Asian Journal of Chemical Sciences

10(2): 23-31, 2021; Article no.AJOCS.70943
ISSN: 2456-7795

Physicochemical Evaluation of *Eichhornia crassipes* and *Pennisetum purpureum* used for Production of Drilling Mud Additives

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Authors' contributions

This work was carried out in collaboration among all authors. Author LCO designed and supervised the research, author IUE wrote the first draft of the manuscript, was involved in field sampling and laboratory analysis of data; author MCO wrote the introductory section, managed the analyses of the data to the point of publication. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AJOCS/2021/v10i219087
Editor(s):
(1) Prof. Pradip K. Bhowmik, University of Nevada, USA.
Reviewers:
(1) Kamal I. Aly, Assiut University, Egypt.
(2) Anthony Chukwunonso Opia, Universiti Teknologi Malaysia, Malaysia.
Complete Peer review History: https://www.sdiarticle4.com/review-history/70943

Original Research Article

Received 13 May 2021
Accepted 18 July 2021
Published 27 July 2021

ABSTRACT

Cellulose isolated from *Eichhornia crassipe* (Water hyacinth) and *Pennisetum purpureum* (elephant grass) were evaluated using Fourier Transform Infra-Red spectroscopy and standard analytical methods for production of drilling mud. The physico-chemical analyses were carried out after chlorination and alkaline process using sodium chlorite and sodium hydroxide for the extraction of cellulose from the two biomass samples under same experimental conditions. Results of physico-chemical analysis of *Eichhornia crassipes* showed pH: 7.30; conductivity 0.028; bulk density 0.1097g/ml. *Pennisetum purpureum* showed pH: 7.50; conductivity 0.192; bulk density 0.1378g/ml. *Pennisetum purpureum* has a higher cellulose yield of 31.39% compared with *Eichhornia crassipes* with a percentage cellulose yield of 21.88%. Both biomass samples have Herzberg strain of Violet-blue. The results of the Fourier Transform Infra-Red spectroscopy showed prominent peaks at 3353-3164, 1655, 1629, 1320, 1033 and 1019 cm^{-1}. The broad absorption bands around 3353-3164

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cm^{-1} indicated stretching of –OH groups due to inter-molecular and intra-molecular hydrogen bonds of polymeric compounds. The sharp bands at 1655 cm^{-1} and 1629 cm^{-1} showed C=C stretch of aromatics. The sharp absorption bands observed at 1320, 1019 and 1033 $cm^{-}1$ were characteristic of C—O stretch and C—O—C asymmetric stretch of cellulose. The FTIR results proved that the products extracted from the two samples were aromatic hydroxyl compounds. The results of the physicochemical analyses showed that cellulose isolated from the biomass samples which are persistent noxious weeds that invade the aquatic and terrestrial environment can be utilized in industrial applications for drilling fluid production.

Keywords: Drilling mud; Eichhornia crassipes; Pennisetum purpureum; renewable resource; cellulose.

1. INTRODUCTION

Different chemicals and polymers are used as additives in formulating drilling muds that help in improving their functional requirements [1]. Cellulose is one of the few natural polymers that can be utilized. Production and utilization of cellulose materials indicate one of the great and major sources of national income. Several industries today owe their very existence to scientific discoveries concerning the basic chemical reactions of cellulose. These include the conversion of cellulose into cellulose ethers and esters, which are utilized for plastics, transparent sheeting, fabrics, cosmetics and pharmaceuticals. Cellulosic plant material has been included in certain oil and gas fluids [2]. Cellulosic ether derivatives such as carboxymethyl cellulose (CMC) are known to be useful additives of down-hole fluids used for oil and gas production. These materials are added to drilling fluids to provide a large particle bridging sealant which alone or in combination with smaller particles plugs porous and fractured formations. Cellulose is a chemical substance obtained mainly from many different botanical sources. These botanical sources include woody matter, cotton fibers, jute fibers, straw, stalks, hulls, and even marine plants [3].

Since cellulose is the major constituent of the cell wall of plants, it is important that those plants which have no industrial utilization, as well as the residual wastes from utilized plants should form a major potential source of cellulose. The most important of these wastes and residues, as far as their present utilization is concerned, belong to the grass family. Cellulose is not confined to the vegetable kingdom but also found in the mineral and animal kingdoms [4].

Technologically, cellulose is obtained by subjecting biomass materials to certain pulping processes. The pure cellulose which may be used for experimental studies was obtained from raw biomass after fat, wax, pectin and other soluble impurities have been removed by extraction with organic solvents. The residue obtained was freed from other non-cellulosic substances (hemicellulose and lignin) by careful treatment with dilute alkali, washing and bleaching with hypochlorite.

The content of cellulose can differ with the pulping process and with the source of material used. Chemically, cellulose is a polysaccharide of sufficient chain length which is insoluble in water or in dilute acids and alkalis at ordinary temperatures. The chemical formula for cellulose is $(C_6H_{10}O_5)_n$ consisting of anhydroglucose unit linked together through the 1 and 4 carbon atoms with a β-glucosidic linkage [4,5].

The chemical structure of cellulose offers the advantage of it being modified such that new characteristics can be achieved for several industrial applications [6]. The hydroxyl groups which appear on the surface of cellulose make it possible for it to undergo various chemical modifications such as esterification, etherification, oxidation, and reduction reactions [7].

The current trend in the drilling fluid development is to come up with drilling fluids that will replace the present day imported drilling fluids in terms of cost, performance and efficiency [8]. Non-woody biomasses are the most abundant resources for cellulose production. They are very promising alternative materials because they are economical and renewable raw materials that have been shown to exhibit equal or better physical and mechanical properties comparable to some commercial wood species [9]. Therefore, it is very important to establish sustainable, cost effective and renewable raw materials for cellulose production.

Biomass is described as any organic matter obtained from renewable sources such as agricultural, animal and manufacturing sectors

[10]. It contains organic carbon, hydrogen, oxygen and nitrogen (C, H, O and N) and its components are identical to carbon and hydrogen fossil feed stocks. (C and H) and applied in combustion operation [10,11]. The comprehensive use of agricultural products from land, marine and waste recycling to bio-energy, lubricants and additives, chemical products and other materials attracts considerable attention to bio-researchers [12,13]. The increasing interest in research is based on the global substitution of petroleum lubricants with bio-lubricants and additives, because of the growing awareness on the harmful effects and emissions by petroleum source to the global environment of the greenhouse gas impact [14]. Non-woody wastes such as Water hyacinth (*E. crassipes*) and Elephant grass (*P. purpureum)* were considered and used in this research. They offer numerous advantages such as being abundantly available, high specific mechanical properties, low cost, low density, safe to handle, environmentally friendly, renewable and biodegradable [15,16]. They are also persistent weeds considered as invaders which can be useful for industrial application to reduce their negative impact on the environment. *E. Crassipes* are active green substitutes for a poisonous inorganic substance such as sulfur (S) and phosphorus (P) compounds that are treated with certain excellent potentials. It is also able to synergistically improve other standard additives [17].

This research intends to use *P. purpureum* and *E. crassipes* (Plates 1 and 2) to generate cellulose which can be utilized in production of environmental friendly drilling fluid additives to minimize the cost of drilling operations.

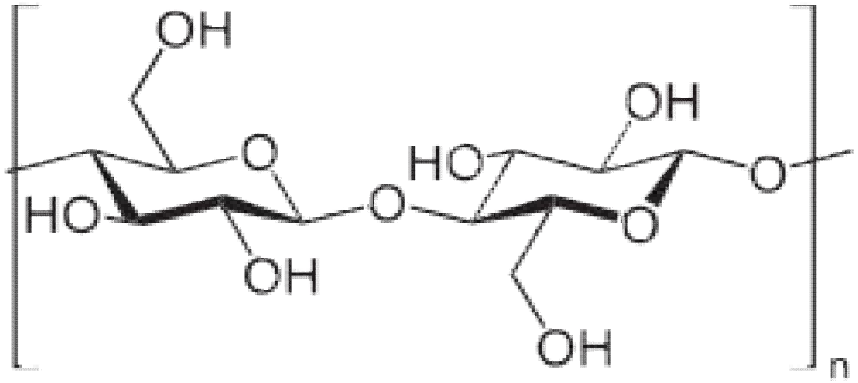

Fig 1. The structure of cellulose

Plate 1. *Pennisetum purpureum* (Elephant grass)

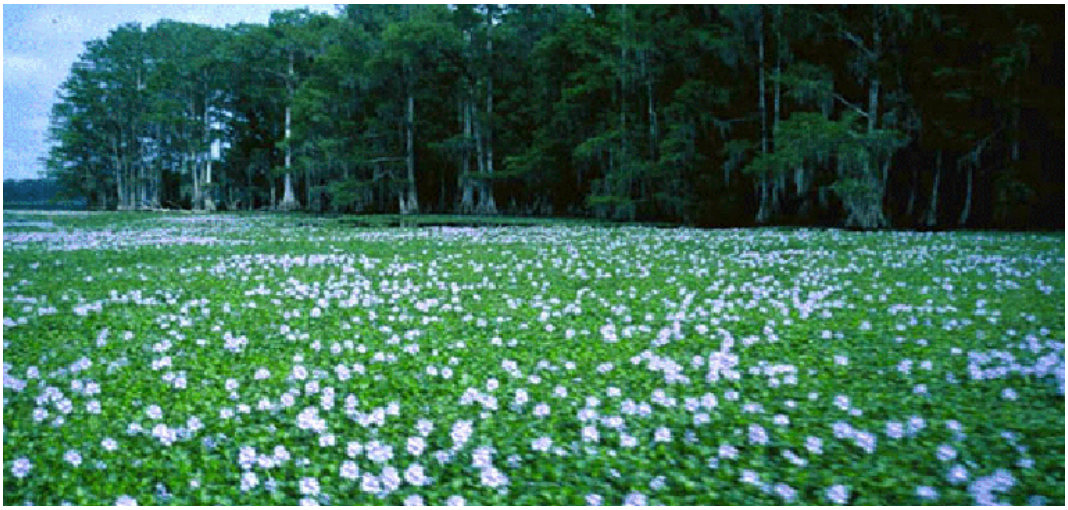

Plate 2. *Eichhornia crassipes* (Water hyacinth)

2. MATERIALS AND METHODS

2.1 Sample Preparation

P. purpureum and *E. crassipes* were collected from Port Harcourt, Rivers State and Gbaran, Bayelsa State respectively. The samples were taken to the plant science and biotechnology laboratory of the University of Port Harcourt for proper identification by experts. These samples were washed several times with distil water to remove extraneous impurities and thereafter sun dried for a week. The dried samples were cut into small pieces, ground with a blender and kept in a hot air oven for 24hrs at 105°C to remove the moisture.

2.2 Extraction of Cellulose

Twenty (20) grams of each samples were weighed into a round bottomed flask, placed on a water bath, 70% ethanol was added, stirred, heated and refluxed for 120 mins at 85°C. The fluids were drained and residues rinsed with 50% ethanol to complete the degreasing processes. At this stage pectin, fats, waxy matters, and aqueous extracts were separated from the residues.

The dewaxed elephant grass and water hyacinth samples were treated three times each with 17.5% NaOH solution at 50 ºC for 60 mins in a fiber to liquor ratio of 1:20 with occasional stirring to remove the hemicelluloses. The mixtures were filtered and the residues washed in 8.5% NaOH solution using a fiber/liquor ratio of 1:20. These were also filtered and washed with 5% acetic acid, tap water and subsequently with distilled water to neutralize the reaction. The residues were then bleached twice with 2% sodium chlorite ($NaClO_2$) for 120 mins at 50 ºC in an acidic solution (pH 4 - 4.2 adjusted by the addition of acetic acid-sodium acetate buffer) using fiber/liquor ratio of 1:20. The pulps were separated from the filtrate and washed severally with tap-water followed with distilled water. At this stage, rich content of cellulose were separated by eliminating hemicellulose and lignin from dewaxed samples. The extracted celluloses were filtered and dried in an oven at 105 ºC until constant weights were obtained. The cellulose yield was gravimetrically determined as the weight of the dried cellulose to that of the dried initial sample and samples kept in a desiccator for further studies.

2.3 Fourier Transform- Infra Red (FT-IR) Analysis

The Fourier Transform- Infra Red (FT-IR) analysis of all samples was carried out using Agilent FT-IR spectrophotometer. The infrared absorption spectra were recorded in the 4000-650cm^{-1} region with 32 scans in each case at a resolution of 4$cm^{-1}$and compared with a Reference Spectrum.

2.4 Chemical Identification of Cellulose

Staining analytical methods are simple, rapid, low-cost, available, and are the most frequently used in cellulose identification. The two cellulose

types were stained according to standards with zinc chloride-iodine stain (Herzberg stain). 10 mg of the cellulose powder was dispersed in 2ml of staining solution which was prepared by dissolving 10 g of zinc chloride, 3.25g of potassium iodide in 5.25 ml of water with addition of 0.25 g iodine and mixed vigorously for 15 minutes.

2.5 Conductivity and pH

Five grams (5g) of the samples were mixed with 40 ml of distilled water for 20 minutes, and centrifuged. The supernatant liquid was used to measure the conductivity and the pH of the two samples using well calibrated conductivity and pH meters respectively.

2.6 Bulk Density

Bulk density was calculated by dividing the weight of the powder in a stainless steel cup by the volume of the cup, which was calibrated to a capacity of 25.0 ± 0.05 ml and has an inside diameter of 30.0 ± 2.0 mm.

3. RESULTS AND DISCUSSION

Isolation of cellulose from elephant grass (*P. purpureum)* and water hyacinth (*E. crassipe)* was performed by extraction with alkaline, sodium hydroxide and bleaching agent, sodium hypochlorite. The cellulose extracted from the biomasses was studied using Fourier Transform Infra-Red (FT-IR) spectroscopy and physico-chemical analysis. The results from Table 1 showed that *E. crassipes* had a cellulose yield of 21.88% with a bulk density of 0.1097g/ml and of pH 7.3 while *P. purpureum* had a higher yield of 31.39% and a higher bulk density of 0.1378g/ml and pH of 7.5. The resulting products from extraction had brown colors (Plates 2a and 2b) and on treatment with Herzberg stain turned violet-blue color indicating the presence of cellulose. The brown colour could indicate the presence of some impurities and may probably require more bleaching.

The results of the physicochemical parameters showed high percentage yield of cellulose from the two samples making them good renewable resources for cellulose production. Both samples have low bulk densities which show high porosity. Cellulose extracted from elephant grass has a higher bulk density of 0.1378g/ml than water hyacinth with bulk density of 0.1097g/ml. This indicates that the cellulose from elephant grass has higher compressibility, lower porosity and a lower ability to absorb water or oil than cellulose obtained from water hyacinth, therefore may be a better filtration control agent in the production of drilling mud additive [18]. The pH values obtained from the two products which are slightly alkaline make the products environmentally friendly, safe for the workers and will reduce corrosion on drill pipes which is caused by acidic fluids if utilized in drilling mud additives production [18]. The low electrical conductivity values show that both cellulose produced are insulators and are free from inorganic impurities. This is also a safety property for drilling mud additive production. The organoleptic tests show that the cellulose produced is brown in colour, odourless, tasteless and insoluble in water and in organic solvents. The brown cellulose obtained can be used in drilling mud additives formulation without any further refinement but higher purity may be required for food and pharmaceutical industries. The insolubility of the products shows that they require modification before they can be properly utilized in oil and gas industry [19].

The result of the FT-IR spectrum for *P. purpureum* (Fig. 1), showed strong bands at 3286, 2341, 1320, 1160, 1090, 992, 899, 847, 814 and 676 cm^{-1}. The broad band at 3286 cm^{-1} indicated the intermolecular O—H bond of polymeric compounds while the observed band at 2341 cm^{-1} indicated C—H stretch and C—H deformation in cellulose [20]. The absorption at 1320 cm^{-1} was as a result of C—O stretch in cellulose. The absorption bands at 1160 and 1090cm^{-1} were C—O—C asymmetric stretching and C—O, C==C and C—C—O stretching bonds in cellulose [21]. The absorption band at 992 cm^{-1} was due to C—O valence vibration while 899 cm^{-1} indicated C—O and C–H deformation and stretching vibrations of cellulose [22]. The band at 899 cm^{-1} also showed the amount of amorphous region in the cellulose and β-glucosidic linkages between the sugar units in the sample [23,24]. The absorption bands around 847-676 cm^{-1} indicated C—H out of plane bending on aromatic ring.

FT-IR spectrum for *E. crassipe* showed *bands* at 3245, 2884, 1665, 1629, 1320 and multiple bands around 1153-1033 cm^{-1} indicated hydroxyl aromatic compound. The observed peak beyond 3000 cm^{-1} was characteristic of OH group while the band at 2884 cm^{-1} stands for methylene (CH_2) group of cellulose [25]. The sharp bands at 1655 cm^{-1} and 1629 cm^{-1} showed C=C stretch of

aromatics and O-H bending from absorbed water. The sharp transmittance peaks observed at 1320 cm^{-1} and the multiple bands at 1153, 1104, 1052, and 1033 were characteristics of C—O stretching and C—O—C—O—C bonds in cellulose respectively [25].

Table 1. Results of the physicochemical analyses of water hyacinth and elephant grass cellulose

Parameters	Elephant grass *(P. purpureum)*	Water hyacinth *(E. crassipe)*
pH	7.50	7.30
conductivity (S/cm)	0.192	0.028
Cellulose (%)	31.39	21.88
Bulk density (g/ml)	0.1378	0.1097
Herzberg stain	violet-blue	violet-blue

Plate 2a. Cellulose obtained from Elephant grass

Plate 2b. Cellulose obtained from Water hyacinth

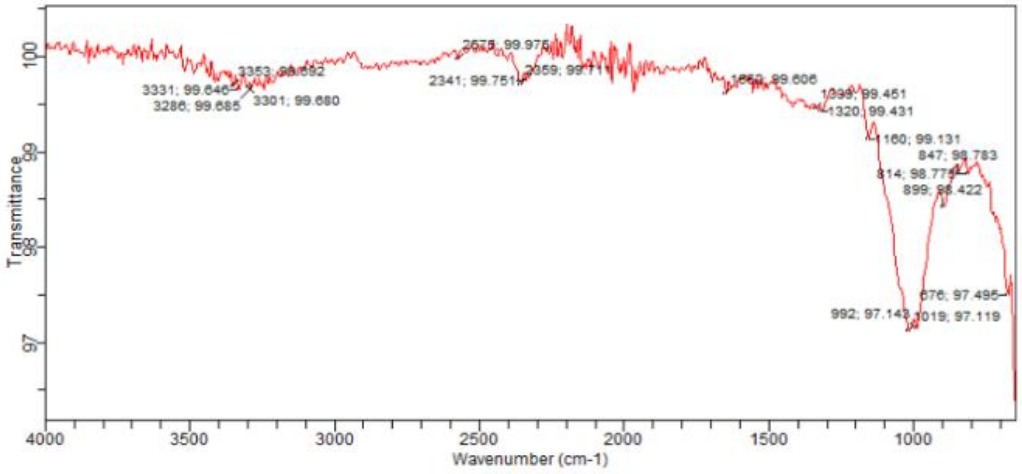

Fig. 1. FT-IR spectrum Elephant grass (*P. purpureum*)

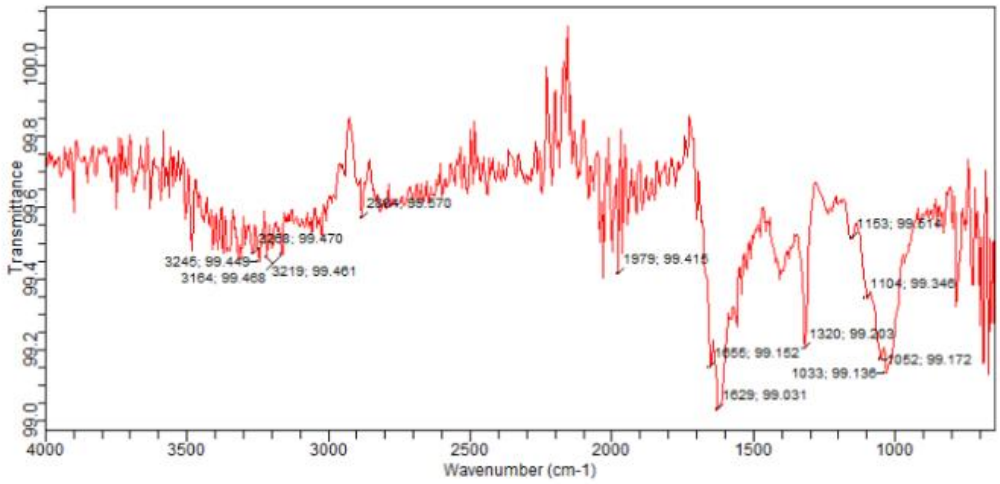

Fig. 2. FT-IR spectrum Water hyacinth (*E. crassipe*)

Lack of peaks between 1750 and 1700cm^{-1} showed that the products were free of the carbonyl groups of carboxylic acid, aldehydes and ketones [26]. Other reduced bands from the two spectra showed reduction from impurities associated with the samples after sodium hydroxide and sodium chlorite treatments. Thus the physico-chemical and FT-IR results showed that the extracted compound from elephant grass (*P. purpureum)* and water hyacinth (*E. crassipes)* were cellulose.

4. CONCLUSIONS

The presence of water hyacinth (*E. crassipes*) as an aquatic plant and elephant grass (*P. purpureum)* as terrestrial weed cause many environmental problems. This research utilized these renewable, persistent, low cost and noxious weeds as a source of cellulose that can be modified into use in the oil and gas industry for production of drilling fluid additives thereby reducing the associated problems with these biomasses. The FT-IR and physico-chemical studies showed that cellulose was successfully isolated from water hyacinth (*E. crassipes*) and elephant grass (*P. purpureum)* by alkaline-chlorination process. Chemical analysis indicates that *P. purpureum* has a higher cellulose yield than *E. crassipes*. The high cellulose yields from these readily available, low cost and renewable non-woody biomasses show that they are good

alternative resources for cellulose which can be utilized for further studies. Based on the properties determined for alkali-chlorine treated elephant grass and water hyacinth, we expect that these fibers will be suitable for use as additives in drilling mud production.

ACKNOWLEDGEMENTS

The authors are grateful to Mr Muhammadu Ya Muallim of Chemistry Laboratory Bayero University Kano, Mr Umar Faruk Lawal of Senior Secondary School Rafindadi Kastina and Mr. Peter of Central Instrument Laboratory University of Port Harcourt.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Peer-review history:
The peer review history for this paper can be accessed here:
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