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Research Article

COMPARATIVE STUDIES ON PHYSICOCHEMICAL PARAMETERS, MINERAL CONSTITUENTS AND MORPHOLOGIES OF CELLULOSE FROM *CARICA PAPAYA* AND *GLIRICIDIASEPIUM* STEMS

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ABSTRACT

Plant stems of *Carica papaya* and *Gliricidiasepium* collected within the Ado-Ekiti metropolis, Nigeria, have been pulped using 17.5% NaOH under heating to obtain cellulose. The resultant products were bleached, washed and oven-dried to obtain *Carica papaya* cellulose (CPC) and *Gliricidiasepium* cellulose (GSC). The functional properties of CPC and GSC were analysed, and the results were compared. The samples were subjected to instrumental analysis using Fourier Transform Infra-Red (FTIR) for functional group determination and Scanning Electron Microscopy (SEM) for morphologicalassessment.CPC and GSC were also analysed using standard analytical procedures for the possible presence of heavy metals. The results showed that the values of some functional properties such as ash content, tapped density, and bulk density for CPC were higher than those of GSC. The SEM showed that CPC appeared as a cluster of tiny, round, and smooth entities while a thread-like pattern was observed for the GSC surface. Elements such as Zr, Cr, Cu, Ni, and Pb were detected in CPC, while the same elements except Pb were detected in GSC.

Keywords: *Carica papaya*, *Gliricidiasepium*, cellulose, heavy metals, pulp, morphology.

INTRODUCTION

*Carica papaya*or papaya plant mostly thrives in the tropicsor sub-tropical regions where it is cultivated for its fruits. Brazil is its second largest abode, according to the report of Evans and Ballen (2012). *Carica papaya* has been tagged as a giant herb which does not possess lignified xylem as generally found in trees. The plant can grow up to 10m high (Kempe *et al.,* 2012). *Gliricidiasepium*are leguminous and perennial trees found in the Caribbean, Asia, Africa, and tropical America. They are woody, having nodules and deep roots. The trees shed leaves during the flowering period (Heuze and Tran, 2015) and are easily propagated through either seed or stem (Abe *et al.,* 2018). Cellulose, a naturally inexhaustible polymer with outstanding features (Klemm *et al.,* 2005), is readily available as a natural polymer (Eichhorn *et al.,* 2010; Hon and Shiraishi, 1990). It has been used in industries for numerous applications since the ancient period (Eichhorn *et al.,* 2010; Sørensen*et al.,* 2010). Cellulose crystallinity is very high due to numerous hydrogen bonds embedded in its hydroxyl groups; its primary sources include hemp, jute, cotton, flax, etc. (Yuvraj *et al.,* 2009). It is a major component of plant tissues and is also found in fungi, bacteria, algae, and animals (Hon and Shiraishi, 1990). This organic material is a viable raw material for economic growth as it can be obtained from renewable sources; its unique characteristics, such as being non-toxic, easy to dispose of after use, as well as excellent mechanical properties, make it to be useful for specific purposes (Klemm *et al.,* 1998). Cellulose has evolved in multiple species over time. As against other major components of plants, such as lignin and hemicelluloses, cellulose chemical structure has been unchanged for ages (Domozych *et al.,* 2012). The availability or abundance of cellulose is not the same in all plants. However, it is often found within the range of 30-40 wt % for trees (Yinon *et al.,* 2018), ranking it the most commonly available biopolymer on the planet (Ververis *et al.,* 2004).

No doubt, cellulose has become a common name in the industrial world. Getting alternative sources other than the conventional ones is one of the targets of this study. *Carica papaya* is known for its plump and delicious fruits. However, its stem is not documented to be useful as it gets decayed as agricultural waste over some time. Also, *Gliricidiasepium* is a wide plant that can easily be cultivated; it thrives so well over a period of time and can withstand harsh weather. This study aims to extract cellulose from these two agricultural waste materials, thereby converting hitherto waste materials into valuable industrial intermediates. Several reports have been on the synthesis or extraction of cellulose from different lignocellulosic materials. However, scanty information have only been reported on *Carica papaya* and *Gliricidiasepium*as sources of cellulose. So, this study compares the physicochemical parameters, functional properties, morphologies, and the mineral composition of their respective cellulose matters.

METHODOLOGY

Isolation of Cellulose

The pulping was carried out according to the methods described by Jonoobi *et al.,* (2009) as modified by Abe *et al.,* (2018). Accurately measured 300 g of the plant stem already ground into powder was contacted with 3 L of 17.5% NaOH. The mixture was heated in a water bath at 90°C for three hours. The mixture was then subjected to a temperature of 100°C for one more hour. The resulting mixture was filtered and washed with 1M HCl to drastically reduce the concentration of NaOH used in pulping the cellulosic materials. The material was washed with distilled water until a neutral sample was obtained. Afterwards, the sample was squeezed with the help of muslin cloth to obtain a semi-solid material from the slurry. The obtained solid component was air-dried at ambient temperature. The resulting air-dried sample was subjected to a bleaching process using 3.5% NaOCl and left for 180 min. The bleached sample was washed severally with warm distilled water, filtered, and oven dried to give powdered cellulose. The cellulose obtained from the *Carica*

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*papaya*stem was tagged CPC, while the cellulose obtained from the *Gliricidiasepium* stem was tagged GSC.

Determination of functional properties of CPC and GSC

The functional properties such as ash content, tapped density, true density, Hausner's Index, moisture contents, and bulk density were determined according to the modified methods of Abe *et al.,* (2018) and Nwajiobi *et al.,* (2019).

Characterisation of cellulose

The obtained cellulose was characterised using Fourier-Transform Infrared Spectroscopy (FTIR) for the functional groups and Scanning Electron Microscopy (SEM) for the surface morphology.

Elemental analysis of CPC and GSC

A gram of each sample was accurately weighed and contacted with 10mL of aqua regia for wet digestion. The resulting mixture was transferred into a fume cupboard and subjected to heating using an electric hot plate until almost dry. Thereafter, 100mL of distilled water was added to the residue, stirred very well for easy dispersion, and filtered using filter paper. The obtained clear filtrate was analysed for heavy metals using Atomic Absorption Spectrophotometer(AAS).

RESULTS AND DISCUSSION

Functional properties of CPC and GSC

The results of the functional properties are presented in Table 1. The moisture content for CPC was 5.60%, while the moisture content for GSC was 8.40%.It was observed here that the moisture content of GSC was higher than that of CPC. Nwajiobi *et al.,* (2018) reported the moisture content of cellulose obtained from the papaya plant to be 7.54%. Moisture content is a function of how dried a powdered sample is after being air-dried, sun-dried, oven-dried, etc. A high level of moisture is one of the factors that aid the deterioration of many powdered samples, including natural polymers (e.g. chitosan. starch, and cellulose); the moisture content of below 10% is often an indication of good quality polymer (Szymanska and Winnicka, 2015). Ash content values for CPC and GSC were 11.3 and 10.6%, respectively. Ash content is the left-over after a sample has been charred at a high temperature (Rydholm, 1965); it is a way of burning out all the organic materials attached to the sample, leaving the inorganic contents such as silica and minerals. In this study, the ash content of GSC was less than the value obtained for CPC. The bulk density values for CPC and GSC were 0.49 and 0.20 g/cm^3. Bulk density and tapped density are a function of the compaction and compressibility of the powdered sample when confined (OgunjobiandBalogun, 2021). The value obtained for GSC was lower than that obtained for CPC. The result of bulk density for CPC was a bit higher than the value (0.219 g/cm^3) obtained by Nwajiobi *et al.,* (2018). The results were not far from the report submitted by Abe *et al.,* (2018), where the bulk density obtained for cellulose was 0.30 g/cm^3. A similar result was reported by Peter (2002). It was also reported that a low bulk density impacts good tablet-ability (Ohwoavworhua and Adelakun, 2010). The values of tapped density for CPC and GSC were 0.21 and 0.10 g/cm^3, respectively. The results showed that the value obtained for GSC was lower than that obtained for CPC. The tapped density value for CPC in this study fell within the range of 0.23 – 0.57 g/cm^3 for cellulose samples obtained from wild *Dioscoreabulbifera*peels collected from different locations by Ogunjobi andBalogun(2021).The Hausner index for CPC and GSC were 0.02 and 0.5. Here, the value for GSC was higher than the value for CPC. The value of the Hausner index for CPC in this study is less than 2.04, as obtained in a study by Nwajiobi *et al.,* (2018).

Parameters	CPC	GSC
Moisture content (%)	5.60	8.40
Ash content (%)	11.3	10.6
Bulk density (g/cm^3)	0.49	0.20
Tapped density (g/cm^3)	0.21	0.10
Hausner index	0.02	0.5

Characterisation of CPC and GSC

FTIR Analysis

The FTIR spectra of CPC and GSC are presented in Figures 1 and 2, and the extracted wavelengths from the spectra are presented in Table 2.The results revealed that the functional group, the OH-stretch of the alcoholic group, was observed at 3419.19 and 3458.21 cm^{-1} for CPC and GSC, respectively. C-H stretch of polysaccharides was noticed at 2929.17 and 2929.80 cm^{-1} for CPC and GSC, respectively. Song *et al.,* (2016) similarly reported that O-H and C-H stretching modes were observed at 3340 and 2907 cm^{-1}, respectively. The wavelengths observed at 1628.72 and 1639.90cm^{-1} for CPC and GSC indicated the presence of N-H bend. The wavelengths observed at 1383.00 and 1425.00 cm^{-1} indicated the presence of C-H bending modes for CPC and GSC; respectively, a similar finding was documented by Sun *et al.,* (2005). C-O stretch was observed at 1079.70 and 1052.21 cm^{-1} for CPC and GSC, respectively. N-H wag of primary and secondary amine was observed at 903.38 and 876.35 cm^{-1} for CPC and GSC, respectively. However, the N-O symmetry stretch, C-N stretch of aromatic amine, and C-N stretch of aliphatic amine found in CPC were conspicuously missing on FTIR spectra of GSC.

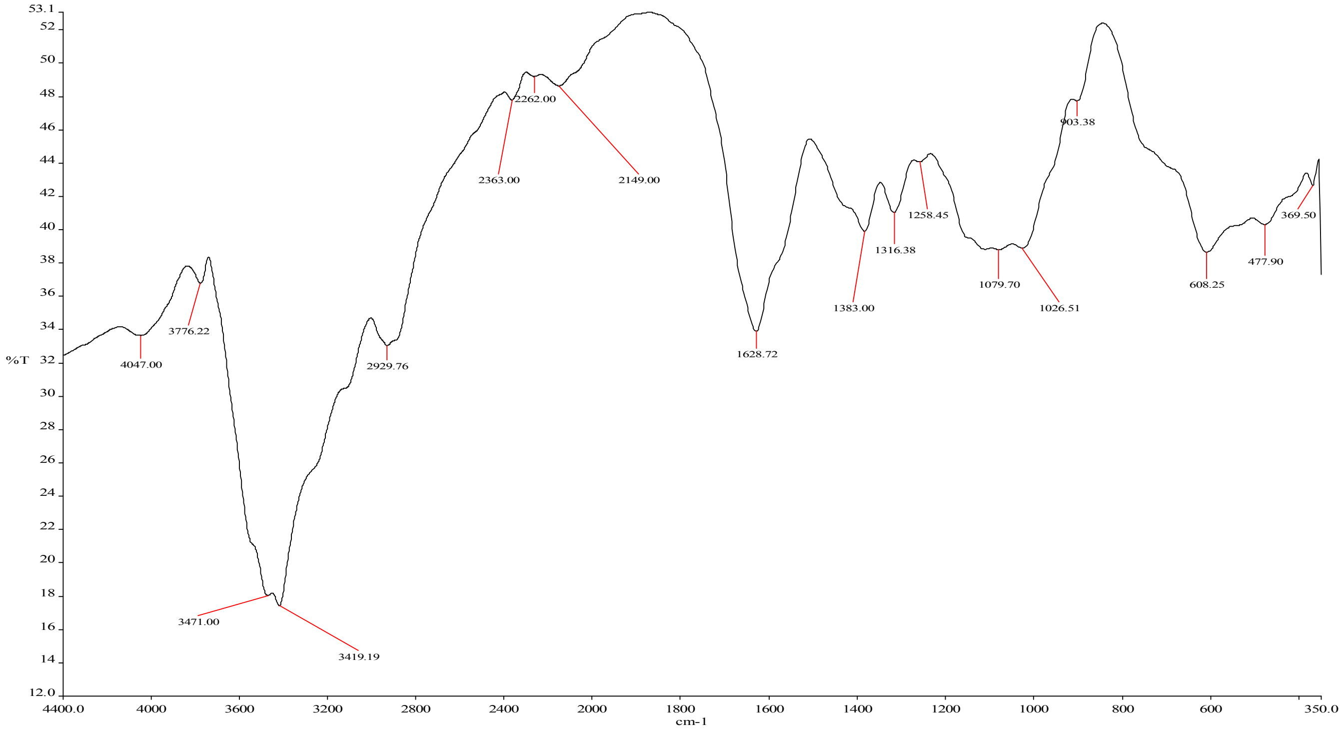

Fig.1: FTIR spectra of CPC

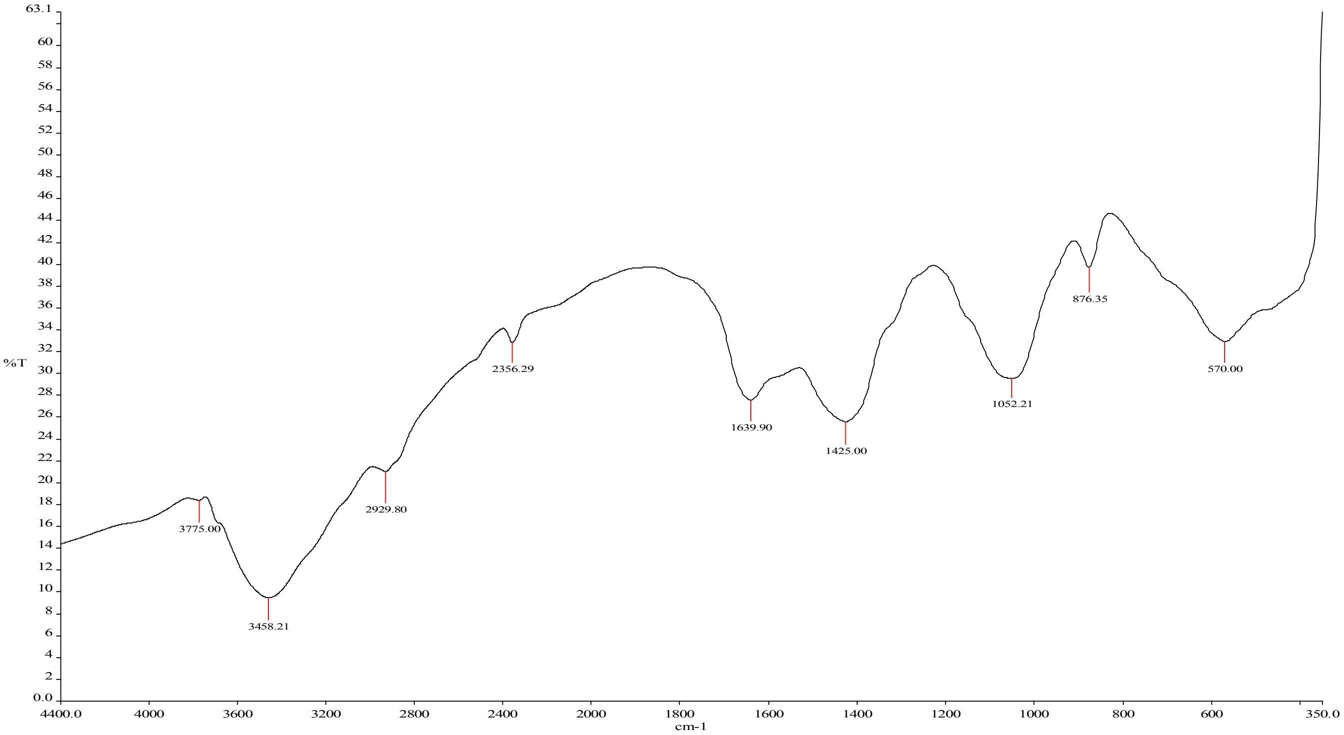

Fig. 2: FTIR spectra of GSC

Table 2. Functional groups extracted from the FTIR spectra of CPC and GSC

Functional group	CPC [Wavelength (cm^{-1})]	GSC[Wavelength (cm^{-1})]
OH stretch	3419.19	3458.21
C-H stretch	2929.76	2929.80
-C≡C- stretch	2149.00	2356.29
N-H bend	1628.72	1639.90
C-Hbend	1383.00	1425.00
N-O symmetry stretch	1316.38	-
C-N stretch (aromatic amine)	1258.45	-
C-O stretch	1079.70	1052.21
C-N (aliphatic amine)	1026.51	-
N-H wag (1 and 2 amines)	903.38	876.35

SEM analysis

The SEM images of CPC and GSC are presented in Fig. 2. The results showed the nature of the surfaces of the two cellulose samples. The SEM results revealed that the surface of CPC was an aggregate of tiny, round, smooth, and fluffy entities that are closely compacted. The fluffy nature could be a result of the soft nature of the *Carica papaya* stems, unlike regular tree stems. However, the nature of the surface of GSC was different from that of CPC, as the surface appeared to be in the form of filaments well knitted into bunches. Many researchers have reported a similar shape (Xiang *et al.,* 2006; Mihranyan *et al.,* 2011; Abe *et al.,* 2018). It is a known fact that *Gliricidiasepium* is a tree with a stubborn and tougher stem; this could occasion the thread-like shape of its pulped stems into cellulose; also responsible for this could be the orientation of the particles and fibrils.

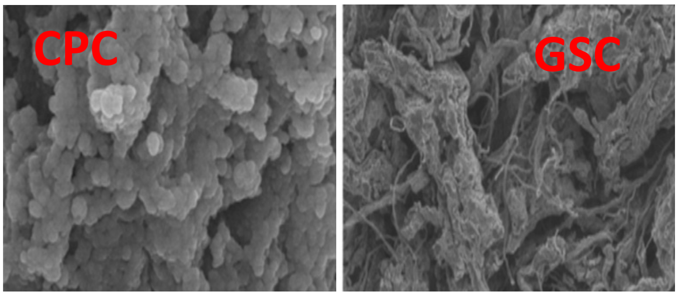

Fig. 2: SEM image for CPC and GSC

Elemental Analysis

Elemental analysis of CPC and GSC was carried out using wet digestion and Atomic Absorption Spectroscopy (AAS) methods. The results of the analysis are presented in Table 3. The analysis was done to ascertain if the uncontrolled disposal of domestic wastes could be felt and picked up by the two plants, especially the edible *Carica papaya*. The following elements: Zr; Cr; Cu; Ni; and Pb, ranging from 0.20 - 0.42 ppm were detected in the CPC sample. Same elements except for Pb, which ranged from 0.20-0.40 ppm, were detected in the GSC sample. The presence of these elements in the samples could be due to locations where the plants were cultivated and harvested.

Table 3. Detected heavy metals inCPC and GSC

Metal	CPC (ppm)	GSC (ppm)
Zr	0.20	0.24
Cr	0.40	0.20
Cu	0.42	0.25
Ni	0.40	0.40
Pb	0.20	-

CONCLUSION

The study revealed that the two plants which are unpopular for cellulose extraction could be harnessed for cellulose production. Interestingly, the two plants can easily be cultivated and withstand various weather conditions. The study revealed that both *Carica papaya* and *Gliricidiasepium* are good sources of cellulose for industrial applications.

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