V. G. Unkalkar A. G. Kulkarni R. S. Tripathi M. B. Jauhari

### Introduction

In order to meet the increasing demand of fibrous raw material for the expanding pulp and paper industry large scale plantations of Eucalyptus are being raised in our country Aspecies of Eucalyptus locally known as "Hybrid" but believed to comprise of hybrids of E. botryoids, E. camal-**E**. dulensis, tereticornis. E. robusta. and E. transversa has attracted special attention. This species was first adapted in forestry practice in the Mysore State and is named as Mysore hybrid or Mysore gum. Since this species grow more quickly and more prolifically under varying conditions of soil and climate its plantations are found all over India.

Since 1962 Mysore gum plantations are being raised by the West Coast Paper Mills Ltd. at Kuluwalli. The area falls within arid region of Mysore State and has one site quality. This place is about 50 miles away from Dandeli. Rainfall in this area is 75"

V. G. Unkalkar, Chemist A. G. Kulkarni, Chemist Dr. R. S. Tripathi, Research Officer. M. B. Jauhari, Senior Research Officer. The West Coast Paper Mills Ltd., Dandeli (N. K.), Karnataka

# In-Tree Variation of Wood and Kraft Pulp Quality of Eucalyptus Hybrid

Within tree (Eucalyptus hybrid) variation of basic density, sapwood content, chemical analysis, pulping behaviour, black liquor characteristics, mechanical properties of handsheets and morphology of fibres were studied. Wood samples were taken at bottom, the top and the mid point between these two from 9-10 year old sound trees.

Ash content showed practically no change with height of tree and did not exceed 0.60%. The basic density showed an upward trend from the bottom towards the top. The water solubles and the alcohol-benzene solubility decreased rapidly from the bottom to the top. Pentosan content increased from about 12.5% at the bottom to 15.8% at the top. Holocellulose content increased from 69% to 75%, lignin content decreased from 25% to 23%, from the base to the top of the tree. The acidity of wood extracts as determined by pH measurements showed a decreasing trend with increasing height of tree. The dilute caustic soda consumption was found to be more in the bottom portion when compared to middle and top positions in the tree.

The hardwood content was at its maximum i. e. 40% (by volume) in the bottom portion, which decreased rapidly to 18% in the middle. The top portion was practically free of heartwood.

High lignin, high extractive content and increased acidity in the bottom portion contributed to higher alkali requirements during pulping. This resulted in lower pulp yield. The pulp yield showed an increase in the middle and then a decrease towards the top The unbleached pulp brightness was low for the plup obtained from the bottom portion. However, the results for bleaching show that differences in unbleached brightness were not generally reflected in bleached brightness obtained with the standard bleaching conditions used. Further pulping quality is not related to changing basic density and for this matter other factors like the changing pentosan, lignin and extractive content must be considered.

to 80". In order to assess the potential of the Mysore gum for pulp and papermaking studies on the effect of age<sup>1</sup>: with intree variation of wood and kraft pulp quality were made. The present study describe the results obtained for in tree variation. a

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# Experimental

A plot of 54' x 42' containing 63 trees was marked in the Kuluwalli plantations raised in 1963-64 by the West Coast Paper Mills. The 63 trees included 13 dead trees. Out of the 50 living and sound trees, each 5th tree was felled. In all 10 trees were obtained and each debarked tree was measured for height, girth, etc. The data is as below :

Average height in metres	s 11
Average girth in cm.	36
Max. height in metres	17
Max. girth in cm.	61
Min. height in metres	7.5
Min. girth in cm.	20

From each tree billets of 2 metre length were cut 6" above the ground levels (bottom portion) 2 metre at the top (top portion) and 2 metre mid point between these two. These were separately weighed and the data for the weights is as below -

 Bottom portion
 ...
 128 Kg. A.D.

 Middle portion
 ...
 65 Kg. A.D.

 Top portion
 ...
 11.7 Kg. A.D.

 i.e., 11 : 5.5 : 1
 ...

Sample discs 1-2 cm thick were cut from both the ends of each billet representing the bottom, middle and top positions in the trees. Some of the discs were used for determination of heartwood content, and basic density. The heartwood content was estimated by forming a boundary between the sapwood and heartwood by spraying Conc. HCL, and allowing the discs to air dry. Purple colour was developed in the heartwood portion. Basic density was calculated from the measurements of the green volume determined bywater displacement method and the oven dry weight of the discs.

Sample discs from the bottom, middle and top portion were reduced to match stick size and finally ground in the Wiley Mill to pass through 60 mesh. The wood meal was subjected to analysis for principal chemical components essentially by Tappi standard methods. Hollocellulose was isolated by chlorite treatment. Six treatments were given and after each treatment the material was washed thoroughly with water. Additional tests for acidity, brightness and alkali demand of wood meal were carried out as below :

### Acidity

1 g. wood meal was treated with 100 ml. of freshly boiled distilled water for one hour at 98°C. The pH of the extract was measured after cooling with the exclusion of CO<sub>2</sub>.

### Brightness

Brightness was measured by forming a tablet 3-4 mm thick of the wood meal In the powder press and determining the reflectance at 457 nm in the Zeiss Photoelectric Reflectance Photometer ELREPHO.

### Alkali demand

The alkali demand was measured by treatment of 1 gram of wood meal with 50 ml O. LN NaOH over a boiling water bath for one hour and then titration with O.1N HCl using a pH meter to determine the end point.

The billets of the bottom, middle and top portions, after the removal of the sample discs were chipped in the mill chipper. Chips were screened with a Williams sieve and the fraction between 3 and 32mm were accepted.

For producing pulps of constant permanganate number and also black liquors with minimum tendency to lump formation, at high dissolved solids concentration. initially pulping tests were carried out in stainless steel bombs of 1 litre capacity, which were heated in the oil bath. The bombs were held in a rotating cradle designed in such a manner that the contents of bomb were mixed thoroughly. After establisthing the pulping conditions in the bomb digester, chips equivalent to 2.5 Kg O.D. were weighed out, and further pulping tests were carried out in a stationary digester of 16 litre capacity provided with indirect heating and ligour circulation. For carrying out the detailed analysis of the black ligonr, at the end of the digestion period, samples were collected through a sampler provided with cooling arrangement. Chemical analysis of the black liquor for Total alkali, Active alkali, Sulphated ash was carried out by Tappi standard methods. Other tests were carried out with some modifications. For concentrating the black liqour to varying solids, a rotary vacuum flash

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evaporator was used.

The unbleached pulps were tested for permanagnate number by T 214 m-50. The unbleached pulps were subjected to bleaching under standard conditions using CEHH sequence. Both the unbleached and bleached pulps were tested for physical strength characteristics by refining the pulps to four beating degree (°SR) in the laboratory valley beater, forming standard handsheets on the British sheet making machine and carrying out physical tests essentially by Tappi standard method. The wet web strength was determined by a procedure similar to the one described elsewhere<sup>2</sup>. The interfibre bonding strength was determined by the delamination method using a two ply sheet.

The bleached pulps were tested for their fibre, vessel and ray parenchyma content. Further dimensional data for the fibres, vessels and ray parenchyma was collected. Beside the figures given for fibre length, fibre width and cell wall thickness, coarseness figures are also given.

# Results & Discussion

The results (Table I) for basic density show that there is a slight increase from the bottom to the top of the tree. The heartwood content was quite high (40% by volume) in the bottom portion which decreased rapidly to 18% in the middle. The top portion was practically free of heartwood, as measured by the staining technique.

The proximate chemical analysis (Table II) showed that there is

TABLE I : Data for Basic Density, Heartwood Content for the<br/>Bottom, Middle & Top Positions of a Tree of E.<br/>Hybrid

	Bottom	Middle	Тор	
Basic density lb.OD wood/cft green vol.	38.1	39.4	41.2	
Heartwood content, % (on volume basis)	40.5	18.4	_	

# TABLE II : Proximate Chemical Analysis of Bottom, Middle & Top Positions of a Tree of E. Hybrid

<u></u>	Bottom	Middle	Тор
Ash	0.49	0.45	0.60
Cold water solubility	12.0	7.0	6.4
Hot water solubility	12.5	7.1	6.9
1% NaOH solubility	25.1	18.3	19.2
(1% NaOH-hot water) solubility Alcohol-benzene (1:2)solubility	12.6 5.8	11.2 2.6	12.3 1.5
Pentosans	12.5	13.9	15.8
Klason lignin	25.2	23.6	23.0
Chlorite holocellulose	72.6	78.9	80.4
Alpha cellulose	38.6	41.8	41.8
Beta cellulose	6.3	1.2	2.9
Gamma cellulose	24.3	29.1	. 30.2
Alpha, BetaΓ cellulose	<b>69.2</b>	72.1	74.9
Brightness (Elrepho), % of wood meal 457 nm	25.0	33.2	39.0
pH—hot water extract g-NaOH/100 g O. D. wood mea	4.7 1 6.5	5.4 4.5	5.3 4.3

All values expressed on 100 gm. oven dry material

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a distinct change in wood constituents within a tree. The water extractives, alcohol-benzene extractives and lignin content were more in the bottom portion and decreased towards the top. The holocellulose yield and the pentosan content showed an increasing trend with height of tree. The alkali demand of the wood meal for the bottom portion was found to be higher compared to middle and top obviously because of increased acidity and more extractives. The brightness of the wood meal varied from 25% th 39% from the bottom to the top. The reason for low brightness of the bottom portion is the increased heartwood content, which is normally rich in extraneous materials such as kinos and tannins.

The chips size classification data for the chips used in pulping tests is given in Table III. To avoid the variation in chips size and its effect on pulping quality, the chips size classification for the bottom, middle and top portions was kept as close as possible. The pulping results for the bomb cooks are given in Table IV. On the basis of the results obtained conditions of pulping were' suitably adjusted in the Research Digester cooks for producing pulps of nearly constant permanganate number i. e. 23. Only the alkali charge and the time at maximum temperature was altered. The results are given in Table V. The results show that the bottom portions of the trees have higher alkali

# **TABLE III : Chips Size Classification Data**

Screen mesh opening,			
mm	Bottom	Middle	Тор
< <b>25</b>	28.3	23.4	25.2
22	12.2	11.7	14.7
19	14.3	14.4	16.8
16	15.9	17.7	17.5
13	11.6	13.4	11.2
6	14.9	17.2	12.4
3	1.9	1.6	1.2
passing through 3 mm Bulk density of the	1.0	0.8	0.7
chips lb./cft.	19.6	20.0	19.6

# TABLE IV : Pulping Results of Bomb Cooks

			••				1.1	
	B	otton	1		Midd	le		Гор
Bomb Cook No.	1	2	3	1	2	3	1	2
						• • •		
Chemicals % as					•			
such (NaOH+Na <sub>2</sub> S)	19	21	22.5	18	19	20.5	19	20.5
Cooking Schedule-'	<b>Time</b> ,	in Mi	nutes	e di si			· •.	
70–120°C	45	45	45	.45	45	45	45	45
At 120°C	45	45	45	45	45	45	45	45
120-155°C	· · · · ·		35	-	, · · <u></u> · · .	35		35
120–160°C	40	40	<u> </u>	40	40		40	* <del>- 11-</del>
At maximum temp	60	60	75	45	60	60	60	( <u>0</u>
Spent liq. A. A.				24 <sup>1</sup>	· · .	÷.,		
gpl as Na <sub>2</sub> 0	5.7	7.0	10.8	5.4	8.1	12.6	7.9	12.1
pH-spent liq.	11.0	11.0	11.0	11.1	11.1	11.0	11.1	11.0
Total solids, % w/w	18.1	18.6	; °,	17.8	17.8	18.5	18.1	18.2
KMnO <sub>4</sub> No. of pulp	22.9	20.6	23.5	23.2	18.0	22.5	17.7	22.3
Screened yield, %	46.4	46.3	46.4	47.9	47.0	47.9	46.3	45.8
Rejects, %	2.5	2.3	×1.5	1.3	1.6	1.3	1.0	1.9
Total yield, %	48.9	48.6	47.9	49.2	48.6	49.2	47.3	47.7
Brightness, % (Elrepho)	30.4	31.7	32.0	34.9	36.7	34.9	39.4	40.0

#### **Constant conditions**

Chips equivalent to 300 g. O D. were used Sulphidity of white liquor, %16.8 Water was used for adjusting the bath ratio i.e., Chips: Liq=1:3.

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requirement and gives lower pulp yield, possibly because of high lignin and extracrive con-The brightness of the tent. unbleached pulp for the lower portions of the trees is also low which again could be accounted for the presence of more heartwood and in turn to the presence of extractives. Higher pulp yield was obtained for the middle position and then a decrease towards the top. It is interesting to see that the top portion though have less lignin, less extractives, still requires more alkali and give low pulp yield. It is likely that in the top it is the higher pentosan content which contributes to lower yield and higher alkali requirement. The pulping quality is at its maximum in the middle portion. Similar results have been reported by Batchelor et al<sup>3</sup> who showed that the pulping quality of Eucalyptus trees increased with height of tree to about 30 percent of the merchantable height and then decreased. The results for the characteristics of black liquors when producing pulps of constant permanganate number are given in Table V. The viscosity data is given in Table VI a and that for boiling point elevation, and specific gravity in Table VI b. Properties of the black liquors have remained practically unchanged. Small differences are noted in the flow behaviour as measured by the viscosity, higher values for the black liquor obtained from the top portion, and no explanation is given as with the TABLE V

Pulping Results & B. L Characteristics Research Digester Cooks

	· •		
	Bottom	Middle	Тор
Chemicals % A. A. as such	21.5	19.0	19.0
Chips : Liquor	1:3	1:3	1:3
Time Min $70-120^{\circ}C$	45	45	45
At 120°C	45	45	45
,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,,	40	40	40
,, ,, 120-100 C	60	50	45
,, ,, At 100 C	46.4	49.0	46.1
Screened yield, $\gamma_0$	1.8	1.6	1.9
Total vield, %	48.2	50.6	48.0
$KMnO_4$ No. (40 ml)	22.7	23.3	. 23.1
Brightness, % (Elrepho)	29.4	32.6	33.8
Black Liquor Characteristics		e e	
Total solids, w/w %	19.0	18.4	18.6
Organics, %	67.1	67.6	68.6
Inorganics, %	32.9	32.4	31.4
Specific gravity (20°C)	1.12	1.10	1.10
Active Alkali as Na <sub>2</sub> O, gpl	9.6	10.5	9.3
Effective ,, ,, ,, ,,	8.7	9.9	8.7
Total ,, ,, ,, ,,	47.5	49.6	45 0
Phenolphthelein ", "	21.3	19.7	18.2
Sulphur »	2.9	3.1	29
NaOH as Na <sub>2</sub> O, gpl	7.8	9.3	8.1
Na <sub>6</sub> S ., ., .,	1.8	1.2	1.2
NaoCO	25.2	19.6	19.0
NaOH-Org.	12.5	19.5	16.7
Surface Tension dynes/cm <sup>2</sup>	52.0	48.0	48.0
Calorific value— Cals/g. dry solids	3543	3400	3489

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Portion of	Total solids	Spindle	Brook (At 6	efield Visco RPM), cp	sity at	Re Vi	lative (I scosity,	R) & D cpatte	ynamic mp., °C	(D)	
nee	₩/₩ %		70°C	80°C	90°C	7	)	8	) ''	90	
<u> </u>	19.0		2.7	1.9	1.6	<b>R</b> 1.90	D 0.78	<b>R</b> 2.20	<b>D</b> 0.70	<b>R</b> 1.80	D 0.54
BOTTOM	35.7	LV <sub>1</sub>	25.0	20.0	14.5	12.0	5.0	90	3.5	9.0	3.0
20110.	50.2	LV2	225	145	105	106	41	76	2 <b>7</b>	52	16
<u> </u>	18.4	TTL.	2.5	2.0	1.7	1.9	0.78	1.70	0.61	1.70	0.52
MIDDLE	35.6		18.0	14.0	11.0	19.0	7.8	12.0	4.3	10.0	3.2
MIDDLL	50.8	LV <sub>2</sub>	200	155	98	95	38	86	30	63	19
	18.6	UL	3.1	2.8	2.4	1.90	0.78	1.84	0.66	1.20	0.37
TOD	35.9		32.0	21.0	16,0	21.4	8.6	14.8	5.3	14.7	4.7
IOP	50.6	LV <sub>2</sub>	350	195	125	140	48	- 111	39	95	28

TABLE VIa : Viscosity Data of Black Liquors at Different Concentrations

TABLE VIb : Some Physical Properties of Weak & Conc. Black Liquor

Portion of tree	Total solids w/w, %	Boiling Point, °C	Elevation of B. P. °C	Specific gravity at 20°C
BOTTOM	19.0	100.0	1.5	1.12
	35.7	102.0	3.5	1.22
	50.2	105.0	6.5	1.27
	18.4	100.5	2.0	1.10
MIDDLE	35.6	102.5	4.0	1.21
	50.8	,105.5	7.0	1.31
	18.6	100.0	1.5	1.10
ТОР	35.9	102.5	4.0	1.23
	50.6	105.0	6.5	1.28

limited data any explanation may seem to be an over simplification of the observation.

The bleachability of the pulps from the bottom, middle and top position remained practically unaffected. This is shown in the results given in Table VII. Except for the major difference in the viscosity which is much higher for the middle and top portion, practically the chemical consumption, shinkage during bleaching have not varied.

The pulp strength properties for the unbleached and bleached pulps of the bottom, middle and top portions were found to vary. It was found that for producing handsheets of constant bulk, the pulp from the bottom portion require less beating. This is shown in fig. 1, which could be

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<b>FABLE VII :</b>	Bleaching Dat	a of Pulps	Using	CEHH	Sequence
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	Bottom	Middle	Тор			
	· · · · · · · · · · · · · · · · · · ·					
Chlorination	· . ·					
Cl <sub>2</sub> added on pulp, %	5.5 Nil	5.5 Nil	5.0 Nil			
Residual chiorine, %	1411	1411	1411			
Extraction		· · · · ·				
NaOH on pulp, %	1.5	1.5	1.5			
Final pH	8.9	9.0	9.2			
Нуро І						
Av. Cl. added. %	1.5	1.5	1.5			
Av. Cl. consumed. %	1.38	1.42	1.44			
Final pH	7.4	6.7	7.3			
Hypo II						
Av. Cl. added. %	0.8	0.8	0.8			
Av. Cl. consumed. %	0.62	0.65	0.69			
Final nH	6.9	7.2	6.9			
Total Cl. added. %	7.8	7.8	7.3			
Total Cl. consumed. %	7.5	7.57	7.1			
Shrinkage. %	6.9	6.8	6.6			
Bld pulp yield % on O. D.						
chine hasis	43.3	45.7 <sup>,</sup>	43.1			
Prichtness % (Flrenho)	74.0	75.5	74.6			
Viscosity (CED), CD	20.2	26.8	25.4			

Constant Conditions	Consistency,	Temp.,	Time
and the second	%	°C	br.
Chlorination	3	30	1
Alkali extraction	5	55	1
Нуро І	5	45	1
Hypo II	5	45	1.5

interpreted to mean that there is increase in beating time to constant bulk from the bottom of the tree to about half way up & then a decrease. The physical strength characteristics of the standard handsheets from the middle portion are also on the higher side compared to those from the bottom and top portions at cons-

\*T/R=fibre wall thickness/fibre radius

tant bulk. Since at constant bulk properties like breaking length, burst factor Table VIII and speiific scattering coefficient Fig. 2 were found to vary, it is obivious that the reasons for the variation in the response to beating and strength development could not be ascribed to a single property like the basic density of the wood



Fig. 1—Bulk of standard handsheets against slowness of pulp



Fig. 2—Specific scattering coefficient against bulk for the bottom, middle and top portions.

and the relationship appears to be a complex one. Besides the chemical components morphology of fibre i.e.,  $T/R^*$  ratio also appears to be a factor determining these variations. The data on the morphology of fibres is given in Table IX. Bleached pulps obtained were tested for their fibre, vessel and ray parenchyma content.

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 TABLE VIII : Physical Strength Characteritics of Standard Handsheets of 60 g/m<sup>2</sup>

	S					
1	U Bottom	nbleached Middle	palp Top	Bi Bottom	eached Middle	pulp Top
All Props, At Bulk	1.60					
Beating time, min.	17.5	23.5	21.5	16.0	22.5	19.0
Schopper-						
Riegler, °SR	36.5	56.0	45.0	25.5	33.0	29.0
Breaking length, kn	1 9.6	9.6	8.9	8.0	8.3	<b>`</b> 7.7
Burst factor	70	76	73	59	69	61
Tear factor	101	111	96	100	113 -	94
Double folds(MIT)	150	320	160	110	110	75
Stretch, %	2.85	3.30	2.90	3.10	3.20	3.00
T. E. A., $J/m^{2}$	64	78	60	60	67	58
All Props. At Slows	ness, 45°	°SR				· . · ·
Beating time, min.	22.5	20.5	21.5	34.5	28.5	28.0
Bulk, cm <sup>3</sup> /g	1.51	1.66	1.60	1.38	1.49	1.43
Breaking length, kn	10.5	9.2	8.3	10.3	9.5	9.4
Burst factor	81.5	72.0	74.0	83.5	80.5	76.0
Tear factor	106	102	97	114	115	<b>99</b>
Double folds(MIT)	360	170	150	980	400	380
Stretch, %	3.1	3.1	3.0	4.5	3.7	3.7
<b>T. E. A.</b> , $J/m^2$	76	67	58	100	86	80

TABLE IX : Weight Proportion of Fibres, Vessels & Rays. AverageFibre Dimensions & Data on Pulp Fibre Coarsenessand Number of Fibers per Gram

	Bottom	Middle	Тор	
% on equivalent fibre with basis				
(a) Fibres	84.5	88.5	87.0	
(b) Vessels	2.0	3.7	5.3	
(c) Parenchyma	8.8	4.3	3.7	
(d) Ray cells	4.8	3.6	4.0	
Decigrex number, m/g100 metre	4.6	5.0	- 4.8	
Number of fibres 106/g pulp	27.8	23.4	28.8	
Fibre length, mm	0.8	0.85	0.72	
Fibre width, microns	13.2	11.0	12.4	
$2 \times $ Cell wall thickness, microns	8.6	7.4	6.8	
Lumen width microns	4.6	3.6	5.6	
T/R, %	60.0	73.0	65.0	

The interfibre bonding strength for a two ply sheet prepared from refined pulps of 42°SR was found to be 0.111, 0.091, and 0.097

Kg/mm<sup>2</sup> for the bottom, middle and top portions. The values for the wet web strength at varying beating degrees are given below:

	Bottom		Middle		Тор	
Slowness, °SR	33	42	31	44	31	42
Wet web strength, metres	44.0	50.2	33.4	42.5	38.4	48.7

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### Conclusions

- 1. Bottom, middle and top portions of Eucalyptus hybrid exhibit variation in heartwood content, proximated analysis, pulping behaviour and pulp properties.
- 1.1 The heartwood content is about 40% in the bottom portion, reducing to about 18% in the middle. The top portion is practically free of heartwood.
- 1.2 Both the holocellulose and pentosan content increased with increasing height of tree and as expected lignin content showed a decreasing trend.
- 1.3 The pulp yield is at its maximum in the middle portion.
- 1.4 The physical properties of the pulps were found to vary. An important trend is found in the increase in beating time (to bulk 1.60) from the bottom of the tree to the middle and then decrease. Similar trend was noted in the degree of beating as measured by the Schopper-Riegler. It increased considerably (to bulk 1.60) from the bottom to the middle and then showed adecrease. Incidentally the middle portion has given the highest pulp yield also. Similar trend was noted by Batchelor et al<sup>4</sup> who generalized that within the tree, those portions which had the highest yield required the most beating and reached lowest freeness.

2. The variation in pulp properties could not be related to a single effect. The relationship is complex one where basic density, fibre morphology & chemical components all exert their influence.

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