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Exploring the prospects of *Calotropis gigantea* as a sustainable source of fiber and cellulose

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ABSTRACT

C. gigantea (CG) is a shrub that thrives in highlands with intricate limestone soils and coastal regions. Apart from the bark, the fruit's interior also contains fine fibers with exceptional potential as a fiber material. The abundant availability of this plant, coupled with the manual fiber extraction process, renders it an affordable and promising fiber source for various applications. This paper aims to explore the research progress on the coastal wild plant C.gigantea as a biomaterial, focusing on its utilization as fiber, cellulose, cellulose nanocrystals, and their applications. The study highlights the potential of C.gigantea in various fields, emphasizing its value as a sustainable resource for advanced material development and innovative applications. The method employed in this study involved collecting research findings from various sources, including reputable international journals and accredited national journals, published within the last 10 years. This approach ensures the inclusion of up-to-date and high-quality studies, providing a comprehensive overview of the topic. Numerous studies have delved into CG plant-based fibers and cellulose nanocrystals (CNCs) as viable solutions to provide raw materials for natural polymer applications. Research endeavors persist in the quest for new natural resources possessing suitable physical, chemical, and mechanical properties to supplant synthetic fibers. These endeavors aim to unveil novel cellulosic materials applicable across diverse fields, particularly in composite material production. CG stands out as an alternative natural fiber endowed with distinctive characteristics, notably its hollow fiber structure, contributing to its low-density nature and excellent thermal insulation properties. Its incorporation as a composite material enhances the overall physical and mechanical properties of the composite. This article presents a concise overview of the unique attributes of CG (bark and seedpod fibers) and their applications, both as cellulose and reinforcement materials.

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INTRODUCTION

Calotropis gigantea (CG) is a wild plant commonly found in tropical regions, particularly in coastal areas, belonging to the *Asclepiadaceae* family. Extensive research has been conducted on this plant for its medicinal properties (Alam et al. 2008; Parihar and Balekar 2016; Hassan et al. 2017, Bairagi et al. 2018). CG is renowned for its medicinal attributes across various parts, including the root bark, flowers, sap, leaves, and fruits, which contain fine silk-like fibers attached to each seed (Sukardan et al. 2017). This shrub can reach heights of up to 3 meters and yields fiber from both its stem bark and fruit seeds. The milky white sap extracted from its stems is known for its health benefits, serving as an herbal medicine and a source of protease enzyme (Elfian et al. 2017, Elamanidar et al. 2022, Farida et al. 2022, Fajriyati et al. 2023). Both the stems and fruits (seedpod) present potential sources of fibers, with numerous studies conducted on CG fibers (Ashori and Bahreini 2009, Chen et al. 2013, Ganeshan et al. 2018, Vinod et al. 2018, Yoganandam et al. 2020).

According to search results using the keywords "*Calotropis*, fibers and cellulose", depicted in Figure 2, wild coastal plants known as the purple crown flower or CG have been the subject of research in up to 50 articles published in 2020. The research data encompass various applications of CG, including antibacterial properties, proteolytic enzymes, and fiber utilization.

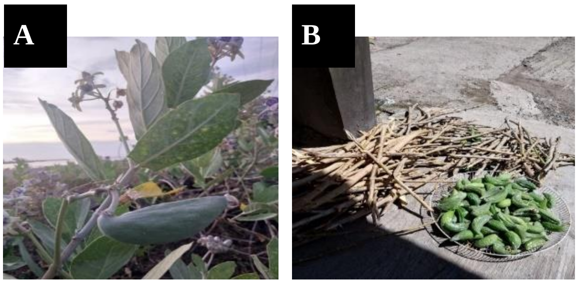

Figure 1 (A) Shrub of CG; (B) wood skins and pods of CG

Research on CG aims to enhance its value, envisioning this wild plant as a viable economic commodity worthy of cultivation and development. A single CG tree can yield a minimum of 300 fruits per month/tree. Considering a fiber yield of 8.9% of the dry weight of the fruit (4.59 grams), each tree can potentially produce at least 122,553 grams of fiber/tree/month. Assuming 2,500 trees per hectare, the potential fiber production could reach 306.382 kg/month/ha (3,676 tons/ha/year). CG's productivity is notably superior to that of kapok trees, which typically yield only 500 kg/ha/year (when intercropped with cocoa trees) and require five years for the first harvest. Despite CG's promising development potential, there is a lack of interest from farmers, agencies, or institutions in cultivating this plant sustainably, possibly due to the downstream industry's need for a guaranteed sustainable market share (Sukardan et al. 2017).

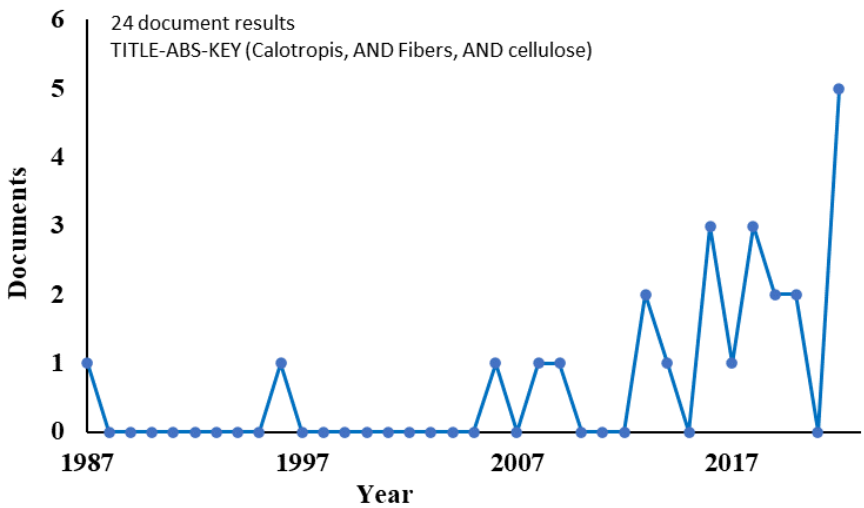

Figure 2 Research searches related to CG (source: Scopus.com)

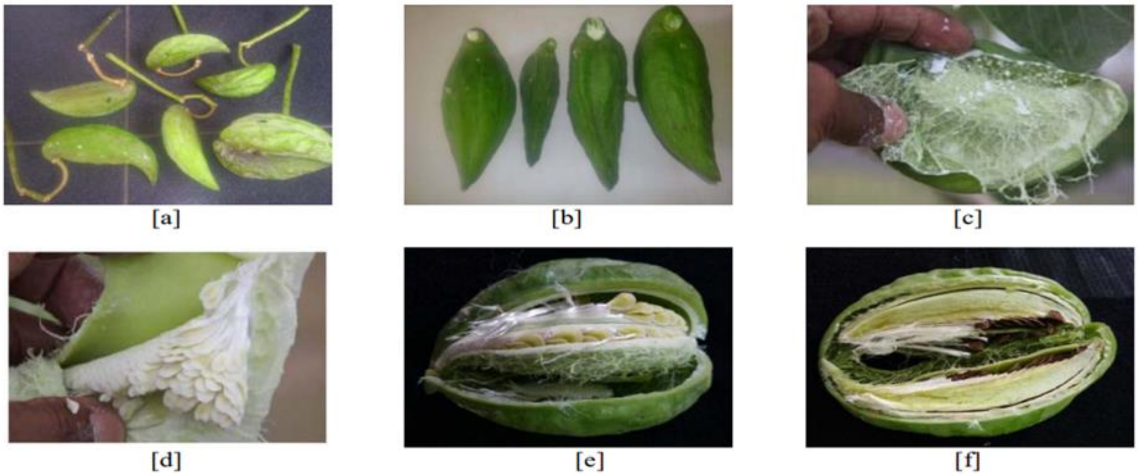

Figure 3 Fruit of CG [a-e] young fruit, still containing sap; [f] old fruit with dark seeds (Alam et al. 2008)

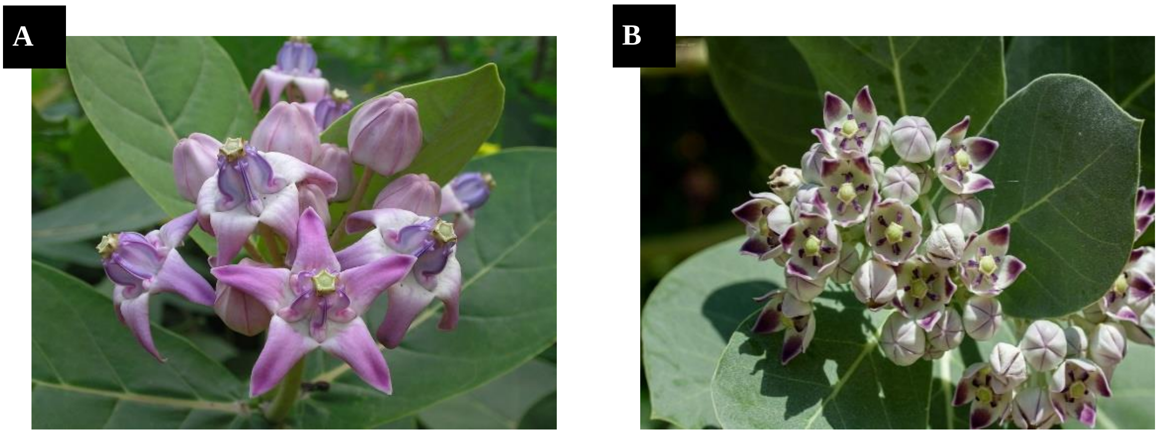

Figure 4 Flower differences of (A) CG and (B) CP

The advancement of technology prompts researchers to innovate renewable materials with enhanced characteristics. One such example is the development of polymer materials like cellulose, involving the exploration of new raw materials and processing technologies. CG stands as a potential fiber candidate for the production of nanocrystalline cellulose and microcrystalline cellulose.

This review aims to summarize previous research on the potential of *C.gigantea* plants as a renewable source of fiber and cellulose. The article presents the performance of fibers and cellulose derived from these plants as materials with promising reinforcement capabilities.

METHODS

The approach employed in crafting this article involved conducting a thorough literature review, which included scrutinizing scientific journals. The literature and journals chosen as reference materials were purposefully selected, specifically those pertinent to the discussed topic: the utilization of C. gigantea plants for fiber and cellulose. Approximately 80% of the selected articles were published within the last decade. The scientific articles were selected based on their relevance to the topic and titles, categorized according to the specific parts of the C. gigantea plant utilized as research raw materials, as well as the characteristics of the fibers and cellulose obtained. Additionally, the manufacturing methods and their applications as reinforcing materials were explored, followed by an analysis of the results obtained from various parts of the CG plant utilized.

The exploration of analogous research in the Scopus database utilizing the keywords: “Calotropis, fibers, and cellulose”.

Calotropis As Fiber And Cellulose

CG proliferates abundantly in various regions, particularly in hot deserts and coastal areas. Known as the purple crown flower in English, widuri/biduri in Indonesian, and bak reubek in Aceh, this plant belongs to the

Asclepiadaceae family, also known as the Milkweed family, characterized by its milky white sap. The most prevalent genus in Indonesia includes CG and *C.procera* (CP), distinguished by the color of their flowers (as seen in Fig. 4). CG bears purple flowers, hence its name "purple crown flowers," while CP features white flowers (Gupta 2018). CP is commonly referred to as the "Sodom apple.". Figure 3 shows that CG plants have milky white sap, hence they are referred to as milkweed plants.

Researchers are increasingly exploring coastal wild plants as raw materials for fibers due to their mechanical characteristics, which are comparable to or even superior to those of commercially used natural fibers, as previously documented (Ashori and Bahreini 2009, Maji et al. 2013a, Oun and Rhim 2016, Zheng et al. 2016, Sukardan et al. 2017, Hassanzadeh and Hasani 2017, Ganeshan et al. 2018, Vinod et al. 2018, Nisah 2018, Qi et al. 2018, Ramasamy et al. 2018, Narayanasamy et al. 2020b, Gao et al. 2020). CG is a type of natural cellulose fiber composed of cellulose, hemicellulose, lignin, pectin, wax, and ash (Nourbakhsh et al. 2009, Ashori and Nourbakhsh 2010, Tarabi et al. 2015, Qi et al. 2018). The cellulose content in CG fibers reaches 71.62%, similar to that of kapok fibers at 72.86%, but lower than that of cotton fibers at 90% (Sukardan et al. 2017). Tubular CG fibers with thin and hollow walls resemble kapok fibers and can serve as reinforcement in composites (Ashori and Nourbakhsh 2010, Ganeshan et al. 2018, Vinod et al. 2018, Jeyapragash et al. 2022). Tubular CG fibers possess a hollow structure with an outer diameter ranging from 15 μm to 26 μm, wall thickness of 0.8 μm to 2 μm, and cavity volume of 92.3 μm to 94.7 μm. These dimensions resemble those of kapok fibers, which have a cavity volume of 92.8 μm to 94 μm, fiber wall thickness of 0.6 μm to 1.3 μm, and fiber outer diameter of 10 μm to 18 μm (Sukardan et al. 2017). The dimensional characteristics of CG fibers render them with low-density values and potential applications as buoyancy materials (floating in water and oil), sound absorbers, thermal insulators, and waterproof and voluminous materials. CG fibers can be obtained from its stems and fruits, as demonstrated by various research findings presented in Table 2.

The morphology of CG fibers, as identified through Scanning Electron Microscope (SEM) analysis, reveals transverse and longitudinal cross-sections, depicted in Figure 5. Specifically, Figures 5a and 5b illustrate the flat (smooth) appearance of the longitudinal cross-sections of CG fibers.

Its hollow structure and thin walls cause CG fibers to have a low density. Previous researchers stated that the density of CG fibers is 0.97 g/cm^3, lower than cotton, which is 1.54 g/cm^3 (Bairagi et al. 2018). The appealing characteristics of CG fibers have garnered significant interest from researchers. Studies on fibers and CNCs highlight CG (stem fiber and stump fiber) as a promising natural fiber source worthy of further development. Table 1 presents some research findings on CG fiber characteristics.

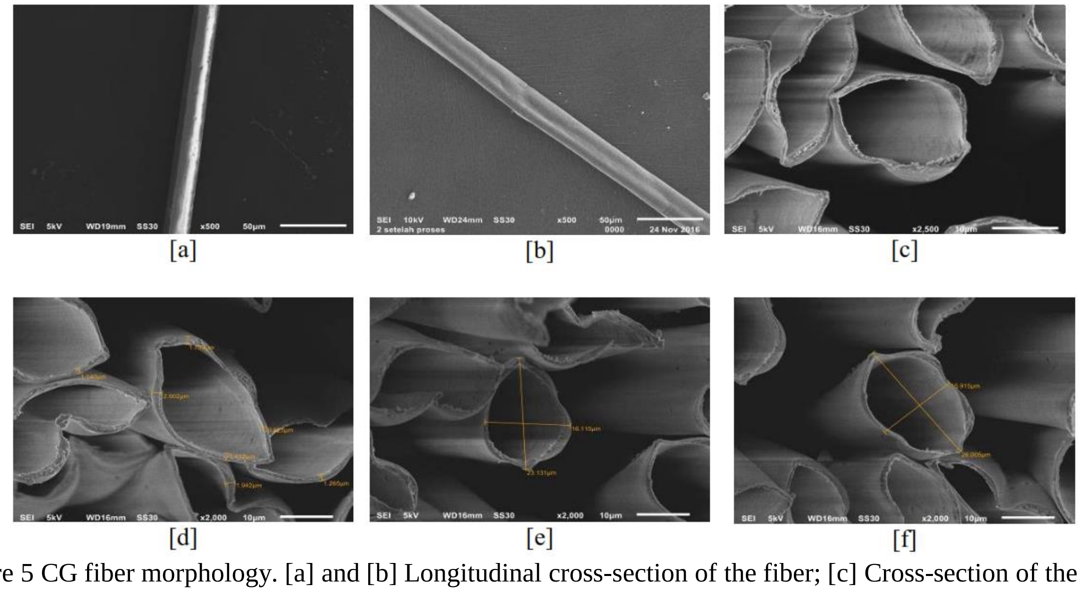

Figure 5 CG fiber morphology. [a] and [b] Longitudinal cross-section of the fiber; [c] Cross-section of the fiber; [d] Size of fiber wall thickness; [e] and [f] Size of fiber outer diameter (Alam et al. 2008)

Table 1 Studies on the use of CG as fiber and CNCs

Source	Type	Result	Ref.
Seedpod	Fiber	This fiber contains C (42.80%); O (50.67%); H (6.32%); N (0.21%), 66% cellulose, 21% hemicellulose, 9% lignin, 42.54% crystallinity, 3.4% elongation, 85.4% crystallinity orientation index (COI)	(Chen et al. 2013)
Fruit	Fiber	Hollow fiber morphology, hollow volume 92.3 - 94.7%, yield 8.9% of fruit weight, cellulose 71.62%, moisture content 7.9%; lignin 14.08%, low density, buoyant (floats in oil and water), oleophilic, and hydrophobic	(Sukardan et al. 2017)
Fruit and stem bark	Fiber	Fruit: holocellulose 69%, cellulose 49%, lignin 23%, tensile strength 296 Mpa, density 0.68 g/cm^3 Stem bark fiber: holocellulose 76%, cellulose 57%, lignin 18%, tensile strength 381 Mpa, density 0.56 g/cm^3	(Ashori and Nourbakhsh 2010)
Stem bark	Fiber	% fiber efficiency: alkali treatment = 6.03%, acid treatment = 26.65 % cellulose composition: alkali treatment = 73.5%, acid treatment = 84.7	(Maji et al. 2013b)
Fruit	CNCs	Yield = 55.37%; rod-shaped CNC with length 242.06 nm, width 8.8 nm. CNC aspect ratio (length: width) = 27.5 times	(Gao et al. 2020)
	Fiber	Crystallinity 43.08%, crystallinity orientation index (COI) 80.50%, humidity recovery 12.65%, thermal conductivity 97.63%	(Qi et al. 2018)
Fruit	CNCs	The resulting fibers (length: 177-415 µm, width 22-38 µm, crystallinity index 0.40, crystallite size 2.05 nm), cellulose (yield 45%, width 10-39 µm, crystallinity index 0.57, crystallite size 3.92 nm), CNC (length 140-260 nm, width 14-24 nm, crystallinity index 0.70, crystallite size 4.12 nm), CNF (width 10-20 nm, crystallinity index 0.59, crystallite size 2.92 nm)	(Oun and Rhim 2016)
Seed	CNCs*	Cellulose yield 64.1%, CNC needle-like with length 250 nm and width 12 nm, aspect ratio 30, crystallinity index 68.7%	(Song et al. 2019)
Stem bark, seedpod	Fiber	42.54% crystallinity index for fiber from stem bark, 35.16% crystallinity index for fiber from seedpod, The fiber morphology of the fruit exhibits a greater degree of hollowness compared to that of the bark.	(Handayani et al. 2024)

Calotropis Fiber Extraction

Fibers are separated from plants through various methods, with dew retting and plain water retting being the most commonly employed. However, these methods typically require approximately 14 to 28 days for the degradation of waxes, pectin, hemicellulose, and lignin (Karimah et al. 2021). Cellulose fibers from CG bark have been effectively extracted using the plain water retting method followed by alkali treatment (Ramasamy et al. 2018). Given that the CG plant contains sap, known as degumming (retting), is necessary to separate the fiber. This stage is critical in fiber production because the sap, which contains pectin and hemicellulose, can impact the final quality of the fiber produced (Novarini and Sukardan 2015). Refer to Figure 6.

One of the primary components of plant cell walls is lignocellulose. This renewable biomass comprises three main constituents: cellulose (35-50%), hemicellulose (20-35%), and lignin (10-25%), which form complex bonds.

Each of these lignocellulosic components offers significant benefits. Numerous studies have aimed to separate (fractionate) and utilize them in the production of chemicals, polymers, ethanol, natural sweeteners, and resins. The composition of each component (lignin, hemicellulose, cellulose) can vary based on factors such as the plant species and environmental conditions, including storage, soil conditions, and climate where the biomass grows.

Pretreatment of lignocellulosic biomass is essential to facilitate degradation and increase the surface area for contact between the hydrolysis agent and the polymer within, namely cellulose, thereby reducing the degree of cellulose crystallinity. The presence of lignin, with its complex structure serving as the primary support of the lignocellulosic wall, poses a challenge to the degradation process, making accessing cellulose and hemicellulose difficult. The formation of matrix polymers due to the crystalline structure of cellulose with hemicellulose fibers further exacerbates this obstacle (Sukardan et al. 2017).

The pretreatment stage involves damaging the lignocellulose structure by partially breaking down the constituent polymers and weakening the heteromeric bonds of lignin and hemicellulose. This results in an increase in pore size and surface area of cellulose and hemicellulose exposed to the hydrolyzing agent, leading to a hydrolysis yield of up to 90%. Various methods, including physical, chemical, biological, or combinations thereof, can be employed for this stage (Elamanidar et al. 2022).

Figure 6 Stages of CG fiber extraction: (left) stem bark fiber, (right) seedpod fiber (Hassan et al. 2017)

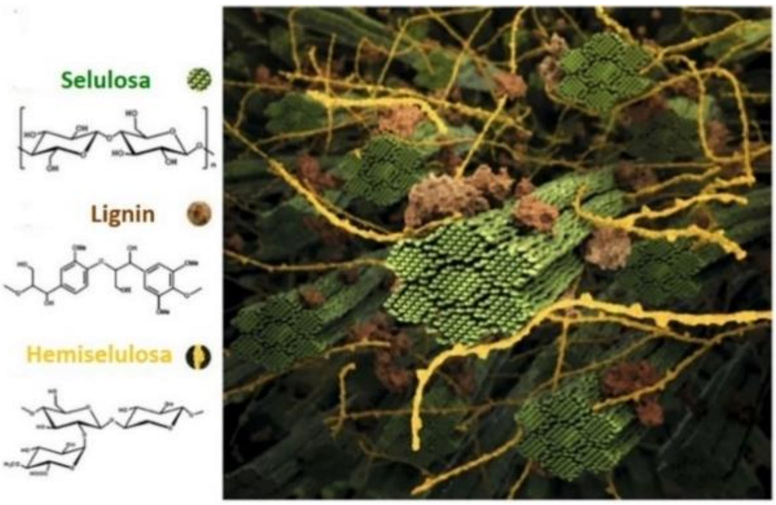

Figure 7 The structure of lignocellulosic biomass and constituent components (Parihar and Balekar 2016)

Table 2 Pretreatment methods for producing natural wood fiber

Method	Advantage	Weakness	Reff
Dew retting	Utilizing bacteria and plant moisture to separate fibers from plants, promoting environmentally friendly and energy-efficient processes. Commonly employed in regions with heavy nighttime dew, daytime warmth, and limited water resources	Excessive retting will cause the fiber to be challenging to detach by itself, and the strength of the fiber is lost Takes a long time, up to 3-6 weeks, depending on the climate Dark-colored fibers and low strength	(Bismarck et al. 2005, Novarini and Sukardan 2015, Ahmed et al. 2018, Karimah et al. 2021)
Water retting	Yields fibers with high cellulose content and exceptional tensile strength Produces low-density fibers suitable for composite applications	Requires water treatment costs due to the odor caused by the gas formed by the fermentation process Takes 7-14 days	(Bismarck et al. 2005, Novarini and Sukardan 2015, Ahmed et al. 2018, Karimah et al. 2021)
Hot water retting	Expedited process duration (3-5 days) for high-quality, uniform fibers	Pollution and high costs	(Bismarck et al. 2005, Novarini and Sukardan 2015)
Enzyme retting	Streamlined process (2-24 hours) ensures fiber integrity without damage	High cost, not suitable for industrial scale	(Bismarck et al. 2005)
Mechanical retting/ green retting)	Shortened processing time to just 2-3 days	The fibers formed become coarser	(Arthanarieswaran et al. 2015, Novarini and Sukardan 2015, Ahmed et al. 2018, Karimah et al. 2021)
Chemical retting (using surfactant/H_2SO_4/NaOH/sodium carbonate)	Accelerated processing, ranging from minutes to a maximum of 48 hours Resultant fibers possess high cellulose content, excellent thermal stability, tensile strength, and crystallinity index Exhibits favorable fiber morphology Demonstrates commendable physicochemical properties.	Produces waste can pollute the environment Requires significant additional costs for waste treatment	(Chandramohan and Bharanichandar 2014, Novarini and Sukardan 2015, Hassan et al. 2017, Ahmed et al. 2018)

The oldest known retting method, water retting, was historically widely employed for wood fiber production due to its numerous advantages. However, this method is associated with drawbacks such as generating significant wastewater and requiring extended processing time. The duration of water retting also impacts the characteristics of the resultant fiber. For instance, fiber extracted from the bark of the Kydia calycina plant subjected to water retting for varying durations—10, 15, 20, and 25 days—exhibits optimal characteristics when retted for 20 days. Fibers processed under this duration display a strength of 4.69 g/d, elongation of 4.01%, fineness of 36.34 denier, density of 1.4 g/cc, and yield of 4.19% (Chen et al. 2013). Moreover, CG fibers subjected to a drying period of one week, followed by a three-day water-retaining phase, and subsequent soaking in 5% NaOH for two hours, yield distinct characteristics compared to fibers solely subjected to water retention treatment without subsequent alkali soaking, as illustrated in Table 3.

These characteristics are confirmed by the results of FTIR, which showed that the untreated bark fiber produced an intense peak at a wavelength of 3333 cm^{-1}, which indicates the presence of hydroxyl groups. The peak at 2898 cm^{-1} indicates O-H stretching due to hydrocarbon components in the fiber, peaks caused by lignin appear at 1646 cm^{-1} and 1317 cm^{-1}, and the presence of hemicellulose is characterized by peaks at 1364 cm^{-1} and 1265 cm^{-1} while cellulose is characterized by the formation of peaks at 1155 cm^{-1}, 1108 cm^{-1}, 1051 cm^{-1}, 610 cm^{-1} (Qi et al. 2018). The decrease in intensity of peaks formed at 1646 cm^{-1}, 1364 cm^{-1}, 1317 cm^{-1}, 1265 cm^{-1} indicates lignin and hemicellulose can be partially reduced through alkali treatment, as well as an increase in peak intensity at 3500 - 3520 cm^{-1} which indicates an increase in free hydroxyl groups due to a decrease in hemicellulose and lignin levels after undergoing alkali treatment (Qi et al. 2018).

In addition to serving as a fiber, CG, like CP, also possesses the potential to yield cellulose. CP, when transformed into Cellulose Nanocrystals (CNCs) via an acid hydrolysis process, exhibits a crystallinity index (Crl) of 68.7%. Prior to hydrolysis, the initial CP wood fiber displays a crystallinity index of 30.1%, with a cellulose content of 64.1% and a lignin content of 9.7% (Vinod et al. 2018). The notable increase in the crystallinity index of CG CNCs compared to CG bark fiber is attributed to the acid's capability to hydrolyze the amorphous portion of the hemicellulose, consequently leaving behind a more crystalline region. This observation is corroborated by the results of FTIR analysis, where the absence of peaks at wave numbers around 1742 cm^{-1} and 1522 cm^{-1} in the CNC spectra indicates the removal of lignin following acid hydrolysis treatment. Moreover, the successful removal is further evidenced by the emergence of a new peak at wave number 1205 cm^{-1}, resulting from the attachment of the sulfate group subsequent to the acid hydrolysis process employing H_2SO_4. Refer to Figure 9.

CG as a Composite

Composites are materials composed of two or more substances with distinct physical and chemical properties, resulting in new materials with characteristics differing from their components. These new materials may be lighter, more robust, or even more cost-effective compared to traditional materials. The constituent components maintain their separate identities within the structure of the new material. Natural fibers from sources like jute, kenaf, bamboo, pineapple leaves, and straw are employed as alternatives to synthetic fibers in manufacturing medium to high-strength biocomposites. Additionally, fibers from *C.gigantea* (CG), derived from various parts such as the fruit and bark, are utilized in composite materials, as shown in Table 4.

Table 3 Characteristics of stem bark fibers from CG with various pretreatments (Ganeshan et al. 2018)

Treatment	Cellulose (%)	Lignin (%)	Hemiselulosa (%)	Tensile strength (MPa)	Density (g/cm^3)	Elongation (%)
Untreated	63.56	10.38	19.29	629	1.324	3.5
Alkali treated	81.31	4.27	10.06	680	1.312	3.4

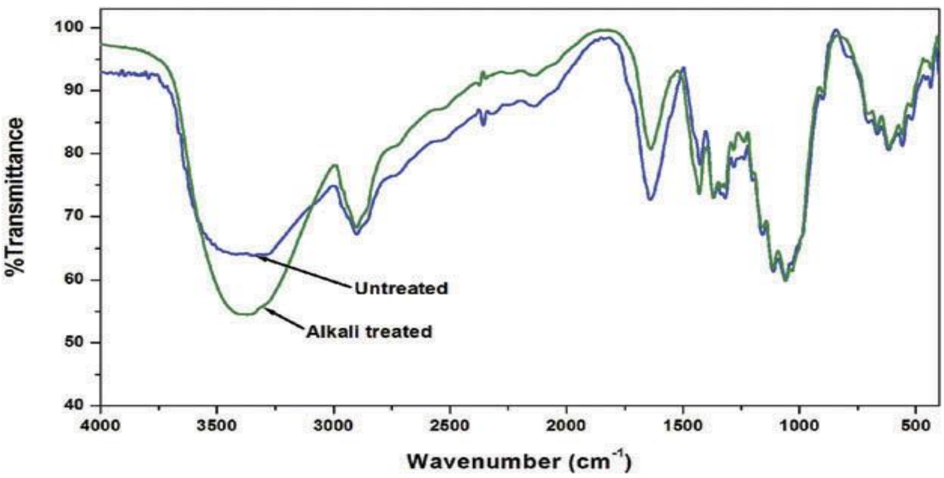

Figure 8 FTIR spectra of untreated and alkali-treated CG bark fibers (Ganeshan et al. 2018)

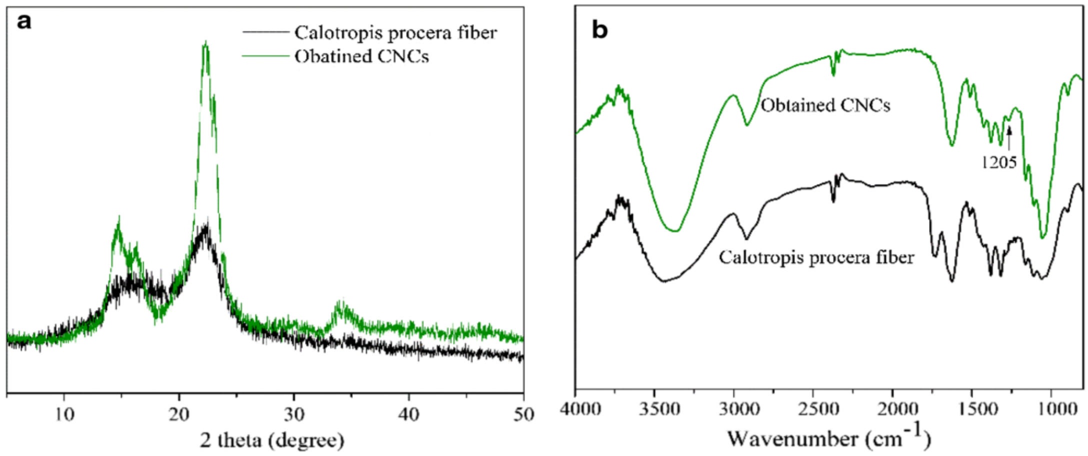

Figure 9 XRD and FTIR analysis results of CP bark fibers and the resulting CNCs

Table 4 Research on *C.gigantea* (CG) and *C.procera* (CP) plants

Source		Result	Ref.
Fruit	Fiber	Applied as a reinforcement in plastic: Increase the tensile strength of the composite to 49.925 N/mm^2, young's mgodulus 1248.125 Mpa, flexural strength 113.3 N/mm^2, flexural modulus 140.37 N/mm^2	(Srinivas and Babu 2013)
Stem bark	Fiber	Applied as a reinforcement in epoxy composites: Improve epoxy composition characteristics, tensile strength 48.73 Mpa, flexural strength 195.19 N/mm^2	(Vinod et al. 2018)
Fruit	Fiber	Thermal stability 282°C, maximum degradation 317°C, crystallinity index without treatment 39.8% and with alkali treatment 36%	(Narayanasamy et al. 2020a)
Stem bark	Fiber	Applied as a filler in composite foam/ porous composite: The fibers were produced using different source materials, including fibers extracted from young and mature stem barks, as well as fruit (seedpod fibers). The optimal performance as a composite filler was achieved with fibers derived from young stem bark due to their highest degree of crystallinity, which also contributes to increased composite porosity.	(Handayani et al. 2024)

CONCLUSIONS

This review concentrates on the latest advancements in renewable materials derived from the CG plant, particularly fibers and cellulose. With their hydrophobic, oleophilic, low-density properties, and hollow morphology, CG fibers are ideal materials for applications in composite manufacturing and filtration industries. Furthermore, due to their hollow morphology, CG fibers are well-suited for thermal insulation and sound absorption purposes. Moreover, C. gigantea plants hold significant potential for use as reinforcement material in composites because of their favorable characteristics as strengthening agents.

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