THE EFFECT OF ORGANOSOLV TREATMENT ON THE PHYSICOCHEMICAL PROPERTIES OF PULP AND PAPER FROM HORSE GRASS (ANDROPOGON TECTORUM).

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Abstract
The delignification of horse grass (HG) was carried out using methanol and water (organosolv pulping); varying the cooking conditions at 30min, 60min and 90min with ratios: 1:1, 1:2 and 1:3. Methanol-water, cooked in autoclave at 105°C. The pulp obtained were selected and macerated in equal volume of glacial acetic acid (ethanoic acid) and hydrogen peroxide (1:1) at a temperature 100°C, to investigate the fiber length; fiber diameter; lumen with their derived indices and paper strength properties was determined according to TAPPI standard methods, T 494 om-96. The best fiber length of 3.21mm, tear index 31.43 mN.m²/g, tensile index 97.79Nm/g and bulk index 5.15 kPa.m²/g was obtained at 1:3 methanol: H₂O at 105°C with cooking time of 60 min. Therefore, it was established that high quality papers of different grades can be produced from horse grass with acceptable strength properties using low concentration of methanol/water pulping with moderate time at boiling temperature. The research concluded that methanol-water was excellently good for pulping of horse grass fibers. The above results showed that horse grass is a good alternative sources of fibers to produce pulp and paper.

Key words: Flexibility ratio, Fiber length, Horse grass, Paper, Pulp, Tear Index

Introduction
The employment of papers for printing, packaging boards and different uses relies upon the paper items quality properties. The fiber morphological properties such as fiber length and diameter are major determinant for pulp and paper properties [1, 2]. Seth, 1998 have been shown that tearing quality of paper depends strongly on fiber length. The pulp yield and freeness is directly proportional to the fiber length notwithstanding tear index and bending stiffness and adversely with fiber width. However, length and width, are most essential characteristics of the pulp fibers [3]. According to [4] the pulp sheets strength properties could be determined by the fiber length and thickness of fibers cell wall. The relationship between fiber size and their derived values on pulp and paper strength properties is well studied. [5] reported that for quality papers to be made, it will have required fibers with good derived indices. Be that as it may, due to the pressure on forest resources, this has led to sourcing for novel kinds of fibrous plant biomass [6]. This biomass might be sourced from ranches of enduring grasses and fast growing fibrous plants such as wheat straw, bamboo, elephant, switch grass and various cereal straws; all these have effectively made financial breakthrough for the pulp business [7, 8]. Fast growing fibrous plants with short life cycle for growing would be available round the year for paper making and would reduce pressure on wood; save desertification and its attendant outcomes [9]. Considering the environmental safety and economic value; types of pulping methods must always take into consideration. Therefore, using natural solvents (ethanol, methanol, glycerol e.t.c) which are biodegradable would be an advantage over other pulping techniques which are Non-degradable [10]. In spite of the fact that the paper making potential of grasses and non-wood materials, including wheat straw, sorghum; smoothbrome grass; Miscanthus giganteus; and most particularly 'sugarcane bagasse' has been assessed and well documented for pulp and paper making [5, 6, 8]. Horse grass (Andropogon tectorum) is a robust perennial grass with culms to 3m high, often stilt-rooted, of shady sites under trees; which reproduces from seeds. It is also often found in bush re-growths and road sides. It is particularly
abundant in Nigeria as a weed. In addition, this is the first work documenting properties of pulp obtained from horse grass and compared with properly-studied non-wood materials. No comprehensive facts regarding properties of pulp and paper for horse grass are presented in the scientific literature to the best of our knowledge. The objective of this work is to pulp the fiber of horse grass and to characterize the pulp and paper properties in order to determine its possibilities for producing good quality paper.

**Experimental**

**Material and Methods**

Fresh samples of Horse grasses (Andropogon tectorum) were collected from Adekunle Ajasin University Boys hostel at Ikare Akoko, Ondo State, Nigeria. The dried samples of Horse grass were chipped manually. Chips with average length of 2-4 cm were used for the production of pulps with known moisture content (oven dry basis). The cooking was performed using different trials to produce organosolv pulp with chopped Horse grass.

Fiber dimensions,

The pulp samples from horse grass of methanol-water ratio 1:1, 1:2, and 1:3, of 30 min, 60 min and 90 min cooking time were macerated in equal volume of glacial acetic acid (ethanoic acid) and hydrogen peroxide (1:1) at a temperature \(100^\circ\text{C}\). Random sample of macerated fiber were measured on slides measured using a stage micrometer and an eye piece reticule under a light at x 80 magnification. Twenty (20) fibers were measured from each representative sample slide to keep error below 5% and for a 95% confidence level [11].

**Derived values and Statistical analysis**

Three derived values were also obtained using fiber dimensions: slenderness ratio as fiber length/fiber diameter, flexibility coefficient as (fiber lumen diameter/ fiber diameter) \(\times100\) and Runkel ratio as \((2 \times \text{fiber cell wall thickness})/\text{lumen diameter}\) according to [12]. Data collected was subjected to SPSS software using compare means to determined (e.g. fiber length; Diameter, Lumen diameter and cell wall thickness).

**Laboratory paper sheet Formation**

Handmade paper sheets were obtained by spreading the slurry on a laboratory sheet former. The paper samples formed were then allowed to dry for about 24 hours. The strength properties of handmade paper samples such as, breaking weight, tensile strength, and tear strength was done using Instron universal testing machine in accordance with TAPPI standard methods, T 494 om-96.

**RESULTS AND DISCUSSION**

**Fiber dimensions**

The pulp fiber characteristics are presented in Table 1. The fiber lengths for horse grass ranges from 1.38-3.12 (mm). In addition, the results indicate a considerable proportion of long fibers for horse grass and the fiber diameters result showed ranges from 18.46-20.55 (µm).
Table 1: Table 1 Fiber morphology parameters of horse grass pulp obtained at 105°C; 1:1; 1:2; 1:3 methanol-water; 30min, 60min and 90min.

| Pulping Time (min) | Methanol/water | FL (mm)  | FD (µm)  | LW (µm)  | CW (µm)  |
|-------------------|----------------|---------|---------|---------|---------|
| 30                | 1:1            | 1.89 ± 0.12 | 20.92 ± 1.60 | 9.59 ± 1.17 | 5.69 ± 0.73 |
| 30                | 1:2            | 1.38 ± 0.13 | 20.55 ± 1.30 | 10.91 ± 0.84 | 4.82 ± 0.43 |
| 30                | 1:3            | 2.30 ± 0.17 | 27.18 ± 1.23 | 8.31 ± 0.34 | 9.44 ± 0.60 |
| 60                | 1:1            | 1.43 ± 0.16 | 15.50 ± 1.37 | 6.73 ± 1.31 | 4.39 ± 0.37 |
| 60                | 1:2            | 1.69 ± 0.15 | 25.35 ± 1.66 | 11.78 ± 1.05 | 6.78 ± 0.70 |
| 60                | 1:3            | 3.12 ± 0.18 | 18.46 ± 1.66 | 10.05 ± 0.65 | 4.21 ± 0.35 |
| 90                | 1:1            | 1.55 ± 0.11 | 34.43 ± 1.70 | 29.53 ± 3.57 | 2.45 ± 1.90 |
| 90                | 1:2            | 1.96 ± 0.36 | 20.25 ± 1.77 | 14.13 ± 1.54 | 3.06 ± 0.37 |
| 90                | 1:3            | 1.73 ± 0.19 | 22.29 ± 1.30 | 11.99 ± 1.16 | 5.15 ± 0.42 |

FL=fiber length, FD=fiber diameter, LW= lumen width and CW= cell wall

From Table 1; the lumen width ranges from 6.73-29.53 (µm) and cell walls thickness ranges from 2.45-9.44 (µm). The lumen width of horse grass was comparable to that of different non-wood fibers such as kenaf and European aspen, but more extensive than that of wheat straw and rice straw. While the cell walls were thinner than sorghum stalks and European aspen as reported by [15, 16].

Fig. 1: Effect of cooking time at 105°C on fiber length of horse grass

The (fig.1-2) showed the results of fiber lengths and fiber diameter. It was observed from (fig.1) that fiber lengths of both tested samples were at the optimum at 1:3 methanol: water with
cooking time 60mins but lowest at 1:2 methanol:water with 30mins cooking time. This showed that shorter time with high concentration might have little effects on fibers of horse grass with the concentration above. It was also observed from (fig.1) that fiber length at 60min cooking time was increasing with decreasing in methanol-water concentration; which shows that diluted methanol used for moderate time of heating gives better results. While the fiber diameter of horse grass with 30min and 90min cooking time was decreasing and later increased with cooking time, while 60min increases and later decreased. However, the average lengths and diameter of horse grass sample were generally in the range expected of grasses, non-wood materials and some wood species [13, 14].

Table 2: Derived indices parameters of horse grass pulp obtained at 105°C; 1:1; 1:2 and 1:3 methanol-water; 30min, 60min and 90min.

| Pulping time (min) | Methanol/water | Slender ratio | Flexibility ratio | Runkel ratio |
|-------------------|----------------|--------------|-------------------|-------------|
| 30                | 1:1            | 90.36        | 45.84             | 1.19        |
| 30                | 1:2            | 67.14        | 53.10             | 0.88        |
| 30                | 1:3            | 84.61        | 30.58             | 2.27        |
| 60                | 1:1            | 92.23        | 43.42             | 1.30        |
| 60                | 1:2            | 61.15        | 46.47             | 1.15        |
| 60                | 1:3            | 169          | 54.42             | 0.84        |
| 90                | 1:1            | 49.09        | 85.78             | 0.17        |
| 90                | 1:2            | 96.80        | 69.77             | 0.43        |
| 90                | 1:3            | 75.64        | 53.78             | 0.86        |

Sr = Slender ratio; Fr = Flexibility ratio; Rr = Runkel ratio

Derived values

Slenderness ratio, felting ratio, and Runkel ratio, are parameters derived from fiber measurements. Table 2 above showed that slenderness ratio of horse grass ranges from 61.15-169. While the flexibility ratio of fibers results, shows that horse grass fibers have flexibility ratio ranges 30.58-85.78, and runkel ratio results showed the ranges 0.43-2.27 respectively. The high slenderness ratios of horse grass make their pulps suitable for printing, writing and/or even packaging paper according to [17]. With the above flexibility ratio results; the fibers of each tried examples can be classified into flexible fibers according to [18]. Since horse grass average runkel proportions of 30 min and 60min was greater than 1 it shows the fiber range are inflexible; while 90 min average runkel ratio was less than 1. Nevertheless, both tested samples have a corresponding slender ratio, flexibility ratio and runkel ratio values that are comparable to other non-wood materials such as bamboo; rice straw; sorghum stalks and river red gum [16, 19].

Strength Properties of handmade Paper

The result in Table 3 shows the properties of horse grass, the result showed that the tear index ranges from 13.73-31.43 (mN.m²/g), while the result of tensile index ranges from 74.26-97.79 (N/mg) and the result of burst index ranges from 1.56-5.15 (kPa.m²/g) respectively.
Table 3: Strength properties of paper sheets made from Horse grass obtained at 105°C; 1:1; 1:2; 1:3 methanol-water; 30min, 60min and 90min.

| Methanol/water ratio | Pulping Time (min) | Basis weight (g.m⁻²) | Tear index (mN.m²/g) | Breaking weight (N) | Tensile index(N/mg) | Burst Index (kPa.m²/g) |
|----------------------|--------------------|----------------------|----------------------|---------------------|---------------------|-----------------------|
| 1:1                  | 30                 | 80.00                | 14.61                | 14.29               | 77.67               | 1.96                  |
| 1:2                  | 30                 | 80.00                | 13.73                | 15.13               | 82.24               | 3.60                  |
| 1:3                  | 30                 | 80.00                | 27.88                | 17.34               | 94.23               | 4.47                  |
| 1:1                  | 60                 | 80.00                | 10.08                | 13.66               | 74.26               | 1.86                  |
| 1:2                  | 60                 | 80.00                | 22.32                | 16.48               | 89.59               | 2.01                  |
| 1:3                  | 60                 | 80.00                | 31.43                | 17.99               | 97.79               | 5.15                  |
| 1:1                  | 90                 | 80.00                | 25.00                | 15.79               | 85.80               | 1.56                  |
| 1:2                  | 90                 | 80.00                | 16.18                | 15.97               | 86.79               | 4.09                  |
| 1:3                  | 90                 | 80.00                | 18.42                | 16.59               | 90.16               | 2.15                  |

Fig. 3, showed that the tear index of both tested samples of horse grass was at the optimum 31.43 mN.m²/g, while (fig. 4), showed that the tensile index was at the optimum 97.79 N/mg and the optimum burst index observed at 5.15 kPa.m²/g. All the optimum result was observed at 1:3 methanol-water with cooking time 60mins. It was also shown that tear index was increasing with methanol-water concentration at 60min cooking time, which shows that the tear index is proportional to methanol concentration with time.

Fig. 5: Effect of cooking time at 105°C on burst index of horse grass

However, the result shows that tear index, tensile index and burst index of horse grass are in the range of grasses as detailed [13, 14, 18].
Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) images has been used for the evaluations and analysis of paper structure [20, 21]. The scanning image of paper prepared from hydrogen peroxide acetic acid (HPAC) bleached pulp and methanol-water pulp are shown in (figure 5A and 5B). The results show that there were no visible pores or areas of destruction on the surfaces of fibers of horse grass paper produced by hydrogen peroxide acetic acid (HPAC) bleaching and methanol-water pulping. This is an evidence that cellulose degradation was very low during methanol-water pulping and HPAC bleaching according to [22]. Therefore, pulp and paper properties of methanol-water and HPAC bleaching were excellently good for horse grass fibers.

![Figure 5. (A) Scanning image of paper produced from hydrogen peroxide acetic acid (HPAC) bleached pulp and 5. (B) Scanning image of paper produced from 1:3 methanol-water pulping](image)

Conclusions

The comparison of horse grass fiber dimension and strength properties with methanol/water pulping process has been presented. The pulp from horse grass was characterized by fairly long fibers. The fiber length; flexibility coefficients; runkel ratios and slenderness ratios results; of horse grass fibers; showed that their pulps are suitable for printing, writing and/or even packaging paper. The research also recommended cooking of horse grass fibers with 1:3 methanol-water; 60min cooking time at 105°C. From the experimental results, it was observed that the best strength properties of tear index, tensile index and bulk index was produced with low concentration of methanol with moderate cooking time and low temperature. The research concluded that methanol-water was excellently good for pulping of horse grass fibers. It is therefore established that horse grass is a good alternative sources of fibers to produce pulp; and paper products.

Conflict of interest

We jointly declare that there is no conflict of interest of any sort.

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