

PULPING AND BLEACHING OF MELIA DUBIA CLONE K10: A SHORT ROTATION PULP WOOD CLONE TO IMPROVE ENVIRONMENTAL AND ECONOMICAL PERFORMANCE

By

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Paper demand in India is constantly increasing due to increasing literacy rate and area need to grow more pulp wood is decreasing due urbanization. One way to meet the increasing pulp demand for more paper production, with the sinking land area is by increasing pulp and pulp wood yield per unit area. In this study, we have identified a short rotational Melia dubia (K10) pulp wood clone with lower lignin content .Pulping and bleaching studies of two and three years old Melia dubia (K10) was carried out and results presented in this paper. The screened pulp yield of (K10) two and three years old was 54.63% & 55.36% with respective kappa number was 27.3 & 22.5 for 16% TAA .Whereas screened pulp yield was 53.66 % & 54.85% and its kappa number was 25.7 & 21.6 for 17% TAA pulp. Keeping constant bleaching condition of Melia dubia K10 (K10) wood pulps can improve the pulp bleachability without affecting pulp strength properties, optical properties, and physical properties and improved bleach filtrate characteristics. The final bleached pulp brightness (Oxygen delignified and D0EPD1) ,was found to be 88.3% and 90.2 % ISO for 16% TAA (K10) pulp and 88.2% and 91.3% ISO for 17% TAA(K10) pulp. Tearing index of two and three year's old K10 bleached pulp was 9.22 & 9.24 mNm²/g for 16% TAA pulp whereas tearing index was 8.88 & 8.67 mNm²/g for 17%TAA pulp respectively. Two and three year old Melia dubia clone K10 can be used as short rotation pulp wood by the pulp and paper industry and this can improve environmental and economical performance of the mill.

Introduction :

During 2010 - 2014 global pulp and paper demand has grown by 7.6 million tonnes. Except china rest of the world fell from 300.3 to 295.8 million tonnes as growth in Latin, East Europe and other emerging Asia was offset by declines elsewhere (1). In the last ten years the intensity of paper and board demand has fallen significantly. Despite the continued focus on digitization, India's demand for paper is expected to rise 5.3% in the next six years.

Though India's per capita consumption is quite low compared to global peers, things

are looking up and demand is set to rise from the current 13 million tonnes to an estimated 20 million tonnes by 2020. An India Ratings report estimates India's per capita paper consumption at 9 kg, against 22 kg in Indonesia, 25 kg in Malaysia and 42 kg in China. The global average stands at 58 kg. This indicates there is a lot of headroom for growth in India (2). From a demand point of view, every one kg incremental per capita consumption results in additional demand of more than one million tonnes per annum. The government's sustained focus on literacy, increased consumerism and expansion in organised retail are expected to

positively affect paper consumption and demand in India. Growing consumerism, modern retailing, rising literacy and the increasing use of documentation will keep demand for writing and printing paper buoyant India (3). Besides, policy factors also have a key role to play in the growth of the domestic paper industry in India. The capacity expansion that took place in the industry through the last few years is now being absorbed due to the rising demand for paper in India. The sector, which faced challenge from increasing wood demand, lesser forest area is now facing shortage supply and increasing of pulp costs. Hence, there is

an increasingly growing demand to get quality pulpwood through plantation. Depletion of forest plantation areas in the country has badly hit the supply of fibrous raw material to the industry and hence more importance has been given to raise fast growing species for use as raw material for paper and cellulosic industries (4). In these circumstances *Melia dubia* has been identified as one of the potential pulpwood species (5). However the age of *Melia dubia* is amenable for pulpwood utility has not been assessed so far and this demands research on pulping characters of *Melia dubia* clone at various age gradations. Hence the current study for wood characterization for pulp and paper properties of *Melia dubia* (K10) wood species is conceived and designed under two years and three years age gradations.

Material and Methods

Sample collection: *Melia dubia* K10 trees from each age gradation of two and three year old was randomly selected from the farm forestry plantations during September, 2016. Five replicates of 1m length samples taken at Breast height (1.37 m) from the trunk of each tree and transported to the laboratory and chipped in the mill chipper. The screened chips was air dried for getting constant moisture homogenization for pulping. After getting constant moisture, the chip are packed in a polythene bag and stored for a day to get homogeneous moisture of the chip before cooking. Wood sample was prepared using Wiley mill to analysis the chemical properties and remaining samples were made into chips with a pilot chipper and used for analysis of physical and strength properties.

Pulping : The chips were pulped (alkali charge 16 % and 17% as Na₂O, bath ratio 1:2.8, cooking temperature: 165°C, time: 60 minutes, H factor: 800) in a Laboratory bomb digester. At the end of cooking, the contents of the bomb were discharged into a bowl followed by sequential washing with DM water, disintegrated in a rod mill for 20 minutes, screened on a flat -

slotted laboratory screen (slot width 0.20 mm), centrifuged, and then granulated. Total pulp yield, amount of screen reject, kappa number and black liquor properties were determined using TAPPI standard methods. After dewatering and fluffing, kappa number, percentage rejects, and total yield were determined.

Oxygen delignification : Oxygen delignification was carried out laboratory rotating digester at 95°C with 45 minutes reaction time of 1.0% Caustic addition and 12.0 kg Oxygen addition. The reactant pulp was washed well and the well fluffed pulp was used for bleaching studies.

Bleaching : The oxygen delignified pulps were bleached in the laboratory to achieve a brightness of 90% ISO using following bleaching sequence D0EPD1. Freshly prepared solutions of chlorine dioxide solution, sodium hydroxide and hydrogen peroxide was collected from plant. 250 g of Oven dried pulp added in polyethylene bags contained bleaching and extraction chemicals. The bags were thoroughly mixed by hand and placed in a temperature controlled water bath. After each of the bleaching stages, pulp was filtered with Buckner funnel to collect the spent bleaching liquor. The pH of the spent liquors was measured, and the liquors stored in plastic containers. Between bleaching stages, the pulp was washed with clean water, which was filtered from the pulp and discarded. The brightness of a sub sample of bleached pulp was measured according to the standard TAPPI Test Method (T 452).

D0 Stage: Then freshly collected ClO₂ was added into the polythene bags containing 10% consistency pulp and mixed well and noted the pH and put the pulp contained polythene bags in a preheated water bath at 75°C for 90 minutes with intermittent mixing of the pulps. End pH of the pulps were noted and the pulps were filtered, washed with hot water followed by DM water and thickened the pulps for preparing second bleaching stage. Kappa number and optical properties of the washed

pulps were determined using standard methods.

Ep Stage: The pulps from the D0 stage were adjusted to 10% consistency with DM water and 1.0 %NaOH addition was chosen to yield a terminal pH of 10.2-10.5. After noting down the pH, 1.0 % Hydrogen peroxide was added and noted the pulp mixture pH. Then (Ep) bleaching stage was performed in a preheated water bath at 75°C for 90 min. At the end of the (Ep) stage, the pulps pH were noted and filtered. The filtrates were collected, and the pulp was thoroughly washed with hot water followed by DM water and thickened pulp for final chlorine dioxide (D1) bleaching stage.

D1 stage: The pulps from the Ep stage were adjusted to 10% consistency with DM water and 0.8% chlorine dioxide charge was given to achieve a target Brightness (90.0 %ISO). After noting down the pulps pH, (D1) stage was performed in a preheated water bath at 75°C for 120 min. After noting down end pH, the pulps were filtered and the filtrates were collected. The pulp was thoroughly washed with hot water followed by DM water. The thickened pulps were stored in polythene bags for determination of its strength and optical properties.

Pulp characterization: At all stages brightness, kappa number and strength properties of the pulps were carried out as per TAPPI standard methods. Pulp brightness was measured with an optical Spectrometer by using white tiles as standard. For calculating tear, tensile and burst indices, the hand sheets were made as per ISO DP 5269 and dried on plates in standard conditions and conditioned at 27±10°C and 65±5% relative humidity and tested according to TAPPI standards. Hand sheet properties are reported on an oven dry basis.

Bleach filtrate analysis : At all stages, the filtrate were collected and tested for Colour, Total Dissolved solids (TDS) inorganic and Chemical Oxygen Demand (COD) as per standard testing methods.

RESULTS AND DISCUSION

Physical properties of wood chips: The physical properties of wood particularly basic density, bulk density and wood moisture are highly essential. The wood density of pulp wood is possibly one of the most influential factors controlling the strength and several other physical characteristics of the paper sheet. In the current study, the physical properties studied had exhibited variation in two and three years age gradation of *Melia dubia* K10 clone (Table:1). The high basic density (481 kg /m³) and bulk density (192 kg /m³) with lowest moisture content (10.0%) were observed in two year old species whereas the basic density (467 kg /m³) and bulk density (208 kg / m³) with moisture content (10.0%) was recorded in three year old wood sample. This result showed that bulk density of *M. dubia* K10 clone increases but basic density was slightly decreases with age while moisture content was same. Similar results were reported among various *Eucalyptus* species for basic

density which ranged between 425 kg m³ and 542 kg m³ (6). The wood density properties are of major importance for the production of quality pulp and paper. The amount of wood needed to produce one ton of air dried pulp is calculated from the density and pulp yield (7). Therefore the basic density recorded in the two and three year old *M. dubia* wood sample play a significant role in production of air dried pulp. Chips classification results revealed that both the wood sample of *M. dubia* yielded the accepted chips (+7 mm) for cooking which was around 85.00% and the dust generation was marginally low. This is the accepted size for pulping due to the optimal chips classification. The heat transfer and chemical penetration during pulping will be uniform in all both cases.

Chemical properties: The proximate chemical analysis gives an idea of potentiality of raw material for paper making (5). The chemical analysis in terms of ash content recorded was highest in two year old sample (0.72 %)

of *M. dubia* and lowest in three year old sample (0.64%) which implies that ash content decreases with the increase in the age of the *M. dubia* wood. Goel and Behl (8) recorded variation in ash content with relation to the age of the tree. High contents of ash will negatively impact the chemical recovery process and, therefore, could constitute a serious drawback (9). Similar results were also reported by several workers (10,11). Hence, the low ash content reported in two or three year old wood species of *M. dubia* indicated that it could be used for paper industries as it is best wood species for chemical recovery process.

In the present study, two year old *M. dubia* wood sample recorded lowest in alcohol benzene extractive, hot water solubility and 1 % NaOH as compared to three years old wood species contains slightly higher alcohol benzene extractive, hot water solubility and 1 % NaOH. The lower extractives will create lesser pitch problems and also proved more homogeneity in paper sheet (12).

Table: 1. *Melia dubia* K10 (2&3 years old) Physical and chemical properties

S. No.	Parameters	Unit	Melia dubia	
			2 years old	3 years old
1	Bulk density	kg/m ³	192	208
2	Basic Density	kg/m ³	481	467
3	Ash	%	0.72	0.64
4	Hot water solubility	%	2.64	2.72
5	1% NaOH solubility	%	12.0	12.8
6	Alcohol: Benzene solubility	%	1.12	1.20
7	Lignin	%	19.3	20.7
8	Pentosan (ash corretd)	%	13.2	13.8
9	Holocellulose (ash corrected)	%	74.2	75.6

Table: 2. Unbleached *Melia dubia* K10 2 &3 years old pulp Properties

S. No.	Particular	K10		K10	
1	White liquor addition,%	16	17	16	17
2	Screened pulp yield ,%	54.63	53.66	55.36	54.85
3	Reject, %	0.99	0.69	0.66	0.55
4	Kappa number	27.3	25.7	22.5	21.6
5	Pulp brightness, %ISO	33.1	33.7	33.6	33.9

Table: 3. Strength properties of 16 % and 17% TAA unbleached pulp

S. No.	Parameters	Unit	16% TAA		17%TAA	
			K10/2Yr	K10/3Yr	K10/2Yr	K10/3Yr
1	CSF	ml	540	520	525	505
2	Bulk	cc g ⁻¹	1.58	1.63	1.55	1.59
3	Tensile Index	N m g ⁻¹	52.2	52.6	49.5	50.1
4	Tear Index	M N m ² g ⁻¹	7.86	8.02	7.61	7.66
5	Burst Index	K pa m ² g ⁻¹	2.52	2.82	2.45	2.77

1% NaOH solubility, which measure low molecular weight carbohydrates, lower in two year old (12.0%) M. dubia sample compared to three year old sample (12.8 %). The holocellulose of content of two and three year old M.dubia K10 was found to be (74.2% & 75.6%) respectively. Based on holocellulose content of M. dubia K10 species is suitable for pulpwood from second year onwards. Similar results were observed in Pinus radiata at different age gradation (13) and in Anthocephalus cadamba at different heights of the tree (14). Low lignin content was reported in two year

old (19.3%) compared to three year old M.dubia species was (20.7%) which show that lignin content increase while age of tree increase. Low lignin content of a lignocellulosic material reduces pulping time and chemical charge (15). While higher contents of lignin are predicted to consume more chemicals (16). This result established that two year old trees of M. dubia K10 are also suitable for paper industry considering lignin content as a parameter.

Pulp yield and kappa number: The optimization of chemical requirement for any industry is a pre-requisite in order to

reduce the pollution hazards. The current investigation on optimization carried out with 20 kappa pulp using different chemical additions in M. dubia K10 at two and three years old. The unbleached pulp yield was 54.63 & 55.36% and its reject content was 0.99 & 0.66% for 16%AA. Whereas the yield was 53.66 & 54.85 % and its reject content was 0.69 & 0.55% for 17%AA respectively. The other study indicated that pulp yield of 45.1, 45.8, 46.85 and 49.34 per cent for two, three, four and twelve year's old Melia dubia wood which are lower than K10 species (17).

Table: 4. Melia dubia (K10 /2 years) Wood bleaching condition

S. No.	Parameters	16% TAA				17% TAA			
		ODL	D ₀	Ep	D ₁	ODL	D ₀	Ep	D ₁
1	Initial pH	8.5	8.1	3.8	8.0	8.4	8.1	4.0	8.1
2	Acid /Alkali addition,%	1.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0
3	pH	12.3	8.1	3.8	8.0	12.3	8.1	4.0	8.1
4	O ₂ /ClO ₂ /H ₂ O ₂ addition,%	1.2	1.4	1.0	0.4	12.0	1.4	1.0	0.4
5	pH	10.4	3.6	11.5	5.0	9.8	4.0	11.5	5.1
6	Brightness, %ISO	38.2	63.3	81.2	88.3	39.0	65.0	80.3	88.2
7	Kappa number	17.9	4.6	2.60	1.17	16.4	4.1	2.50	1.13
8	Kappa reduction ,%	34.4	69.3	43.5	55.0	36.2	68.9	39.0	54.80

Table: 5. Melia dubia (K10 /3 years) Wood bleaching condition

S. No.	Parameters	16% TAA				17% TAA			
		ODL	D ₀	Ep	D ₁	ODL	D ₀	Ep	D ₁
1	Initial pH	7.5	9.0	6.1	8.5	7.7	8.9	6.3	8.4
2	Acid /Alkali addition,%	1.0	0.0	1.0	0.0	1.0	0.0	1.0	0.0
3	pH	12.3	9.0	12.2	8.5	12.3	8.9	12.1	8.4
4	ClO ₂ /H ₂ O ₂ addition,%	12	1.2	1.0	0.4	12	1.2	1.0	0.4
5	pH	10.6	5.0	11.7	6.0	10.8	5.0	11.6	5.6
6	Brightness, %ISO	43.1	69.78	85.62	90.2	46.0	71.8	86.8	91.3
7	Kappa number	13.0	6.9	3.20	1.06	12.0	4.3	2.10	0.88
8	Kappa reduction ,%	39.6	47.7	53.6	66.9	44.4	58.65	51.16	58.10

Table: 6. Strength properties of 16% &17% TAA Melia dubia (K10 /2&3 years) bleached Pulp

S. No.	Parameters	unit	16 % TAA		17% TAA	
			D1 2Yrs	D1 3Yrs	D1 2Yrs	D1 3Yrs
1	CSF	ml	500	500	480	485
2	Bulk	cc g ⁻¹	1.59	1.66	1.57	1.64
3	Tensile Index	N m g ⁻¹	47.6	38.2	46.2	36.9
4	Tear Index	M N m ² g ⁻¹	9.22	8.88	9.24	8.67
5	Burst Index	K pa m ² g ⁻¹	3.02	2.66	2.96	2.44

Strength properties of unbleached pulp: The strength properties of paper in terms of tear, burst and tensile factors were very important for paper quality (18). The strength properties viz., tear, tensile and burst factors coupled with the brightness and opacity are the major indicators for pulp quality. In the current study, two year old M. dubia K10 pulp tensile index was (52.2 & 52.6 Nm g⁻¹), tear (7.86 & 8.02 mN m² g⁻¹) and burst index (2.52 & 2.82K Pa m² g⁻¹) for 16 & 17% TAA respectively. For three year old, tensile index was (49.5 & 50.1 Nm g⁻¹), tear (7.61 & 7.66 mN m² g⁻¹) and burst index

(2.45& 2.77 K Pa m² g⁻¹) for 16% & 17% TAA respectively (Table: 3). According to Guha (18), the tropical hardwood pulp which has breaking length greater than 6.0 km are very good in strength properties and can produce quality paper. So in the current study, two year old and three year old M. dubia K10 clones scores high and confirm that this clone can yield very good pulp strength properties. In the short period of time. Similar results were earlier reported in tensile and burst indices of paper obtained from one year old Leucaena leucocephala (19) which supports the findings of current result.

Bleaching properties of Melia dubia K10 pulp: Results presented in Table: 4, 5 & 6 shows that no major change in bleaching efficiency was noticed between the ages of Melia dubia K10. In spite of the fact that same (1.40%) ClO₂ was consumed in both wood pulps; the kappa number after extraction was more or less identical. In the present case, M dubia K10 pulp delignification efficiency in the D0 stage, expressed as kappa number reduction was 69.3 & 56.2 % for 16%TAA, as compared with 68.9 % & 64.2 % for 17%TAA.. As shown in Table: 4,5 & 6, there was no Brightness

Table: 7. Melia dubia (K10 / 2 years) filtrate properties

S. No.	Parameters	Unit	16% TAA				17%TAA			
			ODL	D ₀	Ep	D ₁	ODL	D ₀	Ep	D ₁
1	Colour	Pt. Co.,	4300	1020	520	60	4200	1000	500	60
2	Total dissolved solids	mg/l	3401	4042	2997	1004	3795	3331	2917	986
3	Suspended solids	mg/l	346	424	272	286	340	446	282	290
4	TDS Inorganic	mg/l	1920	2280	1898	482	1928	2092	1736	460
5	Total COD	mg/l	1992	1356	876	239	1713	1356	902	257
6	Total Hardness	mg/l	200	1080	80	120	170	1000	80	120
7	Calcium Hardness	mg/l	70	620	40	50	60	530	40	50
8	Magnesium Hardness	mg/l	130	460	40	70	110	470	40	70
9	Chlorides as Cl	mg/l	3672	472	199	284	352	489	178	263

Table: 8. Melia dubia (K10 / 3 years) filtrate properties

S. No.	Parameters	Unit	16% TAA				17%TAA			
			ODL	D ₀	Ep	D ₁	ODL	D ₀	Ep	D ₁
1	Colour	Pt. Co.,	5700	850	450	80	5250	820	430	80
2	Total dissolved solids	mg/l	3786	3036	3012	1022	3912	2820	3122	986
3	Suspended solids	mg/l	256	240	150	148	137	382	338	216
4	TDS Inorganic	mg/l	2158	2258	2170	486	2320	2126	2168	478
5	Total COD	mg/l	2412	717	319	323	2669	808	402	386
6	Total Hardness	mg/l	170	970	480	320	190	950	160	300
7	Calcium Hardness	mg/l	80	550	90	140	90	550	90	140
8	Magnesium Hardness	mg/l	90	420	90	180	100	400	70	160
9	Chlorides as Cl	mg/l	220	503	220	206	255	525	220	199

variation with respect to delignification efficiency, which is sensitive to operating conditions and perhaps also to wood species characteristics.

It is apparent that there is no difference in D0(Ep)delignification efficiency between 16% and 17% TAA pulps, or between the

two kappa numbers investigated in this study and also in the observation found in previous study. Extraction stage kappa reduction was (43.5 & 39.0%) for two years old pulp whereas (49.1% & 51.2% for three year old 16% & 17%TAA pulp with desired pH in Do stages suggests that the relative bleaching of Melia dubia K10

clone has ease of lignin removal of both unbleached kappa numbers. The extraction stage brightness was 81.2 & 80.3 %ISO for two years old and 85.6 & 86.8%ISO for three old Melia dubia K10 clones. This study indicates that this K10 clone can be bleached without any significant variation in bleaching response.

Chart : 1&2 . 16%&17% Na₂O Cooked K10 (2yrs) Bleached pulp properties

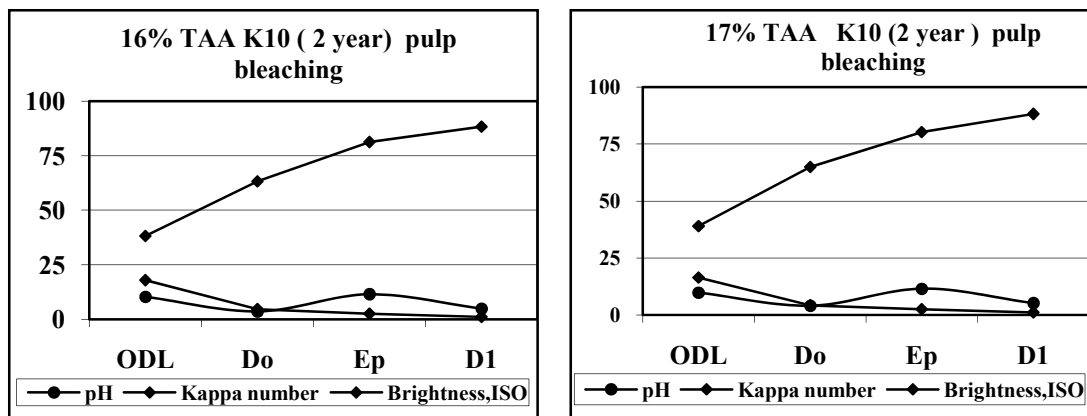


Chart : 3 & 4. 16% &17%Na₂O Cooked K10 (3yrs) Bleached pulp properties

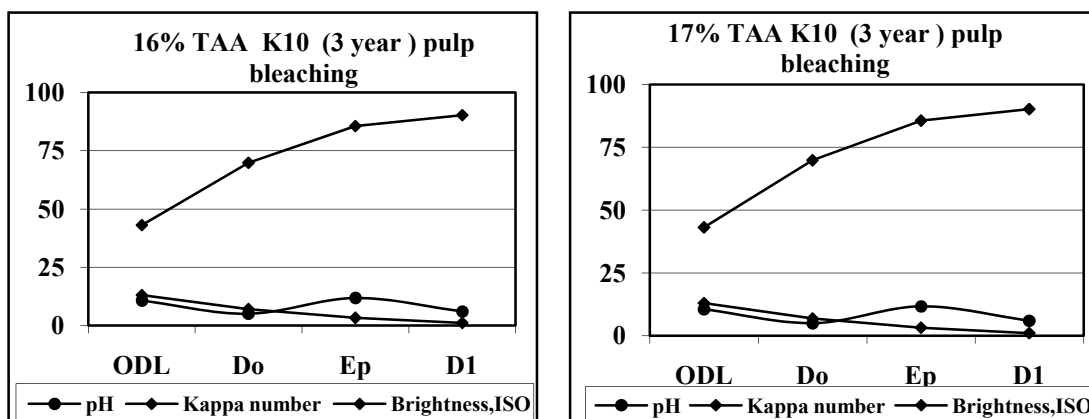


Chart : 5. 16%&17% Na₂O Cooked K10 (2year) Bleached pulp strength properties

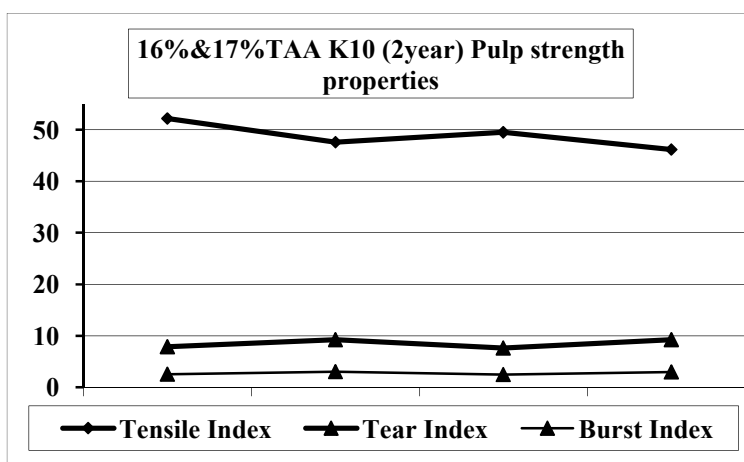
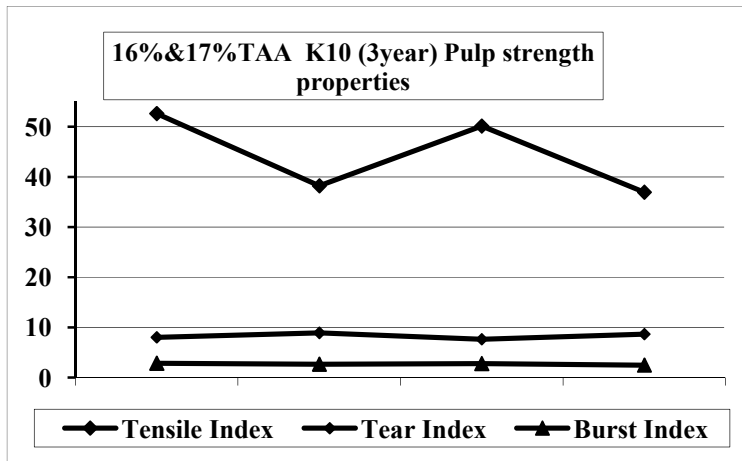


Chart :6. 16%&17% Na₂O Cooked K10 (3year) Bleached pulp strength properties



This Final chlorine dioxide (D1) stage kappa reduction was (55.0 & 54.8%) for two years old whereas (60.7% & 58.1% for three year old at 16% & 17% TAA charge respectively. The D1 stage brightness was 88.3% & 88.2%ISO for two years old and 90.2% & 91.3 %ISO for three old Melia dubia K10 species at 16% & 17% TAA charge respectively.

Bleach filtrate properties: Information on the mechanisms of delignification of different age gradation and their potential environmental effects was obtained by collecting and analysis filtrates from each of the bleaching stages. The resulting data are presented in Table: 8 & 9, the result shows that ODL stage filtrate Colour was (4300 & 4000 Pt.co.unit), Total Dissolved Solids was (3502 & 3790

mg/l) and COD was (1992 & 1713 mg/l), TDS inorganic, sulphate and chlorides was shown in Figure-3 & 4. These filtrate characteristics clearly indicates that Melia dubia K10 pulp bleaching with chlorine dioxide bleach filtrate have low Colour, TDS and COD. The lower TDS inorganic in washing filtrate with lower temperature is a major environmental benefit, because there is no commercially viable technology available to reduce TDS inorganic for large scale integrated pulp and paper mills

CONCLUSIONS

In present study pulping, bleaching and its filtrate indicates analysis indicate that Melia dubia K10 species of two and three years old is the most suitable species to manufacture paper making. This is achieved by terminating cook

at slightly higher kappa and bleaching without using any acid or alkali in chlorine dioxide bleaching at optimum temperature with OD0EpD1 sequence. The pulp yield, bleachability and bleached filtrate characteristics were superior when compared to the regular hardwood ECF plant pulps. The great economical and environmental benefit derived from the significant pulp yield even for two years old species makes Melia dubia K10 clone as a best short rotation pulpwood suitable for pulp and paper industry.

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Chart :7. 16%and 17% Na₂O Cooked K10 (2yrs) Bleach filtrate charecteristics

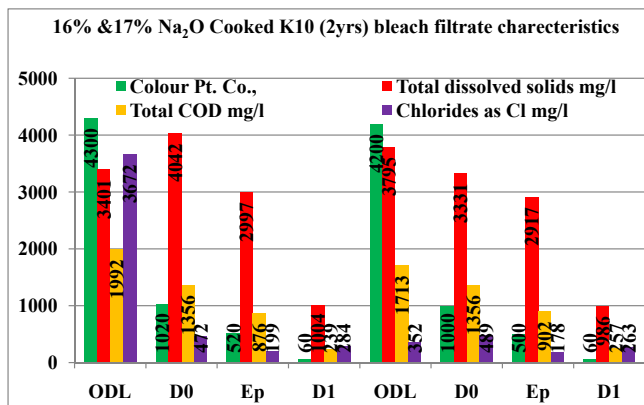
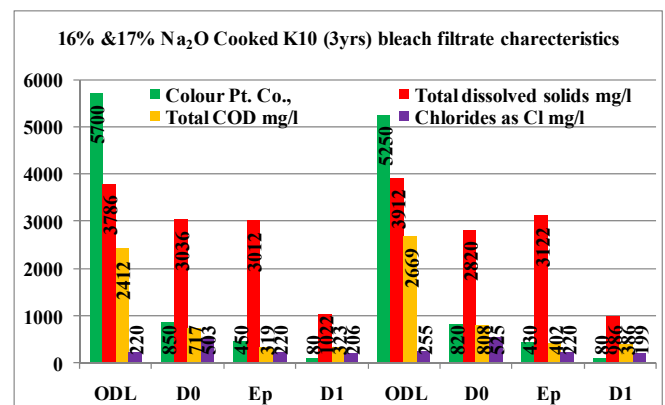


Chart :8. 16%and 17% Na₂O Cooked K10(3yrs) Bleach filtrate charecteristics



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