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

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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* used for the study were pulped with both sodium hydroxide (NaOH) and soda-ethanol (NaOH-C₂H₅OH) liquors. To determine the yield and kappa number of the pulp of *T. daniellii* stalk, the stalks were pulped in a 5-litre electrically heated stainless steel digester. Cooking temperature was varied at 140, 150 and 160 °C while the concentration of the cooking liquor was kept constant at 12% NaOH and 12:40% by volume of NaOH-C₂H₅OH 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 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 effect on the yield, kappa number and the 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 significant 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: Soda and soda-ethanol pulp; DEDD bleaching; handsheet properties; *T. daniellii* stalk; non-wood fibre source.

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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]. Nigeria, being a rapidly growing population with an estimate of over 167 million people has a steady increasing requirement for pulp, paper and paper products. This is as a result of increase in literacy level, industrialization and quest for information resulting in considerable growth in computer services and newspaper industry. All of these make paper consumption almost inseparable from the people's way of life. This increase in consumption of paper and paper products in Nigeria necessitated the establishment of Nigerian Paper mill Limited, Jebba, Kwara State, Nigerian Newsprint Manufacturing Company, Oku-Iboku, Akwa-Ibom State, and Nigerian National Paper Manufacturing Company, Iwopin, Ogun State with total rated capacity of 265,000 metric tonnes [2]. The manufacture of pulp and paper products in Nigeria was conceived as part of the second National Development Plan, 1970-74, which had the objectives of a united, strong and self-reliant nation with vigorous import substitution [3]. However, the policy of import substitution was not achieved due to large dependence on importation of long fibre pulp. The huge investment on imported long fibre pulp has been identified as one of the limiting factors against the growth of the pulp and paper industry in the country. For example, in the peak years of 1980 – 1981, over 85,000MT of long fibre pulp were imported which represented nearly 95% of total fibre supply to the paper mills. Similarly, from 1978 to 1984, about N106.5million was spent annually on importation of paper and paper products [4]. These among others was responsible for the below performance and final closure of the mills. However, the current effort of the Federal Government to revamp the pulp and paper sector coupled with climatic restriction to the establishment of long fibre pulpwood species in Nigeria has prompted researchers to explore the option of non-wood plant species in pulp and paper production. The utilization of non-wood fibre is an ethically sound way of promoting sustainable paper production especially in areas lacking long fibre pulpwoods but having abundance of annual fibre resources. Among the various plants that thrive well in Nigeria, *T. danielli* stands prominent.

T. danielli (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) [5]. It is indigenous to the rainforest of West Africa particularly the southern parts of Ghana, Cote d'Ivoire and Nigeria [6]. It is also known to exist in the Princes Islands, Angola, the Central African Republic, Uganda and Indonesia. The plant offers several advantages including high annual yield per hectare, self regeneration after harvest through its rhizomes, moderate fertilization requirement [7], ease of harvesting and transportation [8]. The main economic use of the plant is the extraction of thaumatin from its fruit for sweetner [9] and the use of its leaves for wrapping [10].

Recent researches have been focused on the use of its stalk as a non-wood fibre source. Oluwadare and Sotannde introduced this plant as a non-wood fibre source for pulp and papermaking [11,12,13]. It has fibres comparable to major non-wood fibres used in papermaking [13] and good pulping properties from different ecological zones [14]. 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 its soda and soda-ethanol pulps.

2. MATERIAL 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 pulped with both sodium hydroxide and mixture sodium hydroxide and ethanol. The soda cooking liquor was prepared from a standard concentrated solution of sodium hydroxide by dilution with de-ionized water to 12%. Soda-ethanol pulping liquor was prepared by mixing equal volume of sodium hydroxide and ethanol at 12-40% by volume (i.e. 12% sodium hydroxide and 40% ethanol). The stalks were pulped in a 5-litre electrically heated stainless steel digester. The cut stalks of 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 160°C and cooking time of 60 minutes. The resulting pulp was thoroughly washed with tap water and the pulp yield determined after air-drying for 72 hours in the laboratory while the kappa number was determined in accordance with Tappi T 236 method.

2.2 Pulp Bleaching

The pulps were bleached using three-stage chlorine dioxide and alkaline (D₀ E₁ D₁ D₂) 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% ClO₂ 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/m² 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 25°C and 50% relative humidity (RH) for 48 hours prior to testing. Based on Tappi methods, the bulk (cm³/g), apparent sheet density (g/cm³), tear index (mN.m²/g), burst index (kPa.m²/g), tensile index (Nm/g) and breaking length (Km) of the handsheets were evaluated based on Tappi standard methods.

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 140 °C for 60 minutes. Higher pulp yield and higher Kappa number were obtained at the lower temperature of 140 °C, while lower pulp yield and lower Kappa number were obtained at elevated temperatures. This implies that high rate of lignin-carbohydrate complexes dissolutions [15] and solubilisation of significant amount of hemicelluloses [16] occurs at elevated temperatures. Hence, during pulping both lignin and cellulose are dissolved at different rates. This rate is much accelerated by increasing the temperature of the pulping medium [17]. The decrease in pulp yield and kappa number became gradual afterwards as evidenced in 14.37% and

120 13.45% reduction in pulp yield and, 12% and 1.35% in kappa number at elevated
121 temperatures of 150 °C and 160 °C, respectively.

122
123 Meanwhile, when soda method was modified with ethanol, the pulp yields increased while
124 the kappa numbers of the resulting pulps decreased. Hence, addition of 40% ethanol to 12%
125 soda pulping liquor at 140 °C resulted in 18.12% increase in average pulp yield and
126 reduction in kappa number by 9.43%. The trend continued at elevated temperatures of 150
127 and 160 °C. This revealed that adding ethanol to the conventional soda process was
128 effective for improving both delignification [18,19] and the physical properties of the pulps
129 produced from *T. daniellii* stalk. Thus, the delignification proceeded more rapidly and more
130 selectively with alkali-ethanol than with alkali alone, giving higher yields and lower kappa
131 numbers. Similar trend was reported on the addition of organic solvent to soda pulping
132 mixture of *Arundo donax* [20], Nigerian grown *Bambusa vulgaris* [21] and cotton stalks [22].
133 A higher pulp yield with soda-ethanol, compared to the soda method, could be attributed to
134 higher hemicellulose retention [18,19]. Meanwhile, beyond 150 °C, marginal decrease in the
135 yield and kappa number of both soda and soda-ethanol pulps were obtained. Thus it might
136 not be economical to pulp *T. daniellii* stalks beyond 150 °C. Hence 150 °C was selected as
137 its optimum cooking temperature.

138
139 **Table 1. Influence of pulping conditions on the yield and kappa number of *T.***
140 ***daniellii* pulp**
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Cook Number	Soda (%)	Ethanol (%)	Cooking temperature (°C)	Cooking time (Minutes)	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	-

142 * Average pulp yield in parentheses.
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144 3.2 Physical and Strength Properties of the Handsheets

145 3.2.1 Bleaching Response of *T. daniellii* Pulps

146 The result of the bleaching response of both Soda and soda-ethanol pulps of *T. daniellii* stalk
147 is presented in Table 2: Both pulp types were easy to bleach, required low chemical
148 consumption and exhibited remarkable high brightness. Though, soda-ethanol pulps had
149 higher brightness, the brightness of both pulp types was almost similar at similar kappa
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factor (which is the percentage equivalent of chlorine per kappa number). 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. This 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* [23]. Tobacco pulp required 4.18% ClO_2 to reach a brightness level of 78.2% [24].

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

	D ₀ (% ClO_2)	E (%NaOH)	D ₁ (% ClO_2)	D ₂ (% ClO_2)	Brightness (% ISO)
Soda pulp					
Kappa factor (0.12)	2.66	2.0	1.0	0.68	79.4
Kappa factor (0.12)	3.04	2.0	0.38	0.27	83.3
Soda-ethanol pulp					
Kappa factor (0.12)	3.04	2.0	0.38	0.27	82.6
Kappa factor (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 Figure 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 beating to 3200 to achieve the lowest freeness (455 mL for soda-ethanol pulp and 490 mL, for soda pulp).

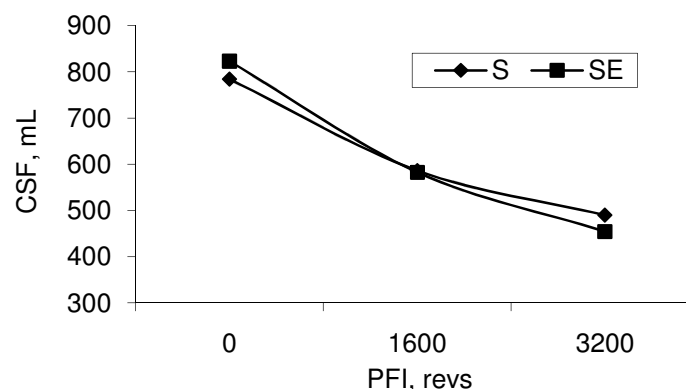


Fig. 1. Effect of PFI revolution on the freeness of soda (S) and soda-ethanol (SE) pulp

Table 3. Strength properties of the handsheets

Strength Properties	Pulp type	PFI revolutions		
		0 (Unbeaten)	1600	3200
Bulk (cm^3/g)	Soda	2.389 ^a	2.230 ^a	1.771 ^b
	Soda-ethanol	1.922a	1.514b	1.395c

Apparent density (g/cm^3)	t-test (significance)	4.35*	6.72*	4.40*
	Soda	0.455 ^c	0.572 ^a	0.513 ^b
	Soda-ethanol	0.527 ^c	0.650 ^b	0.722 ^a
Tear index ($\text{mN.m}^2/\text{g}$)	t-test (significance)	3.82*	4.38*	6.73*
	Soda	3.022 ^a	2.464 ^b	2.267 ^b
	Soda-ethanol	3.488 ^a	2.728 ^b	2.320 ^b
Burst index ($\text{kPa.m}^2/\text{g}$)	t-test (significance)	1.87 ^{ns}	1.45 ^{ns}	0.27 ^{ns}
	Soda	1.258 ^b	6.347 ^a	6.226 ^b
	Soda-ethanol	2.326 ^b	7.780 ^a	7.633 ^a
Tensile index (Nm/g)	t-test (significance)	22.98*	10.54*	9.30*
	Soda	31.06 ^b	69.28 ^a	70.07 ^a
	Soda-ethanol	42.346 ^c	84.463 ^a	76.065 ^b
Breaking length (Km)	t-test (significance)	11.97*	2.60*	1.32 ^{ns}
	Soda	3.14 ^b	7.06 ^a	7.13 ^a
	Soda-ethanol	4.31 ^c	8.61 ^a	7.76 ^b
	t-test (significance)	12.93*	2.60*	1.34 ^{ns}

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

* = significance, ns = not significance

3.2.3 Strength Properties of the Handsheets

In unbeaten state, heterogeneity of the fibres of both pulp type 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 0.572 g/cm^3 . Conversely the average bulk of soda-ethanol pulpsheet decreased from 1.922 to 1.514 cm^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, [25] and [26] 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 difference in the 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 2). Similarly, at a given freeness level, soda-ethanol handsheets exhibited lower bulk and higher 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 previous reports on soda organosolv of Jute [27], alkaline-ethanol pulp of Giant reed [20] and alkaline-ethanol pulp of cotton stalk [22].

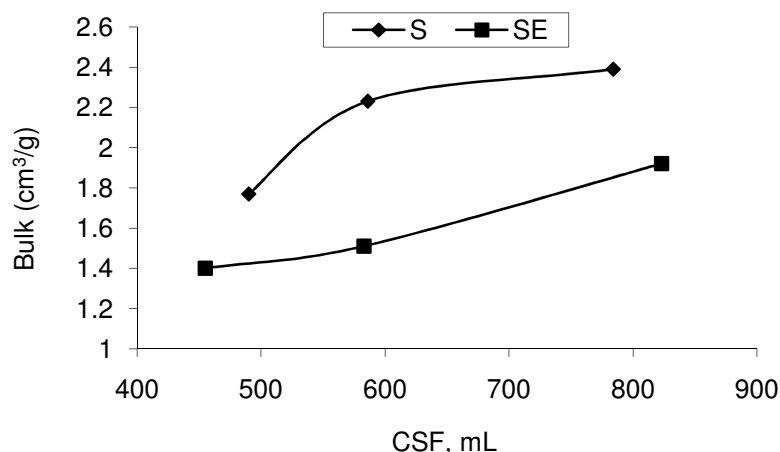


Fig. 2. Effect of pulp freeness on the bulk of soda (S) and soda-ethanol (SE) handsheets

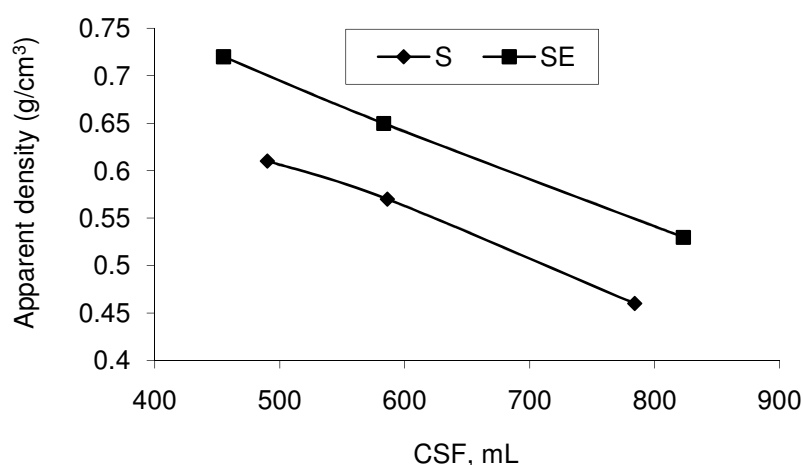
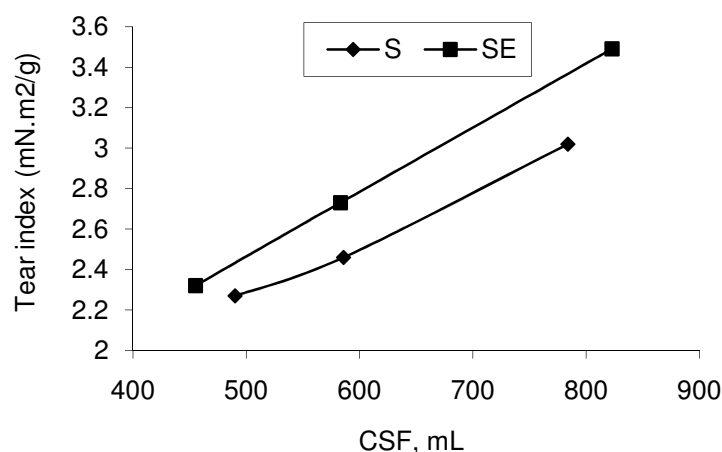


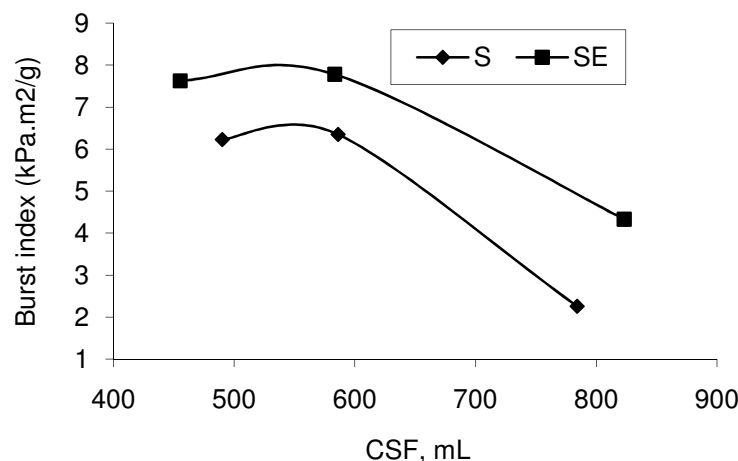
Fig. 3. Effect of pulp freeness on the apparent density of soda (S) and soda-ethanol (SE) handsheets

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 [28]. 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 [29]. 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.m²/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 pulpsheets 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. 4, 5, 6 and 7). By implication, the better delignification and fibre stabilization of soda-ethanol

230 pulp fibres made the pulp easier to beat and rendered the fibres more flexible and
 231 conformable in sheet consolidation, yielding increased fibre bonding. Due to the degradation
 232 of its fibres, handsheets produced from soda pulp had significantly lower strength properties
 233 in unbeaten state and at 1600 PFI revolution compared to soda-ethanol pulps with much
 234 longer fibres. However, beyond 1600 revolutions, the fibres became relatively well refined
 235 and fibrillated. Consequent, the differences in tear tensile and breaking length handsheets
 236 produced from soda-ethanol pulp fibres and those of soda pulp became narrowed and
 237 insignificant, meaning that the effect of fibre length was overridden by the increase in
 238 bonding capabilities of the fibres. On the average, the properties of the handsheets
 239 produced from bleached soda and soda-ethanol pulps *T. daniellii* stalk are quite remarkable
 240 compared to those reported for notable non-woods like bleached Nalgrass kraft pulp [30],
 241 bleached soda-AQ Jute pulp [31], bleached *H. cannabinus*, *H. sabdariffa* and *C. sativa*
 242 alkaline sulphite-AQ pulps [23] and bleached kenaf bast kraft pulp [2]. This further buttressed
 243 the enormous potentials of *T. daniellii* stalk for pulp and paper production. The increase in
 244 burst index, tensile index and breaking length of the handsheets with beating revolution
 245 could be attributed to both external and internal fibrillation of the fibres which exposed the
 246 fibrils in the secondary wall to polyelectrolyte adsorption and inter-fibre bonding thereby
 247 increasing the paper strength. This observed trend is similar to the observation on kenaf bast
 248 fibres [2], Cotton stalk [22], *Paulownia elongata* [32] and Jute [31].
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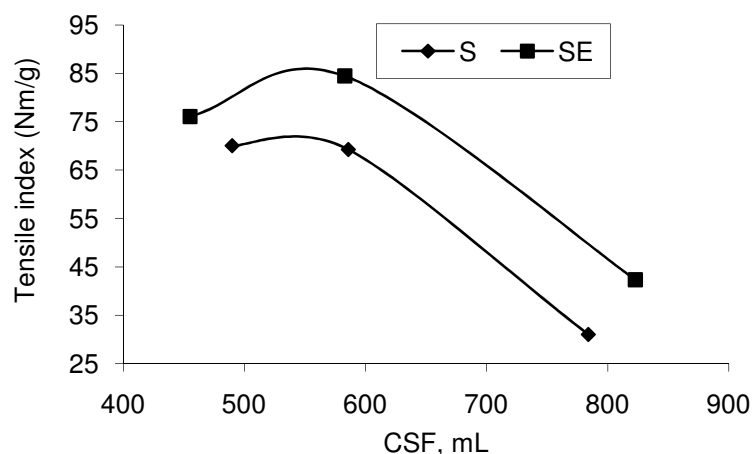


250 **Fig. 4. Effect of pulp freeness on the tear index of soda (S) and soda-ethanol (SE)**
 251 **handsheets**
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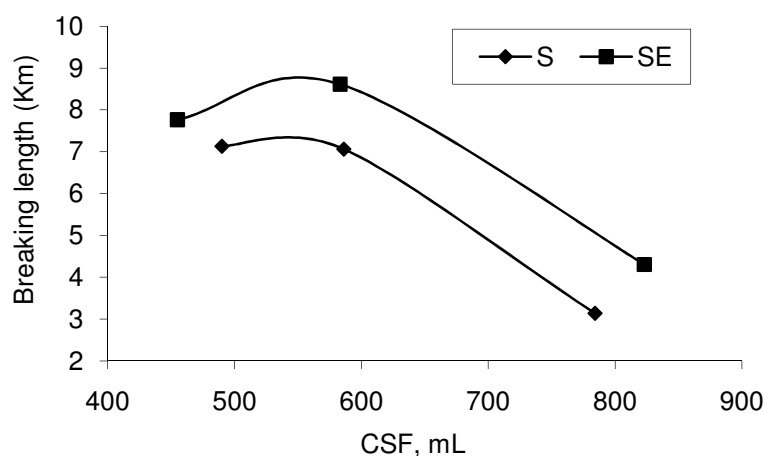


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255 **Fig. 5. Effect of pulp freeness on the burst index of soda (S) and soda-ethanol (SE)**
 256 **handsheets**
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258 **Fig. 6. Effect of pulp freeness on the tensile index of soda (S) and soda-ethanol (SE)**
 259 **handsheets**
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262 **Fig. 7. Effect of pulp freeness on the breaking length soda (S) and soda-ethanol (SE)**
 263 **handsheets**
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266 4. CONCLUSION

268 *T. daniellii* stalk possess many qualities required for a good non-wood fibre source for
 269 papermaking. The yields derived from its soda and soda-ethanol pulps were quite
 270 remarkable. Addition of ethanol to soda liquor leads to an increase in pulp yield and
 271 reduction in kappa number. The pulps were easy to bleach and required low chemical
 272 consumption to achieve high brightness level. Soda pulps required 4.02% ClO_2 consumption
 273 to achieve 81.4% brightness while soda-ethanol pulp required 3.69% ClO_2 to attain 84.5%
 274 pulp brightness. The pulps were also easy to beat and the handsheets produced also have
 275 good strength properties comparable to those from notable non-wood fibre sources. The
 276 pulping, bleaching and papermaking properties of soda-ethanol pulps exhibited
 277 characteristics that are superior to those of soda pulp. Meanwhile, beating did not improve
 278 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.

COMPETING INTERESTS

There is no competing interest.

AUTHORS' CONTRIBUTIONS

This research study is a concerted effort of all the Authors involved. Author SOA carried out the pulping experiment, statistical analysis and preparation of the manuscript. Author OAO carried out the bleaching studies, handsheet preparation and testing with the active involvement of Author AFG. All the Authors read and approved the final manuscript.

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 right

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