KRAFT PULPING CHARACTERISTICS OF THREE MOROCCAN EUCALYPTI. PART 1. PHYSICAL AND CHEMICAL PROPERTIES OF WOODS AND PULPS

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Two eucalyptus hybrids (EGC 39, EGC 241), resulting from crosses between *Eucalyptus grandis* and *Eucalyptus camaldulensis*, were investigated to see if they could produce kraft papermaking fibers with low lignin and adequate physical properties. The two hybrids were harvested at an age of 8 years along with 6-8 year old *Eucalyptus camaldulensis* (Rostrata). All three eucalypti were grown in the area of Gharb in the North-West of Morocco. The tracheids in the two hybrids had a very high Runkel ratio (2 x cell wall thickness/lumen diameter) and produced kraft paper sheets with low tensile strength due to a low degree of fiber collapse thus a low relative bonded area. These fibers could be used to increase the stiffness of a papermaking furnish. The lignin in the EGC 39 chips was more reactive in kraft pulping as compared to the other two eucalypti. Methoxyl analyses and nitrobenzene oxidation (NBO) of the in-situ lignin (wood meals) were performed, and it was concluded that the syringyl content of the EGC 39 lignin was less than or equal to those in the other two *eucalypti*. Differences in the guaiacyl fraction of the three samples will be discussed in Part 2 of this series.

Keywords: Eucalyptus; Kraft pulping; Fiber dimensions; Pulp strength; Lignin characterization

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INTRODUCTION

Eucalyptus, originally a genus native to Australia, has gradually been introduced to many parts of the world, notably South and Central America, Sub-Saharan Africa, the Mediterranean region, and Asia. There are several reasons behind the success of eucalypti as exotic species including; i) they are generally fast-growing, ii) generally tolerant to non-fertile soil, and iii) resistant to many arboreal diseases, thus making them easy to manage. The major drawback of eucalypti is their high moisture demand (average rainfall >600 mm/annum).

Morocco has about 10% of its total land covered with forest, and the Government has taken many wood plantation initiatives to increase the forest cover. Eucalyptus is the leading genus for plantation forestry programs in Morocco, and during 2002/2003 about 38% of the total area involved in plantation forestry (~209,400 ha) was covered with eucalypti (Menioui 2004). One of the intended uses of the wood was for the production of pulp for papermaking.

Morocco is far behind many countries in regards to forest coverage and it's approximately 10% value is well below the Global average of 20-25% (Hansen et al. 2010). Being inspired by recent advances in silvicultural practices and genetic engineering in forestry, the Moroccan Government started a tree improvement program in 1987 that was based on a 1983 road map document (DEFC-CDM-AFOCEL 1983). One of the objectives was to increase the country's production of eucalyptus wood. The first phase of the tree improvement program involved the selection of eucalyptus species with above average properties such as growth rate and modulus. The selected species or their natural hybrids were then to be multiplied by vegetative or clonal propagation (DEFC-CDM-AFOCEL 1983).

The main objective of this research was to study the fibre morphology, chemical composition, and the papermaking potential of two of the fast-growing eucalyptus clones, EGC39 and EGC241, that resulted from hybridization of *E. grandis* and *E. camaldulensis*. The results from these two clones will be compared to those from *E. camaldulensis* (Rostrata), which is a well established species in Morocco.

MATERIALS AND METHODS

Wood supply

The two hybrids chosen for this study were cultivated on a plantation in the area of Gharb in the North-West of Morocco and were harvested after 8 years of growth. Five logs were harvested for each clone. *E. camaldulensis* logs were also harvested in Gharb and were 6 to 8 years of age. The logs were debarked, chipped, and screened before being air-dried. The chips were then stored in sealed polyethylene bags for morphological, chemical characterization and pulping experiments. The bark content of the two hybrids was determined by mass of bark and mass of bolewood (oven-dried or OD basis) taken from each of the five logs. Only one bolt was used for *E. camaldulensis.*

Morphological characteristics

Fiber separation was achieved by treatment of mini-chips in a 50/50 mixture of 30% hydrogen peroxide and acetic acid at 40°C. Tracheids were analyzed for fiber length, fiber width, cell wall thickness, and lumen diameter by light microscopy at a magnification of 400. Morphological indices such as Runkel and aspect ratios were calculated from the data.

Chemical wood properties

Wood chips were converted to 40 mesh wood meal with the aid of a Wiley mill. The wood meal was ethanol-toluene extracted according to TAPPI method T 204 om-88. Other methods that were used include: TAPPI method T 207 om-88 for hot water solubility; TAPPI method T 211 om-85 for ash content; TAPPI Method T 222 om-88 for Klason lignin; and TAPPI method UM 250 for acid-soluble lignin (ASL).

Kraft and soda-AQ pulping

Kraft cooking was performed in a 20 L electrically heated rotary digester. The chips were impregnated with white liquor for 20 min at 40°C before the temperature was increased to a maximum of 165^{o} C over a period of 120 min (H-factor = 117). The active alkali varied from 17.0% to 20.0% Na_2O on chips, while the sulfidity varied from 30% to 32%. The liquor to wood ratio was 4:1 and the time at 165^{0} C was either 40 min or 45 min. Soda-AQ or SAQ pulping of solvent extracted 15 mesh wood meal in the presence of ethylguaiacol (SAQ + EG) was previously described by Kanungo et al. (2011).

Pulp Analyses

Kappa number was determined by ISO Standard 302 and brightness according to ISO Standard 3688. The pulp was beaten in a JOKRO Mill (ISO 5264-3), and the drainability of unbeaten and beaten pulps were measured by the Schopper-Riegler method following ISO Standard 5267. Handsheets were made from both beaten and unbeaten pulps according to ISO standard 5269-2, conditioned (ISO 187), and then tested for tensile index (ISO 1924) and tear index (ISO 1974).

Other analyses

Nitrobenzene oxidation was by the procedures described by Chen (1992) with a 0.694 correction factor applied to the S:G ratio obtained (Bose et al. 2009a; Masingale et al. 2009). Carbohydrate composition was by sulphuric acid hydrolysis followed by monomer quantification by ^{1}H NMR (Bose et al. 2009b; Alves et al. 2010). Methoxyl content analyses were by Galbraith Laboratories, Knoxville, Tennessee, and the hydriodic acid or Zeisel method was used.

RESULTS AND DISCUSSION

Tracheid/Fiber Properties

The average dimensions for tracheids from the three eucalypti are presented in Table 1. The most interesting of the tracheid (fiber) dimensions were those resulting in the high Runkel ratio for the two hybrids. The Runkel ratio is known to be quite variable amongst eucalypti. Dutt and Tyagi (2011) recently reported values for eleven eucalypti. While the value for two E. *grandis* were 0.40 and 0.52, seven of the remaining nine samples had values >1.0. Those authors reported a value of 1.11 for their E. *camaldulensis*, while a value of 0.84 was obtained in this study. The very high Runkel ratios of 1.33 and 1.37 for EGC 39 and EGC 241 would suggest that fibers from their kraft pulps would be unable to produce paper sheets with a high tensile index. Fiber collapse as measured by sheet density is known to decrease as the Runkel ratio increases (Gurnagul et al. 1990). When other fiber parameters are normalized, relative bonded area is known to decrease as sheet density decreases (Hieta et al. 1990). However, these fibers have low values for their diameter and as such should produce pulp fibers with a low coarseness (mg/m). Fibers with low coarseness and high Runkel ratio can be blended with flexible fibers to give sheets with a superior combination of smoothness, strength and stiffness. In one study, birch kraft fibers were replaced by kraft fibers from a eucalyptus in the preparation of three-ply sheets. The result was lower roughness and a higher bending stiffness (Haggblom-Ahnger 1999). The eucalyptus had a lower fiber diameter (16 μm vs. 22 μm) and a high Runkel ratio (0.375 vs. 0.273) as compared to the birch (Haggblom-Ahnger 1999).

Table 1. Fiber Characteristics and Morphological Indices of *E. camaldulensis, EGC39 and EGC 241*

	Eucalyptus clones		*Eucalyptus Camaldulensis*
	EGC 39	EGC 241	
Fiber Dimensions			
Fiber length, mm (L)	0.81	0.72	0.70
Fiber width, μm (*w*)	11.9	12.8	10.1
Wall thickness, μm (c)	3.4	3.7	2.3
Lumen width, μm (l)	5.1	5.4	5.5
Morphological indices [1]			
Flexibility coefficient, *100(l/w)*	43	42	54
Cell rigidity, *2c/w*	0.57	0.58	0.46
Aspect Ratio, *L/w*	68	56	69
Runkel ratio, *2c/l*	1.33	1.37	0.84

[1] According to Dutt and Tyagi (2011)

Bark Content and Wood Chemical Properties

The results are summarized in Table 2. The first issue to be addressed is bark content, since it can be quite high (12 to 20 wt%) for young bolewood from *E. camaldulensis* (Sesbou 1990; Ranasinghe and Mayhead 1991; Khristova et al. 2006). It can be seen that all three of the present eucalypti had bark content in the 12% range (Table 2). *E. camaldulensis* had a much higher content of ethanol-toluene extractives, as compared to the two hybrids. However, the value of 6.6% is not atypical for young *E. camaldulensis* (Ona et al. 1997; Dutt and Tyagi 2011).

The glucan and lignin content obtained for *E. camaldulensis* (45.8% and 30.6%) were both very close to values of 45.0% cellulose and 31.3% lignin reported by Sjöstrom (1993). The two hybrids contained slightly more xylan and slightly less lignin as compared to *E. camaldulensis* (Table 2). The summative analyses for the extractive-free wood meals were in the range of 99.1% to 100.4%. A 0.2 mole ratio of 4-O-methylglucuronic acid (Me-GluU) to xylose was assumed based on the recent results of Magaton et al. (2011). Similarly a 0.10 mole ratio of other uronics (galacturonic and glucuronic) to xylose was assumed based on the recent results of Alves et al. (2010).

Kappa Number and Pulp Yield

Preliminary cooking trials were conducted on all three eucalypti using 16% Na_2O on chips and ca. 30% sulfidity. It was observed that a kappa number lower than 20 could be obtained for EGC 39 but not for the other two samples. Actually, a pulp with low rejects (<5%) was not possible using only 16% Na_2O on *E. camaldulensis.* It was decided to compare the two hybrids using 17% and 18% Na_2O. Unfortunately, higher alkalinities were required to achieve pulps with kappa numbers in the range 14 to 20 from *E. camaldulensis.* The results with ≥17% Na_2O are presented in Table 3, and it can be seen that the EGC 39 hybrid achieved a significantly lower kappa number than the other two eucalypti. The higher alkali requirement for *E. camaldulensis* chips to achieve kappa number <19 is probably due to its higher extractives content. There were no significant differences in the fiber yield amongst the three eucalypti kraft pulps.

Table 2. Bark Content and Chemical Compositions of the Three Eucalypti

	E. *camaldulensis*	EGC 39	EGC 241
Whole Wood			
Bark, %	12.0 ± 2.0	10.5	11.2
Un-extracted Debarked Wood			
Hot Water Extractives, %	6.7	7.6	8.6
Ethanol-Toluene Extractives, %	6.6	2.0	3.5
Extracted Debarked Wood[1]			
Klason Lignin, %	27.2	25.0	25.7
Acid Soluble Lignin, %	3.4	2.8	3.2
Glucan, %	45.8	44.5	44.6
Xylan, %	11.8	12.6	12.3
Arabinan, %	1.4	2.1	1.7
Mannan, %	1.9	3.0	2.1
Galactan, %	<1.0[2]	<1.0	<1.0
Acetyl, %	2.4	2.8	2.6
Me-GluU, %[3]	3.4	3.6	3.5
Other Uronics, %[4]	1.6	1.7	1.7
Ash, %	0.5	0.5	0.7
Total, %	100.4	99.6	99.1

[1] Ethanol-toluene extracted; results on extractives-free basis and all sugars as anhydrides
[2] Counted as 1.0% in the summation
[3] 0.2 mole ratio of Me-GluU to xylose (Magaton et al. 2011)
[4] 0.1 mole ratio of other uronics (galacturonic and glucuronic) to xylose (Alves et al. 2010)

The higher reactivity of the EGC 39 lignin in alkaline cooking was confirmed by SAQ + EG treatment of the wood meals. After the standard treatment (H-factor 441), the kappa numbers were 37.9, 31.7 and 38.6 for *E. camaldulensis*, EGC 39 and EGC 241, respectively. The SAQ + EG treatment condition is part of an analytical procedure that can be used to estimate the concentration of uncondensed guaiacyl units in native hardwood lignins (Kanungo et al. 2011). The method will be applied to these three eucalypti in a companion paper in this Journal (Alves et al. 2012).

Table 3. Kraft Pulping Results

	E. *camaldulensis*		EGC 39		EGC 241	
Active Alkali, %Na_2O[1]	19	20	17	18	17	18
Time at 165°, min	45	45	40	40	40	40
H-factor	575	575	524	524	524	524
Total Yield, %[1]	46.5	45.9	45.8	44.2	46.0	44.8
Screened Yield, %[1]	45.7	45.5	45.0	44.2	45.0	44.5
Kappa Number	*18.8*	16.5	*15.0*	14.5	*19.0*	17.0
ISO Brightness, %	-	-	33.5	35.0	28.2	29.0

[1] % on chips

Physical Properties of Pulps

Extensive research was not performed in this area because low tensile strength was expected from the fibers of both hybrids (previously discussed). In a previous study, it was observed that strong correlations could be developed between tensile index and Runkel ratio as well tear index versus fiber length (Gurnagul et al. 1990). These correlations were observed for fibers from six hardwood kraft pulps both unrefined and refined to 11,000 PFI revolutions at light load. This level of refining afforded tear strength that was close to the maximum value for all six species (Gurnagul et al. 1990). The results for EGC 39 and EGC 241 are presented in Table 4. The results are for unrefined fibers and fibers refined to the point of maximum tear index. The only significant difference between the two pulps is the higher tear strength for the refined EGC 39 fibers, and this was expected based on their higher average length (Table 1). Also, the tensile index at maximum tear index was in the range of 60 to 65 N•m/g for both pulps. These values are very low and are due to the high Runkel ratios of the fibers as previously discussed. Typically, at maximum tear index, tensile index is in the range of 90 to 100 N•m/g (Gurnagul et al. 1990). Strength data were not obtained for the *E. camaldulensis* but a significantly higher tensile index would be likely. Strength properties from 6 to 8 year old *E. camaldulensis* grown in Sudan are reported by Khristova et al. (2006). At a drainability of only 30 °SR, the apparent sheet density was 0.74 g/cm^3, tensile index was 74.4 N•m/g, and tear index was 8.1 mN•m^2/g. The higher apparent sheet density and tensile index would be indicative of a higher degree of fiber collapse as compared to the two hybrids involved in this investigation.

Table 4. Physical and Mechanical Properties of Handsheets

Pulp	°SR	Apparent Density (g/ cm^3)	Tensile index (N•m/g)	Tear index (mN•m^2/g)
EGC 39	16.5[1]	0.44	~10	2.2
	50.0[2]	0.65	61.6	8.6
EGC 241	16.0[1]	0.44	~10	3.9
	35.0[2]	0.69	63.2	7.5

[1] Unrefined fibers
[2] Refined to a tear index close to the maximum

Comparison of Native Lignin Structures

The high reactivity of the EGC 39 lignin was quite encouraging, and both the kraft and SAQ + EG data suggest that a low kappa number pulp can be produced from EGC 39 at a low H-factor. When the temperate hardwood, sugar maple (*Acer saccharum*) was given the standard SAQ + EG treatment it afforded fibers with kappa number 41.9 (Kanungo et al. 2011) as compared to 31.7 for EGC 39. It should be noted that the extracted sugar maple had a lignin content of 24.8% (Kanungo et al. 2011), while the value for EGC 39 was 27.8% (Table 2). An investigation was initiated to see whether the EGC 39 lignin contained a higher syringyl content than the other two eucalypti, since these units are more reactive than guaiacyl units in both kraft and SAQ delignification (Tsutsumi et al. 1995; Kondo et al. 1987). The nitrobenzene oxidation (NBO) method was used to estimate syringyl to guaiacyl (S:G) ratio, but there was some doubt about the initial results. The NBO data suggested that EGC 39 had a lower S:G ratio than the other two eucalypti. The monomers quantified in NBO are shown in Fig. 1, and an unusually low yield of G2 (vanillic acid) was obtained from *E. camaldulensis* and EGC 241. In a previous investigation, 13 poplars were analyzed by NBO, and five of them had S:G ratio ≥1.45 (Bose et al. 2009a). The S:G ratio along with the vanillic acid yield are documented in Table 5 for those five poplars and the three eucalypti involved in the present investigation. It can be seen that the lowest yield of G2 for any of the poplars was 1.2 mmoles/100 mmoles of C_9 units (1.2%) while the yield was 0.7% for EGC 39 but only 0.3% for *E. camaldulensis* and EGC 241. For whatever reason, if the guaiacyl fraction of the lignin in the eucalypti was un-reactive towards NBO, the outcome would be an artificially high S:G ratio. Instead of repeating NBO it was decided to send out the wood meal samples for methoxyl analyses. Based on the summative analyses in Table 2, it was anticipated that high methoxyl values would be obtained, and ca. 85% of the methoxyls would be lignin-derived.

The derivation of S:G ratios from the methoxyl content data is summarized in Table 6. A key assumption is that 0.6 wt% of OCH_3 groups was derived from non-methoxyl sources or from methoxyl sources other than those in lignin and Me-GluU. This deduction was made from literature data. Lebo (1988) reported a lignin content of 26.9% and methoxyl content of 5.07% (Zeisel method) for a white spruce (*Picea glauca*) refiner mechanical pulp. If a C_9 molecular weight of 190 and 96 OCH_3/100 C_9 are assumed for spruce lignin then a methoxyl content of 4.2% would be expected from the lignin. If the 7.0% xylan content and 5:1 xylose to Me-GluU ratio reported for *Picea glauca* (Fengel and Wegener 1989) are used, then 0.3% methoxyl should have been derived from Me-GluU. The difference in Lebo's methoxyl data of ca. 0.6% (5.07 – 4.2 – 0.3) is assumed to have come from other sources.

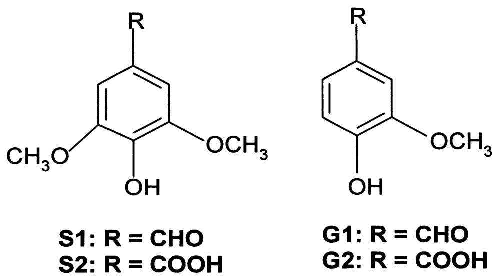

Fig. 1. Products from nitrobenzene oxidation of hardwood lignin

The lowest value for lignin-derived methoxyls as a percent of total methoxyls amongst the three eucalypti was 85% (Table 6). The S:G ratios that were derived all appeared reasonable and in two cases very close to the S:G ratio by NBO. The syringyl fraction of the lignin by methoxyl analyses was 66.0%, 62.0%, and 62.0% respectively for *E. camaldulensis*, EGC 39 and EGC 241, respectively. The corresponding values by NBO were 65.0%, 62.0%, and 63.5%. It should be noted that methoxyl analysis of extracted wood meal appears to give an accurate estimate of the syringyl content of the lignin only when the lignin content and S:G ratios are high and the xylan (Me-GluU) content is low. In the present case, the lowest lignin content was 27.8% while the highest xylan content was 12.6% (Table 2). Based on results obtained in this investigation (not reported), the method gave unreliable S:G ratios for poplars with lignin content <22% and xylan content >17%.

Table 5. Vanillic Acid Yields Associated with S:G Ratio of Poplars and Eucalypti by Nitrobenzene Oxidation (NBO)

	S:G Ratio	Yield of Vanillic Acid[2]
Poplars[1]		
12XAA9005	1.50	1.2
220-5	1.57	1.7
Crandon	1.45	1.5
D 105	1.68	1.5
7300501	1.47	2.7
Eucalypti		
E. camaldulensis	1.98	0.3
EGC 39	1.72	0.7
EGC 241	1.85	0.3

[1] Please refer to Bose et al. (2009b) for further details
[2] mmoles/100 mmoles of C_9 units

The differences in reactivity amongst the three lignins appear not to be due to differences in the amount of reactive syringyl units they contained, because the % S units for EGC 39 was equal or less than the value for the other two eucalypti. It will be shown in Part 2 of this series that the EGC 39 lignin contained a higher concentration of β-O-4 dimeric units, where the ring capable of forming a quinone methide (QM) was an uncondensed G unit. Syringyl units capable of forming QM, few of which are condensed, are known to be quite reactive in both kraft and SAQ cooking (Tsutsumi et al. 1995; Kondo et al. 1987). Uncondensed G units are also quite reactive, while condensed G units are known to be un-reactive (Gellerstedt et al. 1988; Bose et al. 2009c). An uncondensed C_9 unit is defined as one not containing a C-C bond at any ring position except for C-1 (sidechain) nor connected to another C_9 unit by a diaryl ether linkage.

Table 6. S/G Ratios from Methoxyl Contents of Eucalypti Wood Meals

	E. Camaldulensis	EGC 39	EGC 241
Lignin Content, %	30.6	27.8	28.9
C_9 units (mmoles/100g wood meal)[1]	0.144	0.131	0.136
OCH_3, wt% on wood meal	8.46	7.68	7.91
OCH_3 from Me-GluU,%[2]	0.55	0.59	0.58
OCH_3 from other Sources, %[3]	0.6	0.6	0.6
OCH_3, Lignin Derived, %	7.31	6.49	6.73
OCH_3, Lignin Derived, mmoles	0.236	0.209	0.217
OCH_3/Lignin Mole Ratio	1.64	1.60	1.60
% S Units[4]	66.0 (65.0)[5]	62 (62.0)	62 (63.5)
S:G Ratio from OCH_3 Content	2.06[4] (1.98)[6]	1.72 (1.72)	1.72 (1.85)

[1] C_9 molecular wt. of 212 is assumed (Masingale et al. 2009)
[2] Me-GluU to xylose mole ratio of 0.2 (Magaton et al. 2011)
[3] Other than lignin and Me-GluU, see text
[4] Assume S and G units comprise 98% of C_9 units
[5] % S units from S:G ratio by NBO [6] S:G by NBO

CONCLUSIONS

Two eucalyptus hybrids (EGC 39, EGC 241), resulting from crosses between *Eucalyptus grandis* and *Eucalyptus camaldulensis*, were investigated to see if they could produce kraft papermaking fibers with low lignin and adequate physical properties. *Eucalyptus camaldulensis* (Rostrata) was used as a reference species, and all three eucalypti were grown in the area of Gharb in the North-West of Morocco. The logs that were used were harvested after 6 to 8 years of growth.

The two hybrids produced kraft pulp with low tensile strength, and this was due to the high Runkel ratio of their tracheids (1.33 and 1.37). The low coarseness of the EGC 39 and EGC 241 fibers (as inferred from their small width) coupled with their low collapsibility would provide some advantages if they were to be blended with some flexible fibers in a papermaking furnish. Such a furnish would likely have a superior combination of smoothness, strength, and stiffness. One of the hybrids (EGC 39) was much more reactive in kraft and SAQ pulping as compared to the other two eucalypti. Methoxyl analyses and nitrobenzene oxidation (NBO) of the in-situ lignin (solvent extracted wood meals) were performed, and it was concluded that the syringyl content of the EGC 39 lignin was less than or equal to those of the other two eucalypti. Difference(s) in the guaiacyl fraction of the three samples will be discussed in Part 2 of this series.

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