alkaline pH is more effective for unbleached grades containing PAE type resin while for bleached variety repulping at selective two-pH range is more favorable. Moderate to high temperature positively influences the efficiency. Repulping at high consistency would reduce the pulping time, chemical requirement as well as energy requirement.

Process equipment

The combination of right chemical and mechanical energy is pre-requisite to obtain optimal results for repulping of wet strength paper. High density pulpers are more suitable due to low energy and chemical consumption. Further with batch operations control of process variables is easier.

2.4Stickies

Stickies is a term that refers to hydrophobic components used in the manufacture of a paper product where these components are used as contact adhesives or hot melts. The most common components used as contact adhesives are styrene butadiene rubber (SBR), vinyl acrylates, polyisoprene, and polybutadiene. Hot melts are always mixtures of various components (e.g., wax and tackifying resins).

Stickies are gelatinous, tacky contaminants, which originate from the PSA's, while hot melts are broken fragments of the bindings in the magazines. Under normal deinking and paper machine operating conditions, hot melts are not tacky and do not stick as illustrated in **Table - 4**. Conversely, stickies remain tacky under these conditions.

TABLE – 4

TACKINESS OF STICKIES AND HOT MELT

	Tackiness		
Contaminant	25^{0}C	50^{0}C	65^{0}C
Stickies	Yes	Yes	Yes
Hot melt	No	No	Slightly

The characteristics of stickies include hydrophobicity, low surface energy, tackiness, and deformability due to which they frequently agglomerate and deposit on wire, felts, or other parts of paper machines or show on the sheet as spots.

I. Quantification

To control stickies related problems, a method for measuring the active stickies content must first be developed. Till date there is no standard method available to quantify stickies in a pulp sample. Most recycling plants, vendor companies and research organizations have developed their own test methods for stickies but all these methods have a variation to suit the testing location.

An methodology has been developed by CPPRI to quantify the macrostickies, microstickies, colloidal stickies concentration for stickies and hot melts monitoring in mills by integrating Brit Dynamic Jar, Pulmac Master Screen and Image Analyzer equipped with customized Verity IA software for measurement of stickies and hot melts. The detailed method has been given in **Annexure 1.**

II. Control measures

There are different approaches used to combat stickies, which include both chemical and mechanical approaches. These are

Furnish selection

Depending upon the nature of the furnish, final product and specific problem or customer needs, it is desirable to measure the concentration of stickies, plastics, clay, brightness, freeness, ground wood content or fiber length distribution.

Improved pulping & deflaking

The use of drum pulper also facilitates to keep the contaminant size bigger so as to be rejected by associated rotary screens.

Screening and cleaning

Recommended screening schemes utilize forward feed of accepts during secondary as well as the primary stage, using the same screen design and size in both stages.

Additives

Various chemicals evaluated for stickies control in CPPRI have shown variations in stickies and hotmelts control efficiency. The chemicals studied can be categorized according to their nature and control properties as shown in **Table – 5.**

TABLE –'5

ROLE OF DIFFERENT ADDITIVES

Sl. No.	**Chemical Property**	**Action**	**Control / Removed**	**Removal**
1.	Agglomerating (α-amylase)	Agglomerates the stickies	Through efficient slot screening	As rejects
2.	Dispersant (Surfactant)	Disperses the stickies in aqueous medium	Through water clarification	DAF
3.	Detackifier (Talc/PCC)	Passivates the stickies (high dose requirement)	Through fiber mat	Final Product
4.	Fixative (Polymers)	Fixes with fiber (low dose requirement)	Through fiber mat	Final Product

Management of Back water system

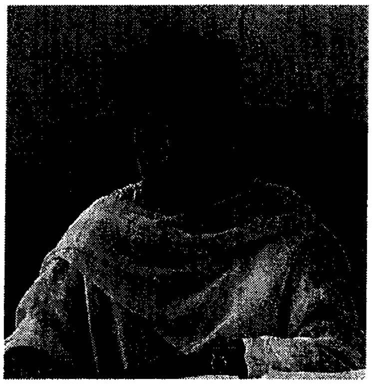
It is very essential to have a proper management of back water system since the process streams that contained high levels of extractives (polymeric compounds) were those rich in fines such as filtrates from dewatering equipment and the back water system in general.

3. CONCLUSION

Although there are no easy answers to waste paper related problems, an organised approach to identify and solve the problems will be rewarded. Adoption of the appropriate technology or combination of technologies (chemical & mechanical treatments) must be co-ordinated into a comprehensive control treatment program specifically designed to solve each individual mill's wastepaper recycling problems. An understanding of mechanical influences and limitations and the optimization of the process and wet-end chemistry will maximize the effectiveness of the treatment

programme. Good problem investigation techniques to identify the causes of a problem will enable the correct technology to be chosen to provide the most cost effective treatment. Contaminant control is the key to making wastepaper payoff.

QUANTIFICATION OF MACRO, MICRO & COLLOIDAL STICKIES AND HOTMELTS IN RCF STOCK



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About The Author

Presently working as a senior scientist in CPPRI, the author has a Master's Degree in Chemistry with specialization in Organic Chemistry. The author with a brilliant academic record has dedicated almost 19 years of service to the Institute working in the area: Paper testing, Papermaking, Black liquor characterization, Chemical Recovery, Secondary fibre processing, Energy & Environmental Management.

She has a wide experience in the area of chemical recovery and secondary fibre processing. Has completed two projects on waste paper namely **"Technological Treatment Of Waste Paper"**, sponsored by AISPMA and **"Availability And Utilization Of Waste Paper"** sponsored by IPMA under which **an indigenous gradation system has been evolved** and proposed for implementation. Currently working on project **"Identification, Characterization And Removal Of Contaminants From Recycled Fibre"** under a plan scheme.

She was associated with development of Desilication technology also and currently working on development of color removal technology for liquid effluents.

She has undergone two months extensive training on "Energy Management in Pulp & Paper Industry" in Cia Suzano Mill in Brazil under UNIDO fellowship training programme. **She has around 30 publications to her credit and author/co-author of number of R&D reports, training & course manual.**

QUANTIFICATION OF MACRO, MICRO & COLLOIDAL STICKIES AND HOTMELTS IN RCF STOCK

RITA TANDON &A. SENGUPTA

1. SCOPE

Macro/micro sticky classification is a dimension based system. The two screen slot sizes, 0.004 and 0.006 in, are specified since laboratories are using both of these sizes for stickies quantification. As the capability to screen out smaller sizes, i.e., narrower slot width, becomes feasible, the definition may change. At the present time, there is no well defined size range for micro stickies. One may set the micro stickies range between 5 microns and 100 microns (or 150 microns). Accordingly, stickies smaller than 5 microns may be classified as colloidal stickies. This given methodology has been developed by CPPRI for monitoring the stickies and hot melts under mill conditions. The method has been established on model pulp (stickies containing mill sample) and has been found to have good repeatability.

2. MATERIALS

I. Pulmac Master Screen (PMS) - for separation and collection of macro stickies from RCF pulp stock.
II. Image Analyzer (IA) - for stickies and hot melts counts
III. Dynamic Drainage Jar (DDJ)
IV. Soxhlet Apparatus

3. METHOD

3.1 Quantification of macrostickies by counts using Pulmac Master Screen and Image Analyzer

A widely used method for determination of contaminants is based upon concentration of macro stickies with the use of a laboratory screen. Several types of screens are available for concentration the contaminants, including Sommerville, Haindl, Pulmac Master Screen and Voith Valley Flat Screen.

CPPRI method involves use of Pulmac Master Screen for collection of macrostickies on the filter paper which is counted using Image Analyzer. The procedure is as follows:

Procedure

(i) Pulp sample equivalent to 50 gm O.D. at 5% Cy (or as such in case of low Cy) is slushed in the pulper at 50^{0}C to 60^{0}C temp. for 10 min (blank) and 20 min (with chemical addition)

(ii) The pulp is then diluted to 1% Cy and passed through Pulmac Master Screen using 100 micron (or 150 micron) slot.

(iii) The screened rejects containing stickies and hot melts are then collected on a filter paper and dried in an oven at 100^{0}C.

(iv) A silicone paper is placed on the dried filter paper which is sandwiched between blotter papers and then kept on a press for five minutes at 135^{0}C and at 68.9 Mpa. This enables the stickies to adhere on the filter paper.

(v) The silicone paper is then peeled off and the filter paper is washed off gently under water with a brush to remove the non-stickies components.

(vi) The wet filter paper is then placed in a separate water bath, held vertical, dripped free of excess water, and then placed in an acrylic box. It is then ensured that air from between the filter paper and the clear acrylic bottom of the box is removed. The flatbed scanner counts the hot melts which are visible only when wet.

(vii) The wet filter paper is then oven dried and placed on the flatbed scanner of image analyzer for stickies count using Verity Software customized for stickies count.

(viii) The results of hot melts and stickies are reported as counts per 100g pulp.

3.2Integrated Method for Quantification of Macro/Micro/Colloidal stickies Concentration in RCF pulp stock (expressed in g/100g) employing PMS, DDJ and Extraction technique.

This methodology has been devised/ developed by CPPRI which involves slushing of 50 gm O.D. pulp sample at 5% Cy followed by separation through Dynamic Drainage Jar (using 200 mesh or 75 microns) of two separate fractions

(i) Fraction A - accept containing micro + macro stickies (> 75 microns)

(ii) Fraction B - filtrate containing micro + colloidal stickies (< 75 microns).

Procedure

I. Quantification offFraction A for macro and micro stickies (> 75 microns)

(i) The accept pulp from DDJ is passed through Pulmac Master Screen (slot size used is 100 microns).
(ii) The reject is collected in a filter paper and macrostickies larger than 100 microns is quantified using image analyzer
(iii) The accept pulp of the Pulmac Master screen is then quantified for micro stickies using DCM extraction technique for 6 hours.
(iv) The difference between the initial and the final weight of the thimble along with pulp gives the weight of micro stickies.
(v) The particle size of the micro stickies thus obtained is (< 100 μm - > 75 μm) and is expressed as concentration in g/100g of pulp.

ii. Quantification of fraction for micro and colloidal stickies (< 75 microns)

(i) The filtrate obtained from DDJ (5 lit volume) is precipitated with NaCl at pH 2 (with the addition of sulfuric acid).
(ii) The filtrate is then filtered through ordinary filter paper.
(iii) The filter paper along with the residue is then oven dried and quantified for micro and colloidal stickies using DCM extraction for 3 hours.
(iv) The difference between the initial and the final weight of the thimble along with the filter paper containing residue gives the weight of micro and colloidal stickies.
(v) The particle size of the micro stickies and colloidal stickies thus obtained is < 75 microns and concentration expressed in g/100 g pulp.

iii. Estimation of total stickies in Pulp:

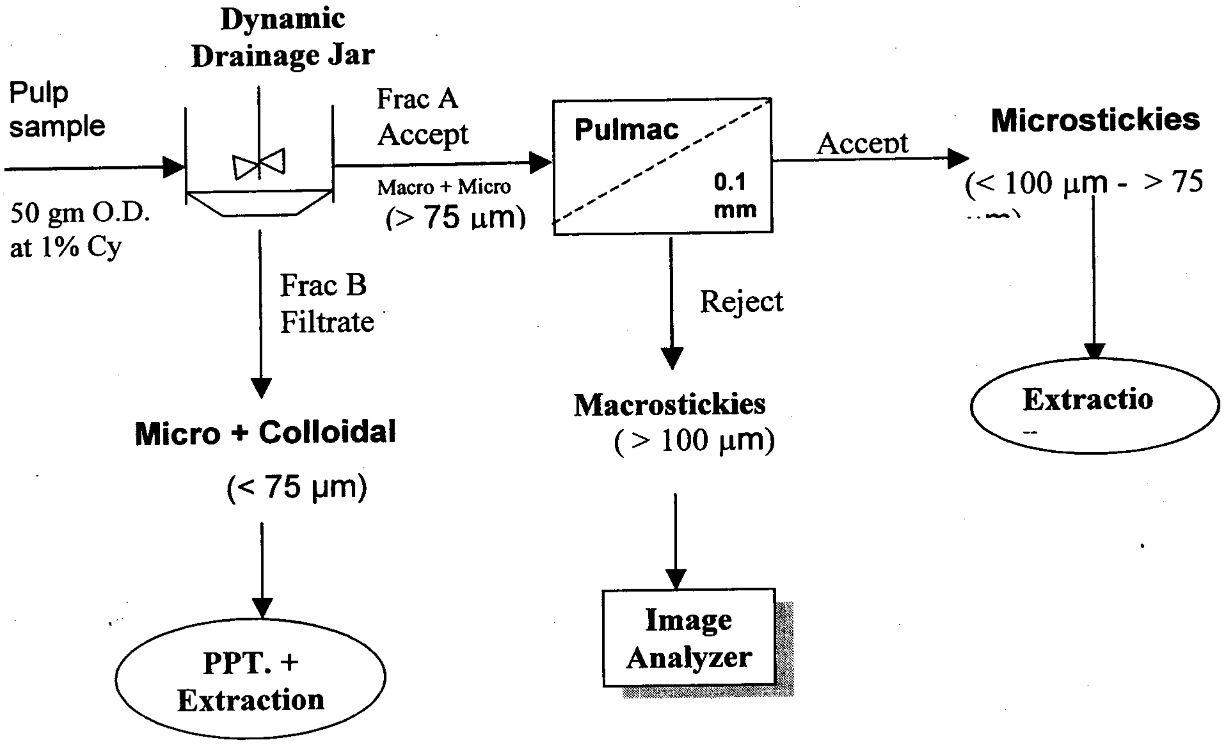
The total stickies content in pulp is quantified by using DCM extraction technique for 6 hours. The difference between the initial and the final weight of the thimble along with pulp gives the weight of total stickies. Macrostickies concentration expressed in g/100g of pulp is calculated as:

Macro conc. = Total conc - [Micro conc. + (Micro + Colloidal) conc.]
(in g/100g) (in g/100g) (in g/100g)(in g/100g)

4. Report

(i) The quantified values of macro stickies employing Pulmac Master Screen and Image Analyzer is expressed as stickies/hot melts counts/ 100gm pulp.

(ii) The quantified concentration of macro /micro / colloidal stickies employing integrated methodology is expressed as stickies conc. in g/100gm of pulp.



SCHEMATIC REPRESENTATION OF INTEGRADTED METHODOLOGY