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A Comparison of Soda and Soda-ethanol Pulps of *Thaumatococcus daniellii* Benth (Miraculous Berry) Stalks

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Authors' contributions

This research study is a concerted effort of all the Authors involved. Author AOO supervised the work, played active role in the bleaching studies, handsheet preparation and strength properties testing with the active involvement of author AFG. Author OAS carried out the pulping experiment, statistical analysis and preparation of the manuscript. All authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

Aims: To evaluate the pulp and papermaking characteristics of *Thaumatococcus daniellii* stalks using soda and soda-ethanol pulping methods.
Study Design: A completely randomized design was used.
Place and Duration of Study: Department of Chemical Engineering, Lakehead University, 955 Oliver Road, Thunder Bay, Canada, between June 2010 and July 2011.
Methodology: The stalks of *T. daniellii* were pulped with both sodium hydroxide (NaOH) and soda-ethanol ($NaOH\text{-}C_2H_5OH$) liquors in a 10-litre electrically heated stainless steel digester. Cooking temperature was varied at 140, 150 and 160 ^{O}C while the concentration of the cooking liquor was kept constant at 12% NaOH and 12:40% by volume of $NaOH\text{-}C_2H_5OH$ and cooked for 60 minutes. The bleachability of the pulp was assessed using DEDD elemental chlorine free bleaching sequence. The bleached pulp was beaten in a PFI mill at revolution of 0, 1600 and 3200 followed by formation of handsheet in a

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standard sheet former. Lastly, the elrepho brightness, bulk, apparent sheet density, tear index, burst index, tensile index and the breaking length of the handsheets produced from the two pulp types were evaluated using standard tappi methods.

Results: The temperature and pulping method had marked effects on the yield, kappa number and brightness of the pulps. Also, beating revolutions had significant effect on the physical and strength properties of the handsheets produced from both pulps ($P = 0.05$). The yield and bleaching properties of soda-ethanol pulps are superior to those of soda pulps. Handsheets produced from soda-ethanol pulps also had significantly higher strength, particularly in unbeaten and when beaten at 1600 revolution ($P = 0.05$). Beyond 1600 revolution, the tear index, tensile index and breaking length of soda-ethanol handsheet were not significantly higher than those from soda pulp ($P \geq 0.05$).

Conclusion: Handsheets produced from *T. daniellii* stalks had physical and strength characteristics that are comparable to those from notable non-wood fibre source. It is therefore expected that the stalk can play a big role in complementing wood fibres in pulp and papermaking.

Keywords: Alkaline-ethanol pulp; DEDD bleaching; pulpsheet properties; T. daniellii stalk; non-wood fibre source.

1. INTRODUCTION

The role of pulp and paper products in the political, educational and socio-economic development of a nation cannot be over emphasized. The per capita growth curve of paper consumption has been identified to correlate with gross national products, gross domestic product and growth in literacy level [1]. Globally, population growth and economic development is undoubtedly increasing the demand for various forest products particularly pulp and paper products. Nevertheless, the demand for wood, which is the principal raw material for papermaking, has been projected to witness a downward trend due to the adverse effect of the large scale harvesting of wood on the environment, high cost of wood and pending environmental legislation imposing new regulations on the disposal of agricultural waste fibres [2]. This notwithstanding, the global demand for pulp and paper products has been estimated to increase to over 490 million tonnes per year by year 2020 [3]. These will more than double the demand for cellulosic fibre for paper and paperboard production. The major problem now is how to meet the continuous increase in the demand for various paper products. It was suggested that the bulk of the future fibre requirements would be sourced from recovered paper [4]. Though this might be logical, it has been established that recycling fibre weakens it, each time it is re-processed thereby placing limitations on paper grades over time [5]. To bridge the extensive gap between paper demand and cellulosic raw material supply, fast-growing non-wood plant of higher biomass has to be screened for their suitability for pulp production. In line with this, recent researches has been directed toward exploring the potentials of non-wood fibres from herbaceous field crops as alternative for pulp and paper production [6,7,8,9,10,11]. The use of non-wood fibre is advantageous based on its virgin fibre source, short gestation period and provision of excellent quality for making speciality grades of paper. Also, it is an ethically sound way of promoting sustainable paper production especially in areas lacking long fibre pulpwoods but having abundant annual fibre resources. Among the renewable and hitherto unexploited source of cellulosic fibre for papermaking, the stalk of *T. daniellii* (Miraculous berry) stands prominent.

T. daniellii (miraculous berry) is a perennial, rhizomatous and monocotyledonous herb which grows through its rhizomes up to 3m in height. It is a member of the Maranthaceae family with *Megaphrynium macrostachyum* (K. Schum) [12]. It is indigenous to the rainforest of West Africa, particularly the southern parts of Ghana, Cote d'Ivoire and Nigeria [13]. It is also known to exist in the Princes Islands, Angola, Central African Republic, Uganda, Brazil, Venezuela and Indonesia. The plant offers several advantages including high annual yield per hectare, self regeneration after harvest through its rhizomes, moderate fertilization requirement [14], ease of harvesting and transportation [15]. The main economic use of the plant is the extraction of thaumatin from its fruit as a sweetener [16] and the use of its leaves for wrapping [17].

Recent researches have been focused on the use of *T. daniellii* stalk as a non-wood fibre source. Oluwadare and Sotannde introduced this plant as a non-wood fibre source for pulp and papermaking [18,19,20]. Its fibres are comparable to those of major non-woods used in papermaking [19] with good pulping properties from different ecological zones [21]. However, no information has been provided on the physical and strength properties of paper produced from its stalk. It is on this background that this study investigated and compared the pulp yield, bleachability and strength properties of handsheets produced from its soda and soda-ethanol pulps.

2. MATERIALS AND METHODS

2.1 Pulping Experiment

The stalks of *T. daniellii* used for this study were harvested as undergrowth in a cocoa plantation in the South-Western part of Nigeria. The stalks were cut into short length of about 5 cm and air-dried to constant moisture content. The air-dried stalks were pulped with both soda and soda-ethanol solutions. The soda cooking liquor was prepared from a standard concentrated solution of Sodium hydroxide by dilution with de-ionized water to 12%. Ethanol solution was prepared by diluting 40ml of 99% ethanol with 60ml de-ionized water in 100ml measuring cylinder. This gives 40% ethanol and 60% water. The soda-ethanol liquor was then prepared by mixing equal volume of 12% sodium hydroxide solution with 40%/60% ethanol-water solution. The stalks were pulped in a 10-litre electrically heated stainless steel digester. The stalks were weighed and charged into the digester with the required amount of chemical at liquor to solid ratio of 4:1. The digester was heated to the operating temperatures of 140, 150 and 160ºC and cooking time of 60 minutes. A total of 18 cooks were carried out and at the end of each cooking cycle, the pulp produced was thoroughly washed in cold water and screened using a laboratory flat screen with 0.25 mm slots. The screened yield was then determined gravimetrically after air-drying in the laboratory environment for 72 hours while the kappa number of the screened pulps were determined in accordance with Tappi T 236 method.

2.2 Pulp Bleaching

Based on screen yields and their corresponding kappa number, only pulp obtained at 150 ^{O}C cooking temperature was used for bleaching and refining experiments. The pulps were bleached using three-stage chlorine dioxide and alkaline (D_0 E_1 D_1 D_2) bleaching sequence. Where D is chlorine dioxide and E is alkaline extraction. The bleaching stages were carried out in polyethylene bags in a water bath for 1.30 hours. In the first stage, the pulp was chlorinated with 8% ClO_2 for 1 hour. This was followed by extraction with 2% caustic soda and subsequent chlorination with 1 and 0.7% chlorine dioxide, respectively. At the end of the bleaching sequence, the brightness of the pulp was evaluated using Photo electric Reflectance photometer (ELREPHO).

2.3 Refining and Handsheet Preparation

Handsheets were prepared from the pulps using British sheet former in accordance with the Tappi standard T205 sp-95, on 60 g/m^2 basis weight. The laboratory beating of the pulps was carried out using Laboratory PFI mill according to Tappi T 248 sp-00. The beating was varied from 0 – 3200 revolutions. The beaten pulp was transferred into sheet former and handsheet formed based on 0.3% pulp consistency. Finally, the sheets formed were dried and conditioned at 25ºC and 50% relative humidity (RH) for 48 hours prior to testing. Based on Tappi methods, the bulk (cm^3/g), apparent sheet density (g/cm^3), tear index (mN.m^2/g), burst index (kPa.m^2/g), tensile index (Nm/g) and breaking length (Km) of the handsheets were evaluated based on Tappi standard methods.

2.4 Statistical Analysis

The means of the screened yield, kappa number and brightness of the handsheets were computed. The influence of PFI revolutions on the strength properties of the handsheets was evaluated by subjecting the data collected to one-way Analysis of Variance test. The significance of the difference between the means was tested using Duncan Multiple Range Test at 0.05 probability level. T – Test was used to express the magnitude of the difference between the strength properties of soda and soda-ethanol pulps at varying PFI revolutions.

3. RESULTS AND DISCUSSION

3.1 Pulp Yield and Kappa Number

The result of the pulp yields and Kappa number is presented in Table 1. The influence of cooking temperature was very pronounced on the pulp yield. About half of the original *T. daniellii* material was dissolved after pulping with 12% soda at 140ºC for 60 minutes. Higher pulp yield and higher Kappa number were obtained at the lower temperature of 140ºC, while lower pulp yield and lower Kappa number were obtained at elevated temperatures. This implies that, high rate of lignin-carbohydrate complexes dissolutions [22] and solubilisation of significant amount of hemicelluloses [10] occurs at elevated temperatures. Hence, during pulping process, both lignin and cellulose are dissolved at different rates. This rate is accelerated by increasing the temperature of the pulping medium [23]. The decrease in pulp yield and kappa number became gradual afterwards as evidenced in 14.37% and 13.45% reduction in pulp yield and, 12% and 1.35% in kappa number at elevated temperatures of 150ºC and 160ºC, respectively.

Meanwhile, when soda method was modified with ethanol, the pulp yields increased while the kappa numbers of the resulting pulps decreased. The addition of 40% ethanol to 12% soda pulping liquor at 140ºC resulted in 18.12% increase in average pulp yield and reduction in kappa number by 9.43%. The trend continued at elevated temperatures of 150 and 160ºC. This showed that adding ethanol to the conventional soda process was effective for improving both delignification [24,25] and the screened yield of *T. daniellii* pulps. Thus, delignification proceeded more rapidly and more selectively with alkali-ethanol than with alkali alone, giving higher yields and lower kappa numbers. Similar trend was reported on the addition of organic solvent to soda pulping mixture of *Arundo donax* [26], Nigerian grown *Bambusa vulgaris* [8] and cotton stalks [27]. A higher pulp yield with soda-ethanol, compared to the soda method, could be attributed to higher hemicellulose retention [24,25]. Meanwhile, beyond 150ºC, marginal decrease in the yield and kappa number of both soda and soda-ethanol pulps were obtained. Thus it might not be economical to pulp *T. daniellii* stalks beyond 150ºC. Hence 150ºC was selected as its optimum cooking temperature. Therefore, screened pulp produced at 150ºC was used for bleaching and refining experiments.

Table 1. Influence of pulping conditions on the yield and kappa number of *T. daniellii* pulp

Cook Number	Soda (%)	Ethanol (%)	Cooking temperature (ºC)	Cooking time (Minutes)	Screened yield (%).	Kappa Number
1	12	-	140	60	44.75	-
2	12	-	140	60	47.95(46.63)*	35.0
3	12	-	140	60	47.18	-
4	12	-	150	60	37.53	-
5	12	-	150	60	41.52(39.93)	30.8
6	12	-	150	60	40.74	-
7	12	-	160	60	33.93	-
8	12	-	160	60	36.02(34.56)	30.2
9	12	-	160	60	33.73	-
10	12	40	140	60	53.09	-
11	12	40	140	60	55.98 (55.08)	31.7
12	12	40	140	60	56.16	-
13	12	40	150	60	47.45	-
14	12	40	150	60	49.87(48.88)	29.1
15	12	40	150	60	49.31	-
16	12	40	160	60	40.19	-
17	12	40	160	60	45.43(42.63)	28.5
18	12	40	160	60	42.28	-

**Average pulp yield in parentheses*

3.2 Physical and Strength Properties of the Handsheets

3.2.1 Bleaching response of *T. daniellii* pulps

The result of the bleaching response of both Soda and soda-ethanol pulps of *T. daniellii* stalk is presented in Table 2: Both pulp types were easy to bleach, required low chemical consumption and exhibited remarkable high brightness. Though, soda-ethanol pulps had higher brightness, the brightness of both pulp types was almost similar at similar kappa factor (which is the percentage equivalent of chlorine per kappa number). For example, at 0.12 kappa factor, soda pulp required 4.34 and 3.69% ClO_2 to reach 79.4 and 83.3% brightness, respectively in both run 1 and 2. Soda-ethanol pulp at the same ClO_2 consumption attained 82.6 and 84.5% brightness in both run 1 and 2, respectively. Therefore, on the average, soda pulp required 4.02% ClO_2 to attain a brightness level of 81.4% while soda-ethanol pulp required 3.69% ClO_2 to reach 83.55% brightness. The implication of this is that, soda-ethanol pulp with better delignification and lower kappa number required lower ClO_2 consumption to attain higher brightness compared to soda pulp. The lower ClO_2 consumption and brightness attained by *T. daniellii* pulp is quite remarkable compared to 8.0% chlorine charge required to achieve 81.2, 82.6 and 81.5% brightness, respectively for soda-AQ pulp of *H. cannabinus, C. sativa* and *H. sabdariffa* [28]. Tobacco pulp required 4.18% ClO_2 to reach a brightness level of 78.2% [9].

Table 2. Bleaching response of *T. daniellii* pulps

	Kappa factor	D_O (%ClO_2)	E (%NaOH)	D_1 (%ClO_2)	D_2 (%ClO_2)	Brightness (% ISO)
Soda pulp						
Run 1	0.12	2.66	2.0	1.0	0.68	79.4
Run 2	0.12	3.04	2.0	0.38	0.27	83.3
Soda-ethanol pulp						
Run 1	0.12	3.04	2.0	0.38	0.27	82.6
Run 2	0.13	3.04	2.0	0.38	0.27	84.5

3.2.2 Effect of beating revolutions on the pulp freeness

The relationship between beating revolutions and Canadian Standard Freeness is illustrated in Fig. 1. In unbeaten state, soda pulp had lower freeness compared to soda-ethanol pulp. At first beating (1600 revolution), the freeness of soda and soda-ethanol pulp decreased by 25% and 29%, respectively. Further increase in beating revolution to 3200 resulted in 35 and 45% reduction in soda and soda-ethanol pulp freeness, respectively. Thus, drainage rate of both pulp types was not significantly different from one another until beating was increased to 3200. What this suggests is that pulping method has a major effect on pulp refining. From this experiment, both pulps of *T. daniellii* had to be beaten to 3200 to achieve the lowest freeness (455 mL for soda-ethanol pulp and 490 mL, for soda pulp).

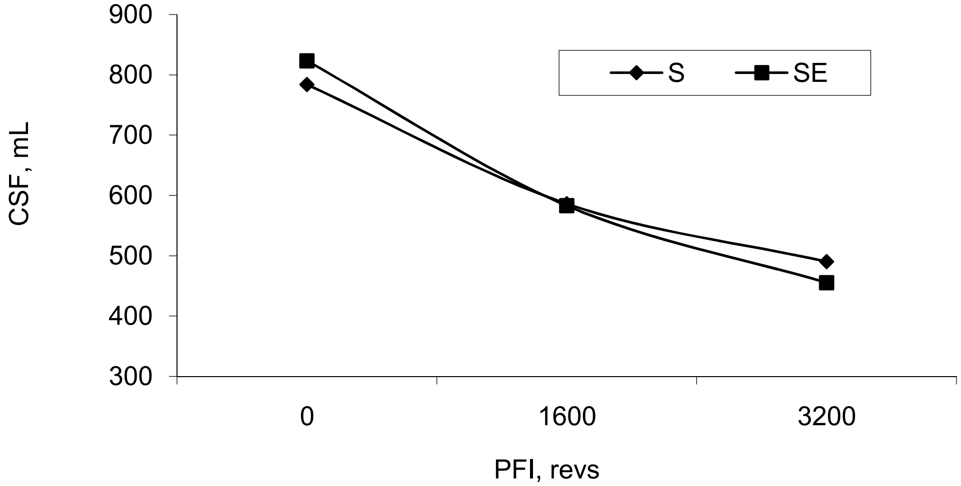

Fig. 1. Effect of PFI revolution on the freeness of soda (S) and soda-ethanol (SE) pulp

3.2.3 Strength properties of the handsheets

In unbeaten state, heterogeneity of the fibres of both pulp types resulted in the production of handsheets with higher bulk and lower apparent sheet density. But, as the pulps were subjected to beating action from 0 to 1600 PFI revolutions, the apparent sheet density of soda-ethanol pulp increased from 0.527 to 0.650 g/cm^3 while those produced from soda pulp increased from 0.455 to 0.572g/cm^3. Conversely, the average bulk of soda-ethanol pulp sheet decreased from 1.922 to 1.514cm^3/g while those from soda pulp decreased from 2.389 to 2.230 cm^3/g. This trend continued with further increase in beating to 3200 revolution. In separate works, [29,30] attributed the bulkiness and low apparent sheet density of unbeaten pulp to the presence of air void in the sheet structures occasioned by poor inter-fibre bonding. Thus as fibre-to-fibre bonding increases with beating revolution, the fine fibres plug the holes in the bonding area thereby resulting in paper with higher density and lower bulk. Meanwhile, at each beating revolution, the differences in average bulk and apparent sheet density of soda pulp and soda-ethanol pulps were always significant as expressed by the student t-test data result (Table 3). Similarly, at a given freeness level, handsheets produced from soda-ethanol pulp had higher apparent density compared to those of soda pulp (Fig. 2). This implied that soda-ethanol liquor with better delignification rate and higher carbohydrate retention produced pulp with longer and more flexible fibres which have capacity for stronger bonds formed denser sheets compared to soda pulped fibres. This behaviour is similar to what obtains in previous reports on soda organosolv of Jute [31], alkaline-ethanol pulp of Giant reed [26] and alkaline-ethanol pulp of cotton stalk [27].

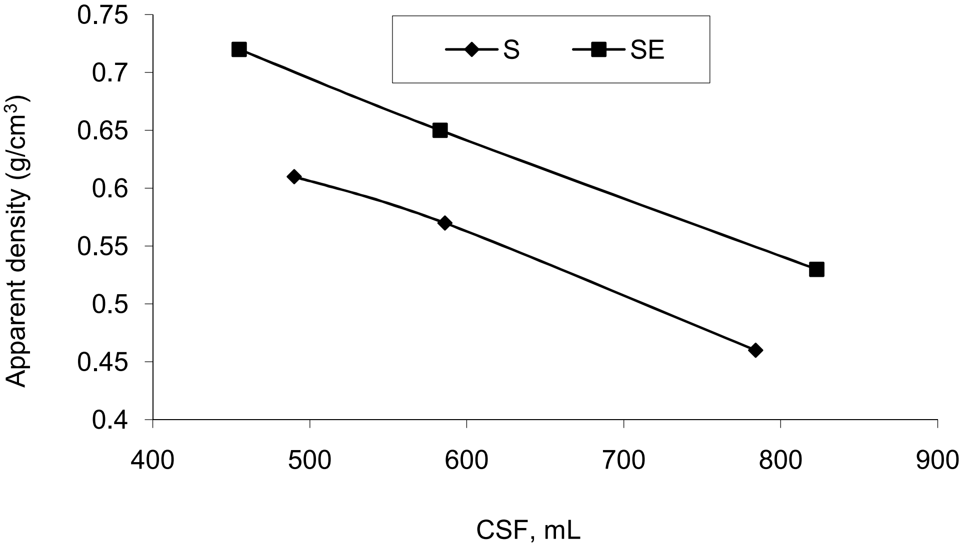

Fig. 2. Effect of pulp freeness on the apparent density of soda (S) and soda-ethanol (SE) handsheets

Meanwhile, except for the tear index, all strength properties including burst index, tensile index and breaking length increased with beating revolutions. Many authors have adduced reasons for the decrease in tear strength with beating revolutions. This can be attributed to the damage of fibre structure which turns out very weak with increased refining intensity [32]. In a separate work, it was suggested that since tearing strength is dependent on fibre length and fibre bonds, the reduction of the fibre lengths occasioned by the cutting action, external and internal fibrillations, and brushing action of the PFI mill was responsible for the decrease in tearing strength [33]. However, as the PFI mill increased from 0 to 1600 revolution, the burst, tensile and breaking length of soda pulp handsheets increased significantly from 1.258

– 6.347 kPa.m^{2}/g, 31.06 – 69.28 Nm/g and 3.14 – 7.06 Km, respectively. Similarly, those produced from soda-ethanol pulp increased from 2.326 – 7.780 kPa.m2/g, 42.346 – 84.463 Nm/g and 4.31 – 8.61 Km, respectively. One major deduction from this study is that the strengths of soda-ethanol pulp sheets were much superior to those of soda pulp as evidenced by the t-test results in Table 2. Also, at a given freeness level, soda-ethanol handsheets exhibited superior strength development compared to those of soda pulp (Fig. 3, 4 and 5). By implication, the better delignification and fibre stabilization of soda-ethanol pulp fibres made the pulp easier to beat and rendered the fibres more flexible and conformable in sheet consolidation, yielding increased fibre bonding. Due to the degradation of its fibres, handsheets produced from soda pulp had significantly lower strength properties in unbeaten state and at 1600 PFI revolution compared to soda-ethanol pulps with much longer fibres. However, beyond 1600 revolutions, the fibres became relatively well refined and fibrillated. Consequently, the differences in tear tensile and breaking length handsheets produced from soda-ethanol pulp fibres and those of soda pulp became narrowed and insignificant, an indication that the effect of fibre length was overridden by the increase in bonding capabilities of the fibres. On the average, the properties of the handsheets produced from bleached soda and soda-ethanol pulps *T. daniellii* stalk are quite remakable compared to those reported for notable non-woods like bleached Nalgrass kraft pulp [34], bleached soda-AQ Jute pulp [35], bleached *H. cannabinus, H. sabdariffa and C. sativa* alkaline sulphite-AQ pulps [28] and bleached kenaf bast kraft pulp [18]. This further buttressed the enormous potentials of *T. daniellii* stalk for pulp and paper production. The increase in burst index, tensile index and breaking length of the handsheets with beating revolution could be attributed to both external and internal fibrillation of the fibres which exposed the fibrils in the secondary wall to polyelectrolyte adsorption and inter-fibre bonding thereby increasing the paper strength. This observed trend is similar to that made on kenaf bast fibres [11], Cotton stalk [27], *Paulownia elongata* [36] and Jute [35].

Table 3. Strength properties of the handsheets

Strength Properties	**Pulp type**	**PFI revolutions**		
		0 (Unbeaten)	**1600**	**3200**
Bulk (cm^{3}/g)	Soda	2.39±1.42^{a}	2.23±0.19^{a}	1.77±0.21^{b}
	Soda-ethanol	1.92±0.12^{a}	1.51±0.07^{b}	1.40±0.09^{c}
	t-test	4.35*	6.72*	4.40*
Apparent density (g/cm^{3})	Soda	0.46±0.05^{c}	0.57±0.06^{a}	0.51±0.18^{b}
	Soda-ethanol	0.53±0.03^{c}	0.65±0.08^{b}	0.72±0.04^{a}
	t-test	3.82*	4.38*	6.73*
Tear index (mN.m^{2}/g)	Soda	3.02±0.26^{a}	2.46±0.32^{b}	2.27±0.33^{b}
	Soda-ethanol	3.49±0.91^{a}	2.73±0.45^{b}	2.32±0.22^{b}
	t-test	1.87^{ns}	1.45^{ns}	0.27^{ns}
Burst index (kPa.m^{2}/g)	Soda	1.26±0.42^{b}	6.35±0.73^{a}	6.23±0.66^{b}
	Soda-ethanol	2.33±0.17^{b}	7.78±0.26^{a}	7.63±0.44^{a}
	t-test	22.98*	10.54*	9.30*
Tensile index (Nm/g)	Soda	31.06±4.0^{b}	69.28±7.2^{a}	70.07±6.4^{a}
	Soda-ethanol	42.35±2.9^{c}	84.46±9.3^{a}	76.07±9.6^{b}
	t-test	11.97*	2.60*	1.32^{ns}
Breaking length (Km)	Soda	3.14±0.54^{b}	7.06±1.33^{a}	7.13±1.20^{a}
	Soda-ethanol	4.31±0.51^{c}	8.61±1.85^{a}	7.76±1.55^{b}
	t-test	12.93*	2.60*	1.34^{ns}

**Values with the same alphabet in each row are not significantly different at α = 0.05*

** = significance, ns = not significance, ± = standard deviation*

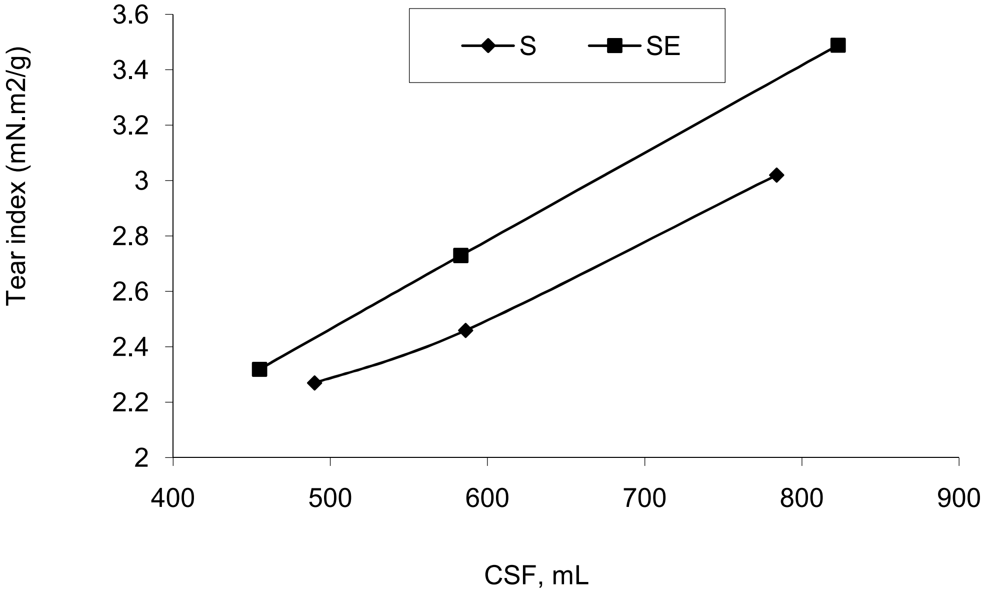

Fig. 3. Effect of pulp freeness on the tear index of soda (S) and soda-ethanol (SE) handsheets

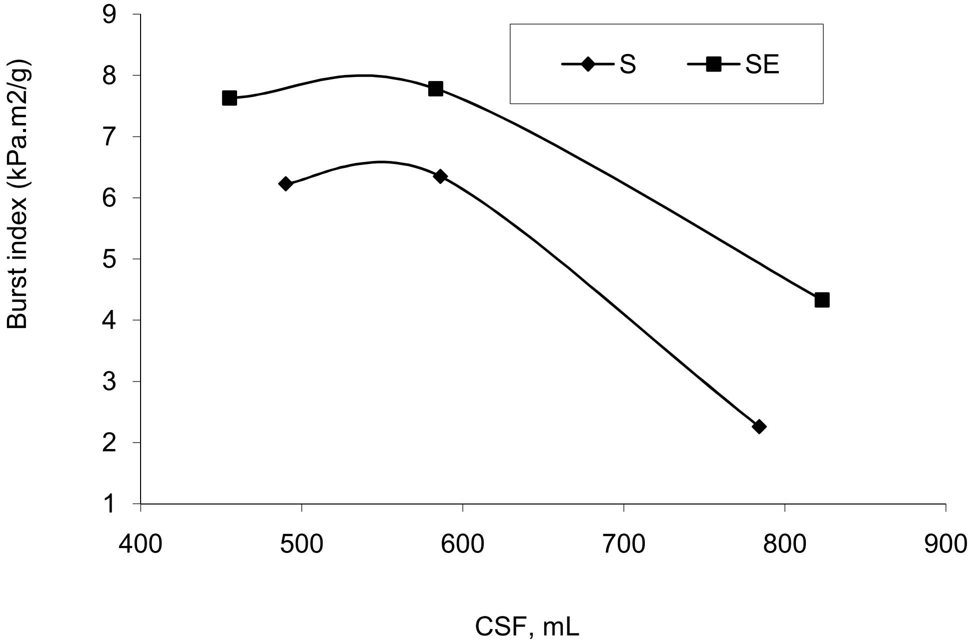

Fig. 4. Effect of pulp freeness on the burst index of soda (S) and soda-ethanol (SE) handsheets

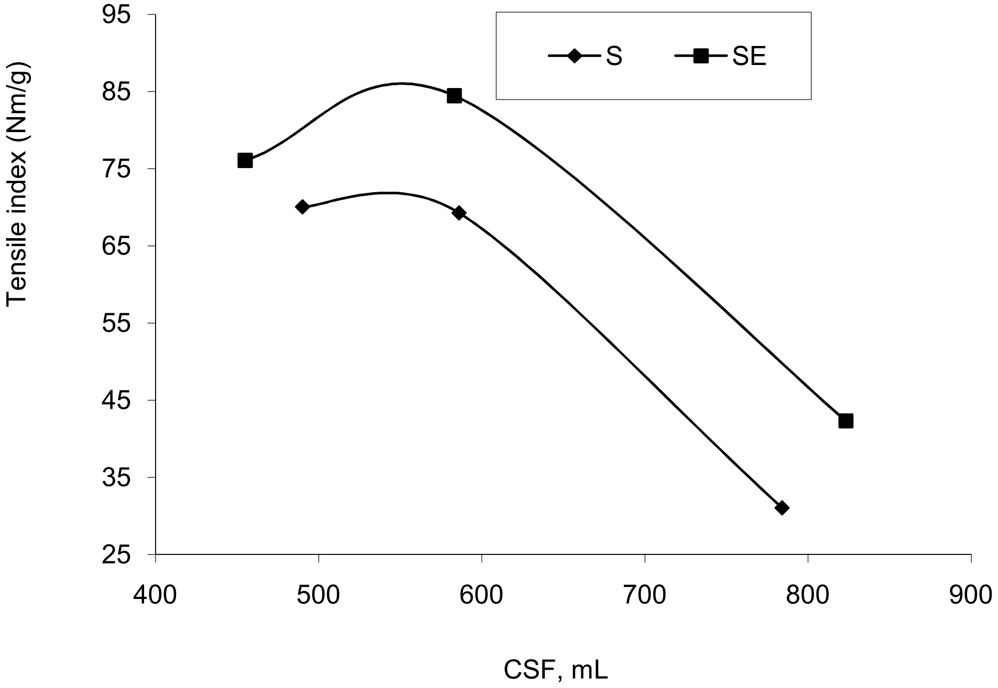

Fig. 5. Effect of pulp freeness on the tensile index of soda (S) and soda-ethanol (SE) handsheets

4. CONCLUSION

T. daniellii stalk possess many qualities required by a good non-wood fibre source for papermaking. The yields derived from its soda and soda-ethanol pulps were quite remarkable. Addition of ethanol to soda liquor led to an increase in pulp yield and reduction in kappa number. The pulps were easy to bleach and required low chemical consumption to achieve high brightness level. Soda and soda-ethanol pulps required 4.02% and 3.69% ClO_2 consumption to achieve 81.4% and 84.5% brightness respectively. The pulps were also easy to beat and the handsheets produced also had good strength properties comparable to those from notable non-wood sources. The pulping, bleaching and papermaking properties of soda-ethanol pulps exhibited characteristics that are superior to those of soda pulp. Meanwhile, beating did not improve significantly the physical and majority of the strength properties of the handsheet beyond 1600 revolution. It therefore makes more economic sense to select 1600 as the optimum revolution for beating *T. daniellii* pulps.

CONSENT

This study was conceived and carried out by the authors involved. No consent is required from other party.

ETHICAL APPROVAL

The study did not violate any human or animal rights.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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