# Physico-chemical Characterization of Lignocellulosic fibre from *Ampelocissus cavicaulis*

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***ABSTRACT:*** *Ampelocissus cavicaulis bast fibres were extracted from the young plant stems using water retting extraction procedure, and the resulting fibres were uniform with almost circular cross-sections. Phytochemical analysis revealed the presence of residual tannins, alkaloids, steroids, glycosides, and proteins, with the absence of free reducing sugars and flavonoids. Further analysis revealed the quantity of tannins, alkaloids and steroids to be 0.01±0.002, 6.40±0.28 and 0.03±0.004%w/w respective ly. Lipophilic and alcoholic extractives obtained using soxhlet technique with n-hexane / methanol as extracting solvents were found to be 1.96%w/w and 9.21%w/w respectively. Moisture and ash content were found to be 5.60± 0.071%w/w and 2.88±0.90%w/w respectively. Structura l component on %w/w basis shows that cellulose is 38.66±1.15%, acid insoluble lignin 33. 21±2.76%, hemicellulose 25.91±1.29%, acid soluble lignin 1.47±0.01%, and the acid soluble lig nin derived product-vanillic, p-coumaric and ferulic acids were found to be 1.346±0.022%w/w, 0.0 34±0.02%w/w and 0%w/w respectively. Cellulose lignin ratio is 1.16. The physical and mechanical parameters of the fibre determined include diameter 0.9174mm, tensile strength 238.28MPa, percentage elongation 3.0 % and Young's modulus 3971.33MPa. The results obtained show that A. cavicaulis lignocellulose bast fibre has comparable properties with other natural fibres and some synthetic fibres.*

## INTRODUCTION

*Ampelocissus cavicaulis* is a woody climber found abundantly in tropical forests of Southern Nigeria and Western Cameroons, and across central Africa from Sudan to Zaïre, Uganda and Angola. After pounding and washing, the stems are traditionally used in making sponge. The leaves are mucilaginous and ornamental. The fruits are used as food, and the leaves are used in ethno medicine for treating haemorrhoids and veneral diseases (Burkill, 2000). Lignocellulosic fibres are polymer composed of crystalline cellulose microfibril-reinforced aromatic lignin and hemicelluloses matrix, as well as non structural phytochemicals or extractives which influences their utilization. Natural fibres have been used to reinforce materials for over 3,000 years. More recently they have been employed in combination with plastics. Many types of natural fibres have been investigated for use in plastics including Flax, hemp, jute, straw, wood fibre, rice husks, wheat, barley, oats, rye, cane (sugar and bamboo), grass reeds, kenaf, ramie, oil palm empty fruit bunch, sisal, coir, water hyacinth, pennywort, kapok, paper-mulberry, raffia, banana fibre, pineapple leaf fibre and papyrus (Taj *et al*., 2007). Agro based lignocellulosic byproducts such as rice husks, wheat straw have been used as raw material for production of bioethanol, chemicals, enzymes, proteins, pharmaceuticals and fibre reinforced polymer composites. The various applications of lignocellulosic materials depend on their chemical composition and physical properties. Cellulose is the main structural component that provides strength and stability to the plant cell walls and the fibre (Paster *et al*., 2003). For example, fibres with higher cellulose content would be preferable for textile, paper and other fibrous applications, whereas byproducts with higher hemicellulose content would be preferable for producing ethanol and other fermentation products because hemicellulose is relatively easily hydrolysable into fermentable sugars (Reddy and Yang, 2005). Mechanically, hemicellulose contributes little to the stiffness and strength of fibres or individual cells (Thompson, 1993). Hemicellulose is more easily hydrolyzed into sugars than cellulose and therefore fibres containing a higher proportion of hemicellulose would be preferable for producing sugars, and eventually for fuels such as ethanol. Hemicellulose is the cell wall polymer with the highest water sorption (Madsen, 2004). This is responsible for the high moisture absorption of natural fibre leading to swelling and presence of voids, which results in poor mechanical properties and reduces dimensional stability of composites (Maya and Sabu, 2008) and microbial corrosion. Water sorption can be reduced by chemical treatment of natural fibre before composite manufacture through substitution of less polar groups for the hydroxyl groups, and this is the underlying principle in acetylation (Rowell, 1986), silanization (Gassan and Bledzki 1999) and formaldehyde treatment (Hua *et al*., 1987). The utilization of lignocellulosic materials is heavily dependent on the lignin content. Lignin provides plant tissue and individual fibres with compressive strength and stiffens the cell wall of the fibres to protect the carbohydrates from chemical and physical damage (Saheb and Jog, 1999). In natural fibre reinforced composite, lignin provides flexibility but in pulping/paper making and bioethanol production, it is a limitation. In the latter applications, lignin affects hydrolysis and would require extensive and expensive delignification if present in large amounts. Lignocellulosic materials with low lignin and high cellulose contents are suitable for pulp making, textile and bioethanol production. This study will explore some of the opportunities and limiting factors that exist in the use of plant fibres from *A. cavicaulis* in composites reinforcement and how the physico-chemical properties (cellulose, hemicelluloses, lignin, ash content, %extractives and some mechanical parameters) can determine its use.

## MATERIALS AND METHOD

**Preparation of Raw material**: The raw material was obtained from Ebonyi state in South Eastern Nigeria and identified as *Ampelocissus cavicaulis* at Bioresources Development and Conservation Programme (BDCP) Research Centre, Nsukka Nigeria. Natural plant fibres were extracted from the stem of *A. cavicaulis* using natural water retting extraction process, giving fibres of different lengths and diameters. Fibres were visually selected in order to verify the absence of defects along the length before tensile testing. The retted fibres were subsequently milled, the residual phytochemicals determined after which the milled sample was pre treated to remove non structural components, using Soxhlet technigue with n-hexane/methanol as the solvent systems.

**Determination of Chemical Composition:**

Phytochemical analysis was determined using the method by Harborne, 1998. The moisture, ash, %extractives contents were determined by the ASTM standard methods. The Kurschner-Hoffer cellulose method was used for the cellulose while the standard method of TAPPI (1998) (acid-insoluble lignin in wood and pulp-T22 om-98, 169) was used for the Klason lignin content. The hemicellulose content was determined using the Neutral detergent fibre method of Goering and Van Soest (1975). Acid soluble lignin and its derived products- Vanillin, p-Coumaric and ferulic acids in the acid hydrolyzate were determined at different wavelengths $\lambda$ using UV-Visible spectroscopy as described by Hyman *et al.*, 2007.

**Determination of Mechanical Properties:**

The mechanical properties of *A. cavicaulis* were determined according to the ASTM (American Society for Testing and Materials) standards. Hounsfield Tensometer testing machine (model 5566) was used to determine the tensile strength, Young’s modulus and elongation at break. An electronic digital caliper of linear capacitive measuring system was used to measure the diameter of the fibres.

## RESULTS

**Figure 1**: shows morphology of water retted fibres from *A. cavicaulis.*

Figure 1*: A. cavicaulis* bast fibre

**Table 1: Residual Phytochemical Composition of Untreated Fibre after Water Retting**

| **Phytochemical** | **Amount** | **Composition %w/w** |
|---|---|---|
| Alkaloids | ++ | 6.40 ± 0.28$^{\Delta}$ |
| Flavonoids | - | n.d |
| Tannins | + | 0.01 ± 0.002$^{\Delta}$ |
| Steroids | + | 0.03 ± 0.004$^{\Delta}$ |
| Free Reducing sugar | - | n.d |
| Glycosides | +++ | 64.566 ± 2.23$^{\Delta}$ |
| Proteins | + | 0.01 ± 0.003 |

**Key:** +++ = strongly present + = moderately present **-** = absent n.d= not determined
$^{\Delta}$Values expressed as Mean±S.D after triplicate analyses

**Table 2: Extractive Content (%w/w) of *A. cavicaulis***

| **Extractive** | **Amount (%w/w)** |
|---|---|
| Lipophilic | 1.96±0.02 |
| Alcohol | 9.21±2.35 |
| Hot Water Soluble | 12.24±3.11 |

**Table 3: Chemical Composition of *A. cavicaulis***

| Parameter | Amount (%w/w) |
|---|---|
| Cellulose | 38.66 ± 1.15 |
| Hemicellulose | 25.906 ± 1.29 |
| Acid Insoluble Lignin | 33.21 ± 2.76 |
| Ash | 2.88 ± 0.90 |
| Moisture | 5.60 ± 0.07 |
| Acid Soluble lignin $\lambda_{205}$ | 2.166 ± 0.01 |
| Vanillin $\lambda_{230}$ | 1.35 ± 0.022 |
| p-Coumaric acid $\lambda_{308}$ | 0.03 ± 0.022 |
| Ferulic acid $\lambda_{322}$ | n.d |
| Cellulose:Lignin ratio | 1.16 |
| | |

**Table 4: Mechanical Properties**

| Parameter | Value |
|---|---|
| Diameter (mm) | 0.92±0.003 |
| Ultimate Tensile Strength (MPa) | 238.28±4.56 |
| Ultimate Young's modulus (GPa) | 3.97±1.21 |
| Ultimate Elongation@break (%) | 3.00±0.60 |

## DISCUSSION

Biochemical composition affects the mechanical and physical properties of fibre, fibre interaction with composite matrix in a biocomposite material and ultimately, fibre performance or quality for a given application. Lignocellulosic biofibres were extracted from young stems of *A. cavicaulis* through natural water retting process and the resulting fibres were uniform and almost circular cross sections. The results obtained from the phytochemical analysis of untreated *A. cavicaulis* plant fibre in table 1 showed the presence of glycosides in great quantity, moderate presence of steroids, proteins, tannins and alkaloids and total absence of flavonoids, and reducing sugar. Further analysis showed that the plant fibre contains alkaloids (6.40 ± 0.28%), tannins (0.0082 ±0.0015%) and steroids (0.0333± 0.004%). The pre treatment of the fibre using different solvent systems showed the presence of %w/w alcohol extractives (9.21%) and %w/w of lipophilic extractives (11.17%). The chemical characterization of *A. cavicaulis* fibre as shown in table 3 revealed that it contains cellulose (38.66±1.15%), acid insoluble (Klason) lignin (33.211±2.969%) and acid soluble lignin (1.466± 0.013%). It also revealed that the fibre contains ash (2.88 ±0.90%)

and moisture (5.60±0.071%). The hemicellulose content was found to be 25.906 ± 1.294%. The cellulose to lignin ratio was found to be 1.16.

The physical and mechanical characterization of the plant fibre as shown in table 4 indicates a diameter of 0.9174mm, tensile strength of 238.28MPa, Young's Modulus of 3971.33MPa and elongation at break of 3.0%. The cellulose content of *A. cavicaulis* fibre is comparable to that of jute (58-60%), sisal (53-66%) and hemp fibre (55-72%) (Bledzki *et al*., 2002).

Its cellulose content is higher than that of Norway spruce (49%), barley straw (43%) and corn stover (33%). The hemicellulose content of *A. cavicaulis* fibre is lower than that of flax (16%) (Kirk and Sawyer, 1998), jute (14-16%), ramie (13%), barley straw (38%), corn stover (33%), sisal (12%), Norway spruce (20%) and cotton (12%) (Han and Rowell, 1996); but is comparable to that of hemp fibre (7-19%). Fibres from hemp, jute, barley straw, corn stover and sisal have higher ash content of 4%, 8%, 5%, 7% and 7% respectively (Kirk and Sawyer, 1998) than that from *A. cavicaulis*. However, the ash content of *A. cavicaulis* fibre is higher than that of Norway spruce and cotton. *A. cavicaulis* has lower extractives (2.33%) compared with fibres from barley straw (5%), corn stover (10%). The acid insoluble (Klason) lignin content of *A. cavicaulis* plant fibre is higher than that of flax (2.9%), kenaf (11.4%), hemp (4.6%), jute (13.3%), sisal (5.9%), abaca (7.7%) and curaua (4.9%). On the other hand, the acid soluble lignin content of *A. cavicaulis* fibre was found to be lower than that of flax (1.6%), kenaf (3.0%), hemp (1.5%), jute (2.8%), sisal (3.0%), abaca (1.4%) and curaua (1.6%). The ratio of cellulose to lignin of *A. cavicaulis* plant fibre was found to be below 2.0 indicating that it may not be suitable for pulp and paper making (Judt, 2001; Ilvessalo-Pfaffli, 1995; Omotoso and Ogunsile, 2009) except with extensive delignification and severe pulping conditions. The high cellulose content of the fibre depicts its high tensile strength (238.28MPa) and as such it will be good in reinforcement technology. In addition its crystalline cellulose confers mechanical stability, rigidity and elasticity to plant (Neville, 1993). Owing to its low moisture content, it may be useful in the textile and automobile industries. The low hemicellulose content of A. cavicaulis implies that its water absorbing capacity will be low (Bakker *et al.*, 2004; Jensen *et al.*, 2001). This particular property reaffirms its use in reinforcement technology. Its low water retention capacity decreases the effect of microbial corrosion. To improve the fibre quality for biocomposite production, it must be chemically modified using silane and alkaline treatment of the hydroxyl groups of cellulose and hemicellulose. *A. cavicaulis* plant fibres are flexible with high elongation point compared to some natural fibres such as flute and sisal which will fracture, when processed over sharp curvature (Judt, 2001). This enables the fibre to maintain the desired aspect ratio for high performance in plastic and textile industries. Plant fibres are renewable resource with production requiring little energy and are biodegradable. They are environmentally friendly since they do not return excess carbon dioxide into the atmosphere when they are composted. This particular property of plant fibres is attributed to the presence of their biocomponents which include cellulose, hemicellulose and lignin which have the necessary functional groups that can enable micro-organisms to degrade them easily. *A. cavicaulis* plant fibre has this property and so when used in the production reinforced materials such as floor tiles, gas cylinder, hot water tank, inner panel of cars, or when delignified for used in pulp making, it will not cause environmental pollution. This is unlike synthetic fibres which are not biodegradable, which usually results to environmental pollution when any of its products is disposed. Plant fibres have low density, so when used in the construction of car parts such as the door panel and roof will lead to reduction in the amount of fuel consumed since it would require little energy to propel a lighter object than a heavier one.

Plant fibres such as flax, jute, sisal, hemp and jute are known for their low price due to their world wide availability. In addition, they have fewer requirements for equipment during reinforcement which reduces cost unlike their synthetic counterpart.

The mechanical parameters of *A. cavicaulis* revealed that it has both high tensile strength and Young's modulus. Since extensibility is directing proportional to the lignin content of a plant fibre, *A. cavicaulis* plant fibre with high lignin content will have increased extensibility. Hence, it will be useful in reinforcement and textile industries. In addition, *A. cavicaulis* is flexible unlike its synthetic counterpart; as a result it will not break when subjected over sharp curvature. Therefore, the fibre will maintain the desired aspect ratio for in plastic and textile industries (Zeronian *et al.*, 1990).

The awareness to develop plant fibre is currently gaining ground, although the ecological aspects of using natural fibres which were the initial reason for their slow place is beginning to ease off.

The trend is now towards increasing performance so that the gap of synthetic products will become very small. Cultivation of fibres especially for technical purpose, continued development of fibre preparation methods, and new processing methods will further improve the properties. To fully realize the ecological advantages it is absolutely necessary to develop matrix materials based on renewable resources.

## CONCLUSION

This study revealed that *A. cavicaulis* plant fibre has potentials in composites reinforcement technology for both structural and non structural applications due to its cellulose and lignin contents which are measures of plant fibre tensile strength and stiffness. Above all, the fibre is environmental friendly unlike synthetic fibres.

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