



Recent developments of the nanocellulose extraction from water hyacinth: a review

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Abstract Nanocellulose, an exceptional natural material acquired from cellulose, has received tremendous attention over the past decades. This is because of its unique physical characteristics, surface chemistry, functional properties, and biocompatibility. Cellulose nanocrystals (CNCs) and cellulose nanofibers (CNFs) are two major types of nanocellulose that have received key interest so far. Nanocellulose is commercially produced from wood-based sources. With the ongoing demand for nanocellulose, agricultural wastes and non-woody plants are getting much consideration as cost-efficient alternatives. Water hyacinth (WH) is an alternative source that has shown great prospects for CNC and CNF fabrication. Nanocellulose from WH is proposed for several interesting applications, such as wet-spun filaments,

aerogels, packaging films, battery separators, and water filtration, showing promising results. Studies have shown the nanocellulose achieved from WH has a comparable diameter and crystallinity related to the nanocellulose derived from other agricultural wastes, but lower than nanocellulose extracted from wood. Studies were more inclined towards chemical treatments for CNC extraction and mechanical processes for CNF isolation. This review aims to emphasize identifying the true potential of WH as a nanocellulose resource by critical evaluation of the isolation processes, affecting factors on nanocellulose properties, and the foundation of future research for producing sustainable materials. It was perceived that a lot of possibilities remain unexplored as many advanced techniques are yet not well studied for WH

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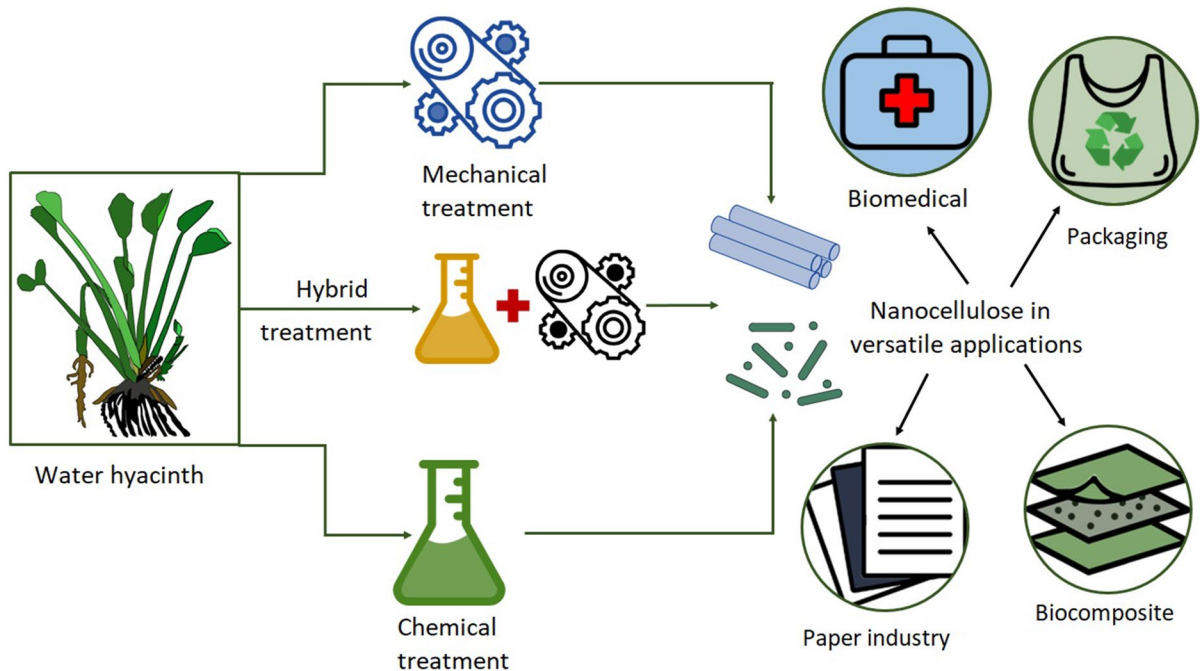
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nanocellulose extraction. Further, the advantage of being a low-lignin raw material was not properly captured by mapping the more sustainable procedures, which should be a future highlight of WH research.

Graphical abstract



Keywords *Eichhornia crassipes* · Cellulose nanocrystals · Cellulose nanofibrils · Chemical isolation · Mechanical isolation · Sustainability

Introduction

The growing interest in sustainable and natural materials worldwide has expanded the use of nanocellulose in the fabrication of varied products with outstanding physicochemical characteristics. Nanocellulose holds unique characteristics (e.g., surface area, functionality, crystallinity and tensile properties) compared to native cellulose and is proven beneficial for achieving different essential properties of cellulose-based materials. (Ferrer et al. 2017; Tanpichai 2022).

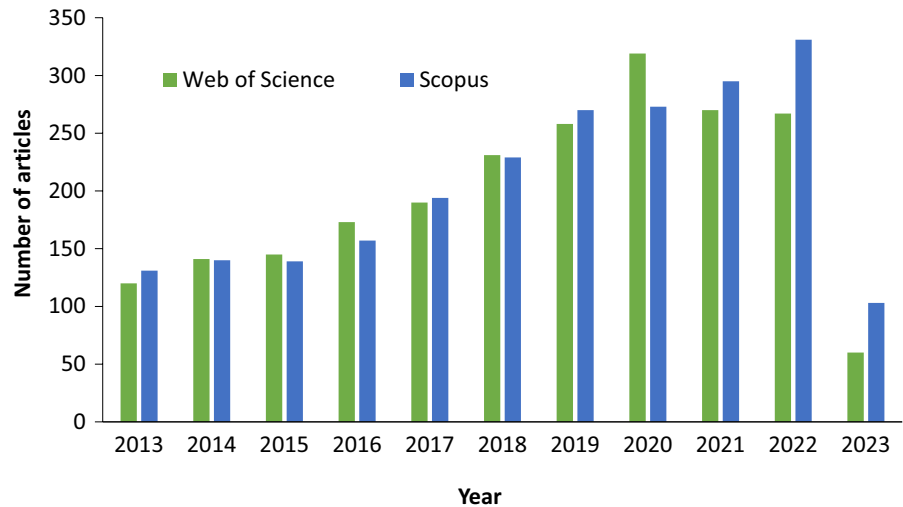
In nanocellulose, at least one dimension of nanoparticles remains on a 1 to 100 nm scale. They can be extracted from common cellulosic resources such as wood, bast and seed fibers, agricultural wastes,

bamboo, bacteria, fungi, and marine animals. Nanocellulose is mainly classified into cellulose nanocrystals (CNCs), cellulose nanofibers (CNFs), and bacterial cellulose (BC) based on its dimension, functionality, and extraction processes (Nasir et al. 2017).

The nanocellulose global market is expected to reach 661 million USD by 2023 (Jordan et al. 2019). To meet this demand, the selection of appropriate raw materials as well as sustainable methods is important, concerning harsh treatments required to isolate hemicellulose and lignin from cellulose and break the cellulose inter-fibrillar bonds.

Water hyacinth (WH) is a rapidly growing herb floating on water. It originated from South America but is widely available in nature, particularly in tropical and sub-tropical regions (Namasivayam et al. 2022; Barua and Kalamdhad 2019). It is an undesirable aquatic weed that frequently spreads, forming closely packed layers that cover the water surface (Islam et al. 2021; Barua and Kalamdhad 2017). It restricts light supply into the water and influences

Fig. 1 The number of research published on WH over the last ten years, searched by the keyword ‘water hyacinth’ in Web of Science and Scopus database on 4 April 2023



irrigation and fisheries (Jirawattanasomkul et al. 2021). WH can produce 14×10^7 daughter plants within a year, covering a 1.4 km^2 area, and generating 28×10^7 tons of lignocellulosic biomass (Gaurav et al. 2020). This fast-growing weed has been considered an invasive aquatic plant in the United States since 1984, in Europe since the 1930s, in Asia since 1902 and in Africa since the early 1900s (Dersseh et al. 2019). Researchers have been showing increasing interest in WH, which is continuing to increase in the recent past. Numerous studies have been performed to make proper use of this abundant low-cost resource in many different applications such as food packaging paper (Hosen et al. 2022), polymer composites (Jirawattanasomkul et al. 2021), aerogels (Ewulonu et al. 2020), textiles (Kusnan and Setiadi 2020), construction (Goel and Kalamdhad 2018), water treatment (Gong et al. 2018) and combustion (Huang et al. 2020). Figure 1 shows the increasing number of WH studies published over the last decade.

Due to its high cellulose content (45–64% reported in the literature), abundance, and biodegradable nature, WH is a potential resource for nanocellulose extraction. This plant contains lower lignin (as low as 4%) (Tanpichai et al. 2019) and higher cellulose (as high as 64%) (Syafri et al. 2019a) than many other cultivated plants, which is an advantage. It requires less-intensive treatments for cellulose isolation and generates minimal in-process residue. Further, nanocellulose extraction can be done at an accelerated rate by avoiding some of the chemical treatments that are commonly required for lignin removal. Besides, using

WH for nanocellulose extraction can significantly reduce the production cost as WH is collectible at negligible cost.

Synopsis of WH

In recent years, nanocellulose from WH has been proposed for composite reinforcement, water filtration, battery separators, and textile fibers. However, there is a current limitation in the literature on the critical evaluation of these attempts to understand the true suitability of the proposed methods and identify future scope. The available reviews on WH are mainly focused on the ecology and socio-economic impact, biological control, biosorption, and bioenergy production.

There is limited research on the impact of WH variability on the properties of the extracted nanocellulose, despite variation in its species composition. Therefore, this review addresses the distinct methods used for nanocellulose extraction from WH and evaluates their advantages and limitations, with an emphasis on sustainable future pathways.

Growing conditions

WH belongs to the species *Eichhorina crassipes* from the Pontederiaceae family (Coetzee et al. 2014). Tropical and subtropical regions are favorable for WH growth. This plant first spread from the Amazon

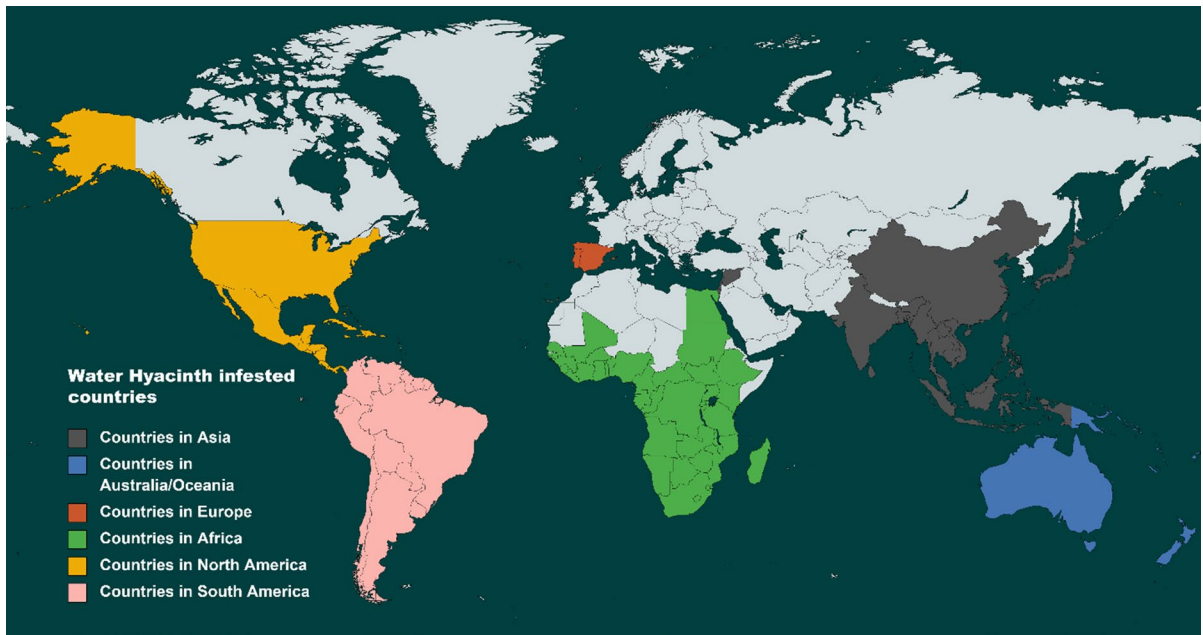


Fig. 2 Illustration depicting the regions around the world where the WH plants are found

basin in the 1800s (Mujere 2016) and now is expanding uncontrollably in many places (Fig. 2).

The Major biological characteristics of WH include morphological adaptation to the environment, varying chemical composition of different parts under several habitats, capability to invade the ecosystem, and persistent spreading by rapid reproduction. Human activities, bird legs, shared and common rivers, and watersheds are some common reasons for spreading WH from one location to another (Dersseh et al. 2019). Environmental factors, such as temperature, sunlight, water pH, salinity, and eutrophication impact the optimum growth and reproduction of WH (Dersseh et al. 2019). Suitable conditions for optimum growth of WH are acidic to neutral pH (5 to 7.5) (Gong et al. 2018), calm and low depth of water (i.e., < 6 m), and temperature of 28–30 °C where the optimal air temperature is between 21 and 30 °C (Yan and Guo 2017). Nutrient concentration in the water body is also a determinant factor for the growth of WH (Wilson et al. 2005). The optimal nutrient requirement for WH survival is 5.5 to 20 mg/L of nitrate, 1.66 to 3 mg/L of phosphate, and up to 53 mg/L of potassium (Gaikwad and Gavande 2017). A high ammonium content (NH_4^+ , ~370 mg/L) and

increased salinity are harmful to WH production (Yan and Guo 2017). A salt concentration of <2% in water is required for WH survival (Nguyen et al. 2015).

WH is known for its high reproduction rate. Its reproduction system can be occurred both sexually (by seed formation) and asexually or vegetative means (by budding and stolon production) (Extross et al. 2022). The sexual reproduction of WH involves the formation of seeds from its flowers. Each plant can produce up to 5,000 seeds (400 seeds per flower). The seeds can survive up to 15 years in water, silt, or mud (Mujere 2016). WH grows into a floated mat structure on freshwater bodies and doubles itself within 4–7 days by covering the surface of water.

In vegetative reproduction, daughter plants or ramets are produced on the stolon. The vegetative propagation is fast with a doubling time of 11 to 18 days under favorable conditions (Mujere 2016). Three parent WH plants can generate 3,000 new plants in 50 days and two parent plants can produce 30 off-springs after 23 days (Gaikwad and Gavande 2017). Vegetative growth of WH occurs through offsets. After separating from the parent plants, the offsets (containing sail-like leaves) float rapidly

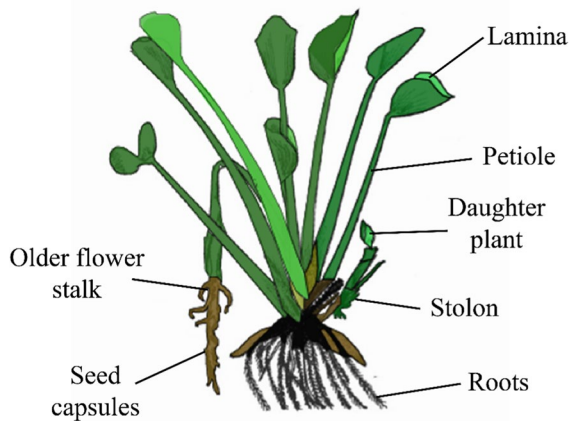


Fig. 3 Schematic illustration of WH plant showing different parts, redrawn and adapted from (Ali and Abbas 2020)

from one place to another and start forming a new population when they find a favorable environment (Bock 1969). It is reported that during the vegetative reproduction system (also known as the uniparental reproduction system), around 3,418,800 WH plants can be yielded in just about 200 days (Gaikwad and Gavande 2017).

Physical structure

WH is composed of bright to dark green rounded leaves and long spongy stalks. The roots are elongated treads, freely hanging, and purple-black. Each

non-branched fibrous root is 1 mm in diameter and contains numerous lateral hairy rootlets with functional root tips (Hadad et al. 2009). WH root surface area may vary from 30 to 60 m² per individual (Zhou et al. 2012) and it can eradicate heavy metals as well as organic pollutants present in sewage. The physical structure of a mature WH that is growing in a favorable condition is shown in Fig. 3.

The presence of spongy tissue in several parts (root, stolon, pseudo lamina) of WH is another special feature of its biology (Zhang and Guo 2017). This spongy tissue increases floating forces and impairs the harvesting efficiency of WH. The density of different plant parts is used to express the floating capability of WH. The whole plant of WH has a density of around 167 kg/m³, while the bottom parts like the root (782 kg/m³) and stolon (818 kg/m³) have much higher density (Yan and Guo 2017) and remain under water.

Chemical composition

Like other lignocellulosic materials, WH contains cellulose, hemicellulose, and lignin as its major parts. Cellulose is a long-chain crystalline unit made of D-glucose having a high degree of polymerization. The D-glucose units in cellulose are connected through β -1,4 glycosidic linkage. Hemicellulose is a branched structure occupied with cellulose and has a lower degree of polymerization. The other part lignin is a heterogeneous three-dimensional nonlinear polymer that binds cellulose micro-fibrils and

Table 1 Cellulose, hemicellulose, and lignin content in WH reported from different sources of origin

Origin	WH parts used	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
Indonesia	Stems	64.07	15.13	10.48	(Syafri et al. 2019a)
Indonesia	Stems	43.01	29.13	6.9	(Asrofi et al. 2018c)
Nigeria	Stems	45.52	21.76	8.31	(Oyeoka et al. 2021)
Thailand	Stems	52.06	17.56	8.66	(Pakutsah and Aht-Ong 2020; Asrofi et al. 2018c)
Thailand	Not specified	57	25.6	4.1	(Tanpichai et al. 2022)
India	Leaves and stems	19.2	40	4.8	(Singh and Bishnoi 2013)
India	Stems	65.4	12.8	7.2	(Arivendan et al. 2022)
Kenya	Whole plant	~33	~23	~10	(Omondi et al. 2019)
China	Whole plant except root	24.5	34.1	8.6	(Ruan et al. 2016)
China	Whole plant	18.07	28.21	7.03	(Zhang et al. 2016)

Table 2 Cellulose, hemicellulose and lignin content in different lignocellulosic wastes and standard wood species reported in the literature

	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
Wheat straw	28–39	23–24	16–25	(Carvalho et al. 2009)
Rice straw	29–35	12–29	17–19	(Passoth and Sandgren 2019)
Cotton gin trash	25–33	7–16	20–25	(Haque et al. 2020)
Oat straw	31–35	20–26	10–15	(Passoth and Sandgren 2019)
Sorghum straw	32–35	24–27	15–21	(Passoth and Sandgren 2019)
Switch grass	31	24	18	(Rao et al. 2010)
Bagasse	41.42	28.11	19.03	(Phinichka and Kaenthong 2018)
Pineapple leaves	32.6	22.5	11.8	(Fareez et al. 2018)
Lemon grass	39.5	22.6	28.5	(Haque et al. 2018)
Beech wood	41.8	26.2	17.3	(Marks and Viell 2021)
Eucalyptus wood	41.6	19.6	27	(Chen et al. 2020)
Pine wood	40	10	30	(Muley et al. 2016)
WH stems	43–65	13–29	7–10	Table 1 summary
Whole WH	18–33	23–40	7–10	Table 1 summary

hemicellulose through cross-linking (Haque et al. 2018). Besides, WH also contains crude protein, moisture and ash (Omondi et al. 2019).

The composition of cellulose, hemicellulose, and lignin in each part of the plant (such as stem, root, and leaves) is still not very clear as there are limited studies on this. Table 1 shows the composition of WH reported across different origins. It is likely that stems of WH contain the most amount of cellulose, as perceived from Table 1. The other reports pertaining with the studies involving roots, leaves or whole plants showed a decreased cellulose content when compared with the stems. This drop was commonly found affected by the rise of hemicellulose percentages, though a lower lignin amount was mostly consistent. Lignin is important in plants to support the overall structure for vertical growth in the air as well as for withstanding gravity. However, WH is stable with a low amount of lignin content. During nanocellulose extraction, lignin is removed from the structure. A lower lignin content is an advantage since it can be removed either in a quicker time or with a lower concentration of chemical, thus making the process more sustainable and efficient. Further, the in-process residue will be lower due to the low lignin content in WH. This was probably a reason why nanocellulose extraction studies from WH only considered the stem in most cases (Juárez-Luna et al. 2019).

Therefore, WH stems can be an excellent source to obtain cellulosic fibers, compared to different common lignocellulosic wastes. Table 2 shows cellulose, hemicellulose and lignin content in different lignocellulosic wastes reported in the literature, which indicates a comparatively better chance of obtaining higher cellulose and holocellulose (cellulose and hemicellulose) yield from WH. Different standard wood (such as beech, eucalyptus, and pine) species also hold a fair amount of cellulose and hemicellulose, but a major drawback is a higher percentage of lignin (Table 2), which needs to be removed by prolonged treatment (compared to WH) during nanocellulose extraction (Tanpichai et al. 2019). Thus, the use of WH could be advantageous as a more sustainable alternative raw material for nanocellulose.

Cellulose nanocrystals (CNCs) and cellulose nanofibers (CNFs)

Nanocellulose extraction from natural resources has received great attention in recent years due to the special characteristics of cellulose at the nanoscale, such as high transparency, high specific surface area, low density, high aspect ratio, and good mechanical properties (Phanthong et al. 2018). Among the major three kinds of nanocellulose, i.e., bacterial cellulose, CNCs

and CNFs, the latter two are the main types, that have been extensively studied in the literature. Besides, elementary nanofibrils are a new class of nanocellulose having a diameter of 3–5 nm (Beaumont et al. 2021). However, studies of WH nanocellulose are mainly concentrated on CNCs and CNFs.

CNCs are highly crystalline nanoscale materials consisting of whisker or rod-like particles. They are lightweight, stiff, transparent, gas impermeable, mechanically strong, able to form liquid crystals, and have adaptable surface chemistry and low thermal expansion (Trache et al. 2017). They commonly have high crystallinity (54–88%) with a high specific surface area and a high aspect ratio (3–5 nm wide and 50–500 nm length) (Moon et al. 2011). CNCs have a tensile strength of 7,500–7,700 MPa, a modulus

of 110–220 GPa and a density of 1.6 g/cm³ (Moon et al. 2011). Acid hydrolysis (Zhang et al. 2021) and enzymatic hydrolysis (Xie et al. 2018) are two of the common methods of preparing CNCs. The enzymatic hydrolysis process requires a longer period than acid hydrolysis. However, acid hydrolysis involves harsh reaction conditions due to concentrated acid. During acid hydrolysis, crystalline regions of cellulose retain their structure and the amorphous parts degrade. Thus, microfibrils from cellulose chains open up and CNCs are obtained (Trache et al. 2017). Different mechanical approaches, such as ball milling, blending, high-pressure homogenization, ultrasonication, extrusion, crushing, and steam explosion have also been widely considered to mechanically crack the fibrous structure during CNC extraction. However, to

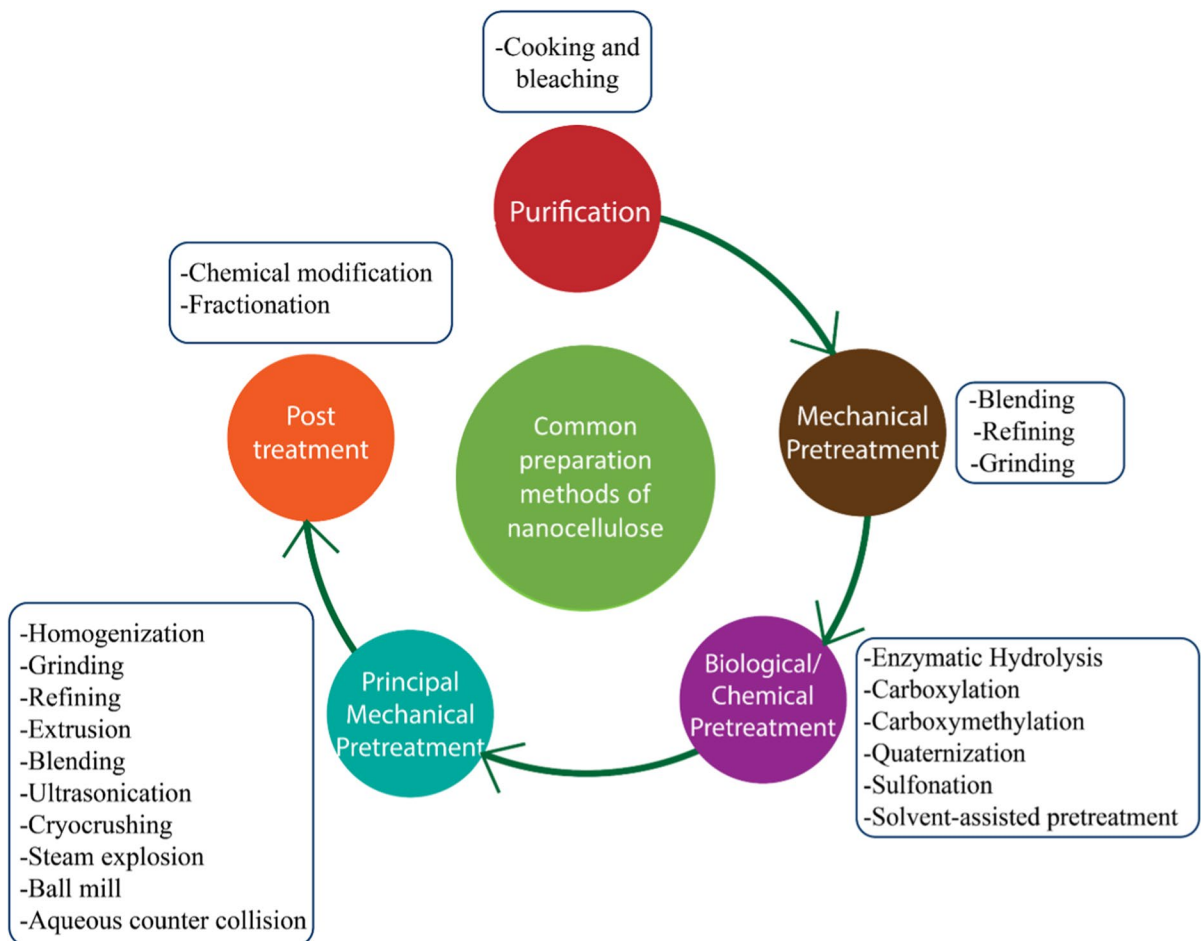


Fig. 4 Schematic of the different routes of nanocellulose production from natural resources, adapted from (Nechyporchuk et al. 2016)

avoid high energy consumption and the use of sophisticated instruments, often mechanical processes are combined with chemical techniques (Zhang et al. 2021), providing rapid extraction in less harsh conditions (Barbash et al. 2022).

Cellulose nanofibers (CNFs), also known as cellulose nanofibrils, nano fibrillated cellulose, or micro fibrillated cellulose commonly have a diameter of 5–50 nm, and can be a few micrometers long (Nechyporchuk et al. 2016). They have cellulose crystalline chains and amorphous regions with entanglement among the fibrils and generally possess a higher aspect ratio than that of CNCs. Mainly, dimension, crystallinity, aspect ratio, and strength are the key criteria to differentiate CNFs from CNCs (Tanpichai et al. 2019). Conventional methods of CNF extraction are strong mechanical processes. Some studies also showed chemical pretreatment followed by mechanical process to form CNFs. Among all the promising techniques, chemo-mechanical techniques, i.e., combined enzymatic or 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO)-mediated oxidation followed by refining, grinding, and homogenization are typically used as

mechanical fragmentation techniques for the production of CNFs (Petroudy et al. 2021).

The overall idea of preparing either CNCs or CNFs from different natural resources is summarized in Fig. 4.

Isolation of nanocellulose from WH

The extraction process of nanocellulose from WH involves initial pretreatments followed by different extraction procedures. Both cellulose nanocrystals (CNCs) and cellulose nanofibers (CNFs) can be isolated from WH (Fig. 5). A combination of mechanical and chemical processes is found more effective to achieve the destined nano-dimension of cellulose.

Extraction of CNCs from WH

The stem part of WH is mainly considered for CNC extraction since it possesses the most fraction of cellulose (Table 1). In most studies, to transform dried WH stem into a chopped or powdered form, a primary mechanical process (such as chopping, grinding or

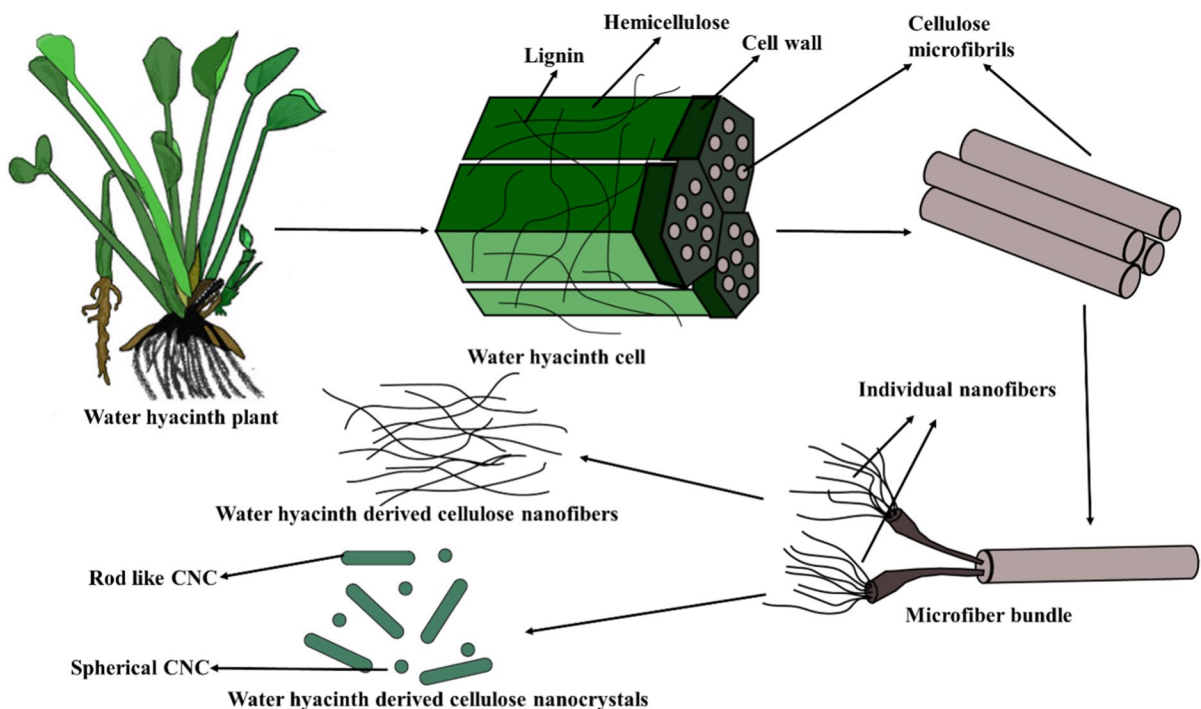


Fig. 5 A schematic diagram of CNC and CNF extraction from WH.

milling) is common. The powdered or chopped WH is considered as a starting material for CNC extraction by chemical treatment. Typically, a chemical pretreatment consists of alkaline treatment (Packiam et al. 2022) and/or Soxhlet extraction to remove wax and impurities, and bleaching (such as treating with NaClO_2 in acidic conditions) as a delignification step (Sundari and Ramesh 2012). Hemicellulose is also removed within these treatments, such as by alkaline treatment as well as acidic bleaching (Abdel-Fattah and Abdel-Naby 2012). These treatments give the fibers a white (Oyeoka et al. 2021) or opaque-white look (Juárez-Luna et al. 2019) indicating the successful removal of the non-cellulosic part from WH. In some cases, bleaching was conducted after the acid hydrolysis to get the CNCs (Asrofi et al. 2017a). For instance, a NaOH (25% w/v) assisted pulping was done in a high-pressure reactor (130 °C, at a pressure of 4 bar for 6 h) to carry out the primary pretreatment operation. Then the hydrolysis was done using HCl followed by bleaching and ultrasonication as the final treatment (Asrofi et al. 2017a). Aside from the mentioned methods, in one study, CNCs from WH were prepared by hydrolysis using cellulase enzyme (Juárez-Luna et al. 2019).

Commonly, hydrolysis using different acids (such as HCl, H_2SO_4), TEMPO-mediated oxidation, and the use of ionic liquids and metal salts are widely used for CNC extraction from different cellulosic resources. However, in the case of WH, the studies are limited to the acid hydrolysis mostly by HCl, with a few exceptions of using H_2SO_4 and enzymatic hydrolysis. In some studies, CNCs were achieved directly after acid hydrolysis (Kusnan and Setiadi 2020), while in some other studies, some chemo-mechanical processes such as ultrasonication (Syafri et al. 2019b) or cryo-crushing (freezing and crushing in liquid nitrogen) after chemical treatment (Ramos-Vargas et al. 2020) were considered to acquire the final CNCs. The diameter of CNCs achieved in some key reports is listed in Table 3.

Researchers often have coupled mechanical and chemical processes to optimize the isolation process of CNCs. For example, Asrofi et al. proposed an ultrasonication-assisted acid hydrolysis technique to obtain CNCs. After initial alkaline treatment, subsequent grinding (3000 rpm) and wet blending (20,000 rpm) were carried out to open more surface area for effective hydrolysis of the cellulose chain. Following acid hydrolysis (5M HCl) and bleaching

Table 3 Common extraction processes of CNCs from WH reported in different studies

Reference	Mechanical process	Chemical treatment	Nanocellulose diameter (nm)
(Juárez-Luna et al. 2019)	Milling	Enzymatic hydrolysis (Cellulase)	15.6–29.4
(Asrofi et al. 2017a)	Cutting	Alkaline treatment → (NaOH) → Acid hydrolysis (HCl) → Delignification ($\text{NaClO}_2/\text{CH}_3\text{COOH}$)	10–40
(Asrofi et al. 2018b, c; Syafri et al. 2019b)	Cutting	Alkaline treatment → (NaOH) → Delignification ($\text{NaClO}_2/\text{CH}_3\text{COOH}$) → Acid hydrolysis (HCl)	15–15.61
(Kusnan and Setiadi 2020; Asrofi et al. 2018b, c)	Blending	Soxhlet extraction (Ethanol + Toluene) → Delignification ($\text{NaClO}_2/\text{CH}_3\text{COOH}$) → Alkaline treatment (NaOH) → Acid hydrolysis (HCl)	100–200
(Oyeoka et al. 2021)	Milling	Alkaline treatment → (NaOH) → Delignification ($\text{NaClO}_2/\text{CH}_3\text{COOH}$) → Soxhlet extraction (Ethanol-Benzene) → Acid hydrolysis (H_2SO_4)	20–50
(Packiam et al. 2022)	Cutting	Alkaline treatment (NaOH) → Delignification ($\text{H}_2\text{O}_2/\text{CH}_3\text{COOH}$) → Acid hydrolysis (HCl)	93.02
(Ramos-Vargas et al. 2020)	Homogenization	Demineralization (H_2SO_4) → Delignification ($\text{MgSO}_4/\text{ClO}_2$) → Alkaline treatment (NaOH) → Acid hydrolysis (H_2SO_4)	8–10

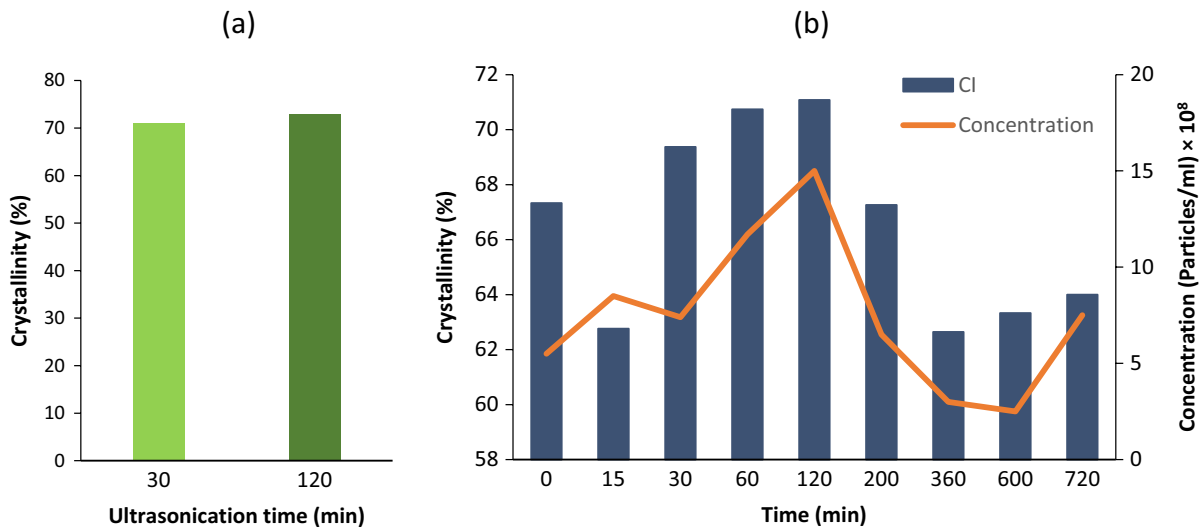


Fig. 6 Deviation in cellulose crystallinity index by the influence of **a** ultrasonication time, plotted using data from (Asrofi et al. 2017a) and **b** impact of cellulase enzyme treatment time

on crystallinity index and concentration of extracted nanocellulose, plotted using data from (Juárez-Luna et al. 2019)

($\text{NaClO}_2 + \text{CH}_3\text{COOH}$), ultrasonication was done for 2 h (energy not specified) to obtain CNCs from WH. It was found that the crystallinity index was raised from 7% (raw WH) to 73% in the final CNCs (Asrofi et al. 2017a). It is important to carry out homogenization (such as by ultrasound vibration) to individualize large cellulose aggregates, as the fibers tend to aggregate with each other.

These aggregates probably do not affect the crystallinity index of cellulose. For example, the crystallinity index of cellulose was mostly unchanged (71% compared to 73%) when only 30 min ultrasonication (Juárez-Luna et al. 2019) was used rather than a 2-hour ultrasonication (Fig. 6a) (Asrofi et al. 2017a). However, often these results were not statistically analyzed, limiting a clear justification. Nevertheless, ultrasonication is reported to have reduced the final diameter of CNCs, probably due to the separation of individual fibers by continuous vibration. For instance, Syafri et al. reported WH CNCs where the diameter of CNCs was found 15 nm after ultrasonication (600 W, 1 h) which was seen as a form of short microfibrils (2 μm diameter) after acid hydrolysis only (Syafri et al. 2019b).

Although acid hydrolysis is more popular and convenient, enzymatic hydrolysis was also reported for CNC synthesis from WH. Enzymatic hydrolysis is advantageous as it requires less processing and

nanocellulose is produced in a single step. For example, Juárez-Luna et al. demonstrated the effect of enzymatic hydrolysis time while synthesizing CNCs from cellulose derived from WH. A citric acid buffer was used to maintain pH 5 and cellulase enzyme was applied for different treatment duration (15–720 min). The nanocellulose was separated from the solution by centrifugation. The maximum concentration of CNCs was obtained by 120 min of enzymatic hydrolysis and a further hydrolysis time with cellulase enzyme led to more cellulose loss (Juárez-Luna et al. 2019). After hydrolyzing the amorphous portion of WH fibers, further reaction time may start to destroy the crystalline portion of CNCs, thus finding an optimum point is important for this kind of treatment. This hydrolyzing effect over time showed a clear influence on the crystalline property and concentration of CNCs which can be observed in Fig. 6b.

Nevertheless, for the extraction of CNCs from WH, many other effective chemical processes are still unused (such as using ionic liquids, metal salts, and TEMPO) which needs future consideration when extracting CNCs from WH.

Extraction of CNFs from WH

Like CNCs, the extraction of CNFs from WH is also conducted using the cellulose-rich stem part (Sahlie

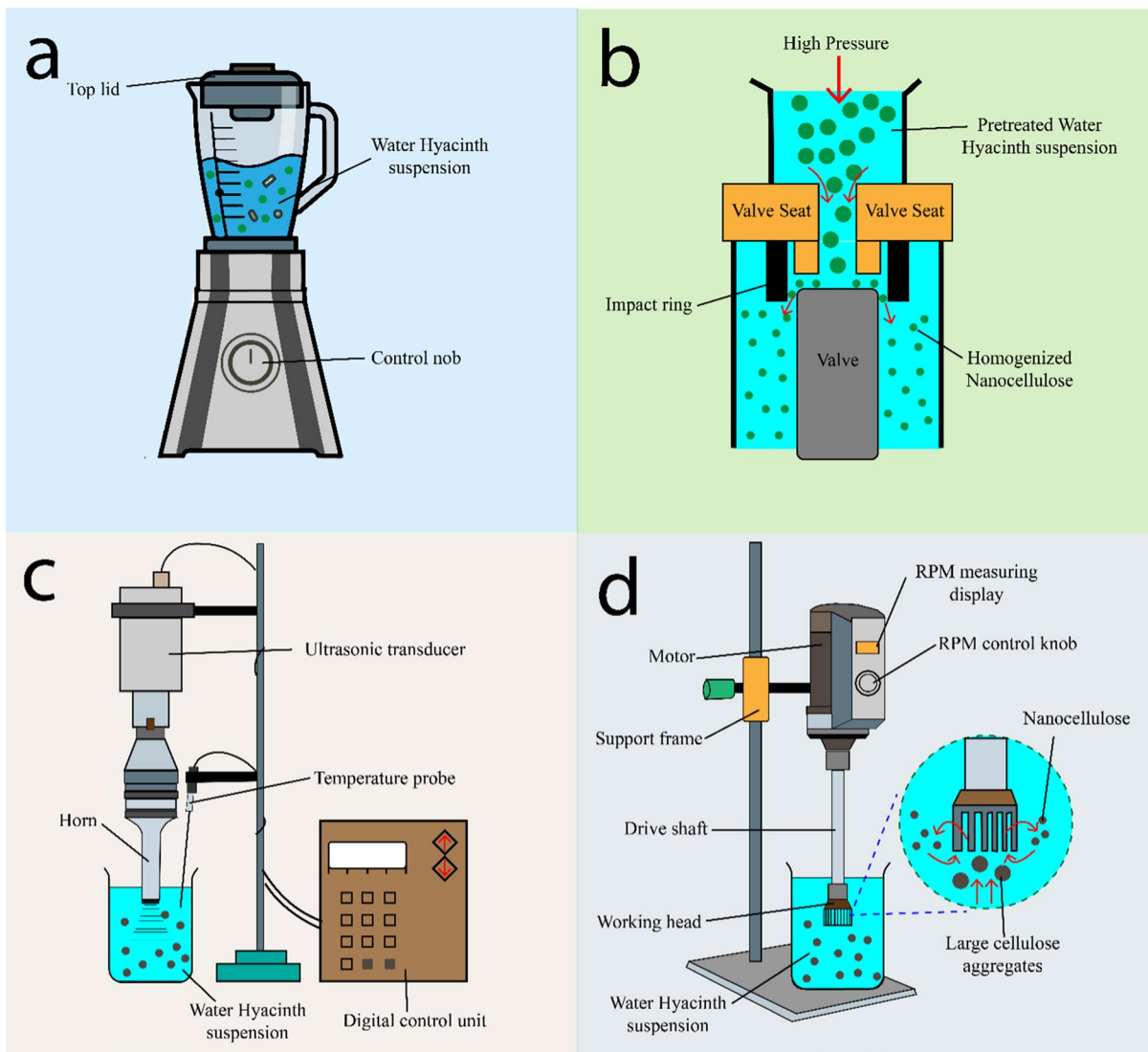


Fig. 7 Different mechanical treatments used for the extraction of nanofibers from WH. **a** High-speed blending, **b** High-pressure homogenization, **c** Ultrasonication, **d** Rotor-stator type homogenization

et al. 2022). Unlike the CNC extraction processes discussed in the last section, strong acid hydrolysis was often avoided for CNF fabrication to inhibit the breakdown of the cellulose chain and get longer nanofibrils. Some chemical processes like alkaline treatment and delignification were common, but achieving the final CNFs was largely dependent on mechanical processing. For example, Sundari and Ramesh obtained CNFs from WH through a hybrid method of cryo-crushing with liquid nitrogen followed by sonication. The diameter of CNFs achieved

by this process was 20–100 nm (Sundari and Ramesh 2012). Some of the commonly used mechanical treatments for producing CNFs from WH include blending, high-pressure homogenization, ultrasonication, rotor-stator type homogenization (Fig. 7) and ball milling. While mechanical approaches like ultrasonication and high-pressure homogenization have gained much popularity, mechanical pretreatments such as blending and grinding also appeared as the prerequisites during the disintegration process as they are

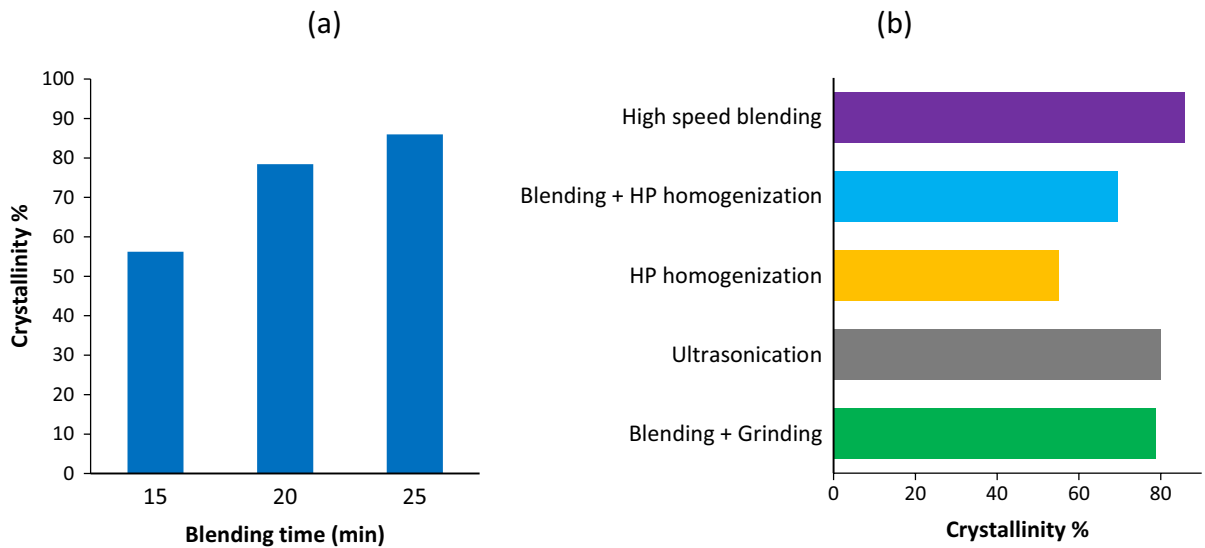


Fig. 8 Changes in crystallinity index of nanocellulose, **a** with increasing duration of high-speed blending (Asrofi et al. 2017b), and **b** its comparison with values reported from different mechanical approaches; high-pressure (HP) homogeniza-

tion (Sun et al. 2020), blending and grinding (Tanpichai et al. 2019), blending and HP homogenization (Pakutsah and Aht-Ong 2020) and ultrasonic crushing (Asrofi et al. 2018a)

chemical-free and deliver particles with an increase in the inner surface.

High-speed wet blending in conjunction with homogenization is a useful method of preparing WH CNFs. Asrofi et al. used a wet blending technique after the alkaline treatment of WH in a high-pressure reactor to obtain CNFs from UV-dried WH stems (Asrofi et al. 2017b). After subsequent washing, multiple cycles of wet blending were done in a commercial blender (25,000 rpm) for the defibrillation of CNFs. It was found that wet blending time has a significant impact on the characteristics of produced CNFs. The increased wet blending time leads to a significant reduction in diameter, i.e., 1 μm in 15 min to 50–100 nm in 25 min. It also showed an increase in the cellulose crystallinity with the increase of wet blending time over 25 min of the experiment (Fig. 8a). The paper formed by CNFs also showed a tensile strength of 18.41 MPa at a wet blending time of 15 min which further increased to 23.41 MPa when the wet blending time was set to 25 min (Asrofi et al. 2017b). In another work, the same group of authors reported two-stage alkaline and acid hydrolysis treatments to isolate smaller and more uniform CNFs (Asrofi et al. 2018c). After bleaching, the WH was subjected to double acid hydrolysis in HCl, i.e., 5 M

in the first stage and 3.5 M in the second stage, followed by ultrasonication (20 kHz, 600 W) to sonicate the fiber suspension. The crystallinity index of raw WH fiber was only 5% which increased to 80.45% in the final sonicated fiber. However, it was also noted that the sonication process slightly reduced the crystallinity index, compared to the crystallinity index of cellulose after acid hydrolysis (84.87) (Asrofi et al. 2018c).

As a common trend, an alkaline treatment was used followed by a bleaching step as the pretreatments. In some studies, bleaching has been carried out before the alkali treatment. For example, Sundari et al. reported a multi-step mechanical process for the CNF extraction from WH, where 3 wt% sodium chlorite (bleaching) was used initially. After that, WH was treated with alkali (1 wt% NaOH) and then again brought to a bleaching treatment by using 1 wt% sodium chlorite. After this subsequent bleaching-alkaline-bleaching treatments of the WH, ball milling, cryo-crushing and finally ultrasonication were applied to isolate the CNFs (Sundari and Ramesh 2012). In some studies, mechanical pretreatment was considered before chemical treatment. For example, Sun et al. reported mechanical pretreatments (blending, rotor-stator mixing, high-pressure homogenizing)

before the alkaline treatment and bleaching processes. The dried WH was first crushed with a household blender and then the mixture was subjected to high shear using a rotor-stator mixer (13,000 rpm) and then a high-pressure homogenizer. After that, subsequent bleaching-alkali-bleaching treatments were carried out as chemical processes. Then the produced mixture was subjected to high-pressure homogenizing. A higher cycle of homogenizing led to the isolation of CNFs but the crystallinity index slightly decreased by this process (Sun et al. 2020). Interestingly, the diameter of CNFs (25 nm) achieved from the study of Sundari et al. (chemical treatment followed by mechanical treatment) (Sundari and Ramesh 2012) was identical to the diameter of CNFs (19.2 nm) reported by Sun et al. (mechanical treatment followed by chemical treatment), despite the methods were different (Sun et al. 2020).

High-pressure homogenization could help the defibrillation of WH by increasing the inter- and intra-fibrillar distance through high shear force in the reaction chamber. However, a higher defibrillation rate may not always improve the characteristics of nanocellulose, and the mechanical defibrillation cycles can impact the final properties of CNFs. Pakutsah et al. reported a two-steps defibrillation technique for isolating CNFs from WH, where after a mild alkali treatment and bleaching, a mechanical defibrillation cycle was done in a varied number of cycles to identify the impact of this mechanical force (Pakutsah and Aht-Ong 2020). An initial mild defibrillation was conducted in a high-speed blender (38,000 rpm) and then the final defibrillation was achieved in a high-pressure homogenizer at a fixed pressure (15,000 psi). The number of cycles in the homogenizer varied from 5 to 30. The increasing number of cycles did not affect the yield that much, i.e., 97% in 5 cycles, and 95% in 30 cycles), but crystallinity decreased from 70.19 to 66.42% for a defibrillation cycle of 5 and 30, respectively. Further, a gradual decline was also observed in thermal stability and degree of polymerization (Pakutsah and Aht-Ong 2020). It was found that a higher defibrillation cycle (30 cycles) led to a higher water retention value of 231.2%. Such increment by water retention high-pressure homogenizing was in agreement with a related work reported by Sun et al. (Sun et al. 2020), as the surface area is likely to increase with increasing defibrillation. Pakutsah and Aht-Ong (2020) also reported an improved specific

surface area of WH CNFs, i.e., 6.6 m²/g to 21.99 m²/g by 5 and 30 defibrillation cycles, respectively.

These results indicated a diverse impact of mechanical treatments on the defibrillation and crystallinity of CNFs collected from bulk WH. Such an assertion is further proved in Fig. 8b where comparative results of the achieved cellulose crystallinity of CNFs are shown among different mechanical methods. Interestingly, high-pressure homogenizing resulted in a lower crystallinity compared to some simpler techniques, such as high-speed blending and ultrasonication. However, this should also be related to the impact of other associated chemical and mechanical processes in the relevant studies, which needs a better understanding.

In a recent study, Tanpichai and Wimolmala (2022) reported a mechanical approach of high-speed homogenization where TEMPO was used for the initial oxidation of cellulose. The composite prepared by reinforcing such CNFs resulted in a slight decrease in thermal stability and an increase in tensile strength and wettability due to the formation of large amounts of hydrogen bonding between smaller pores of nanofibrils (Tanpichai and Wimolmala 2022). In another study, the impact of two different chemical treatments (such as alkaline treatment compared to combined acidified NaClO₂ and alkaline treatment) on the produced CNFs was shown by comparative analysis (Tanpichai et al. 2019). The results indicated that CNFs prepared using those approaches exhibited almost similar mechanical properties, morphology, crystallinity, and thermal characteristics but showed a difference in lignin and hemicellulose content (related to the difference in the extraction methods) and yield. But there is no following study on this to justify the reason behind the minor impact of lignin and hemicellulose content on essential properties of CNFs, such as strength and crystallinity. Moreover, many other useful chemical methods for preparing CNFs, such as carboxymethylation, sulfation, and succinylation are not yet studied with WH. This could be interesting to reveal how the cellulose-rich WH interacts with those chemical reactions and facilitate the overall extraction processes. Hence, the scope is unlimited to explore more effective extraction methods of WH CNFs, using more advanced techniques. Table 4 summarizes the common methods used for CNF extraction till now and the diameter achieved for the resultant nanofibers.

Table 4 Different combinations of chemical and mechanical processing used for CNF extraction from WH.

Reference	Chemical treatment	Mechanical process	Nanocellulose diameter (nm)
(Asrofi et al. 2017b)	Alkaline treatment (NaOH)	Wet blending	50–100
(Sundari and Ramesh 2012)	Soxhlet extraction (ethanol + toluene) → Delignification (NaClO ₂ /CH ₃ COOH) → Alkaline treatment (NaOH)	Ball milling, Cryo-crushing and Ultrasonication	25
(Pakutsah and Aht-Ong 2020)	Alkaline treatment (NaOH) → Delignification (NaClO ₂ /CH ₃ COOH),	Blending and Homogenization	5–50
(Sun et al. 2020; Beg et al. 2021)	Delignification (NaClO/CH ₃ COOH) → Alkaline treatment (NaOH)	Rotor-stator mixing and Homogenization	19.2
(Asrofi et al. 2018a)	Alkaline treatment (NaOH) → Acid hydrolysis (HCl) → Delignification (NaClO ₂ /CH ₃ COOH)	Grinding, Wet blending and Ultrasonic crushing	10–20
(Tanpichai et al. 2019)	Bleaching (NaClO ₂ /CH ₃ COOH) → Alkaline treatment (KOH)	Blending and Grinding	18.4–23.7
(Ewulonu et al. 2020)	Refluxing (ethyl alcohol and nitric acid)	Ball milling, Ultrasonic homogenization	1–19
(Tanpichai and Wimolmala 2022)	TEMPO oxidation	Grinding	5–10

Impact of pretreatment during nanocellulose isolation

Isolation of nanocellulose requires the removal of WH impurities and extraction of pure cellulose and transformation into nanocellulose. Therefore, this is obvious that the practices involve the generation of in-process residues, such as hemicellulose and lignin, and possibly some parts of cellulose. Since the prospects of nanocellulose are undeniable, a low-lignin source like WH could be a suitable choice for nanocellulose extraction since it already contains a lower amount of impurities and is likely to deliver a higher yield.

Pretreatment is an essential step for both CNC and CNF fabrication, which often becomes a key influential factor in the cleanliness of the process. pretreatment is necessary to efficiently disrupt the complex and recalcitrant lignin and hemicellulose from WH. But an intensive pretreatment can reduce the process yield significantly (including the loss of cellulose) and produce more residue. Therefore, the pretreatment method should be cost-effective and simple but also should confirm low consumption of energy, chemical, and water during the disintegration of cellulose. A series of pretreatment is commonly needed to remove lignin and hemicellulose before cellulose disintegration from other agricultural wastes. WH

having a limited amount of lignin, requires fewer steps which may reduce the cost of its nanocellulose extraction process (Tanpichai et al. 2019).

In general, pretreatments are mechanical (such as chopping, grinding, and milling), chemical (such as alkali treatment, Soxhlet extraction, and delignification), physiochemical (such as steam explosion and CO₂ explosion), and biological (ligninolytic enzymes) (Sharma and Aggarwal 2020). Among these, a couple of methods were used separately or collectively to pretreat WH.

As perceived from Tables 3 and 4, the chemical pretreatments of WH, particularly with NaOH and with NaClO₂ in acidic conditions (for bleaching or delignification) are some of the common steps. In this context, an interesting study by Abdel-Fattah and Abdel-Naby (2012) showed how different combinations of chemicals can affect the loss of WH (Abdel-Fattah and Abdel-Naby 2012). Among different pre-treatment conditions, a combination of NaClO₂/NaOH showed no cellulose or hemicellulose loss, but lignin loss (83.7%) was lower than other methods, indicating a low in-process residue generation. Some recent studies proposed to preserve native lignin for the benefit of antimicrobial and ultraviolet shielding properties of lignin during preparing nanocellulose from agricultural waste (Zhang et al. 2021). However, most of the WH studies did not address this point, and

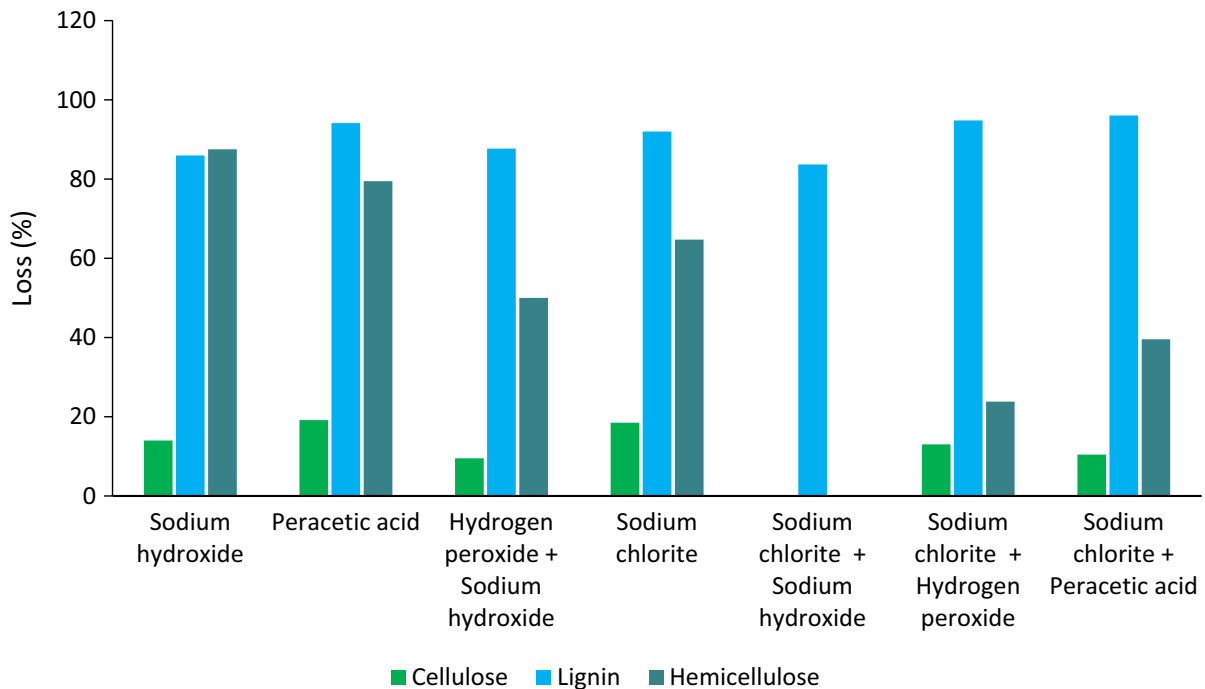


Fig. 9 Loss of cellulose, hemicellulose, and lignin by different chemical pretreatments of WH, plotted with data from (Abdel-Fattah and Abdel-Naby 2012)

the removal of lignin was attempted. Nevertheless, as the removal process of lignin also introduces some cellulose loss, at least a judicious selection of chemical combinations can reduce this amount of loss. For example, Abdel-Fattah and Abdel-Naby (2012) reported pretreatment with NaClO_2 and peracetic acid removed 96% of lignin with 10.5% of cellulose (Abdel-Fattah and Abdel-Naby 2012). This pretreatment method of WH was found more sustainable compared to other chemical treatments where either a lower lignin removal or a higher cellulose loss was seen. Thus, it is required to optimize the pretreatment process of WH to get the desired nanocellulose more effectively, though there has been limited focus on this till now (Fig. 9).

The processes reported involved multiple washing cycles in between the chemical treatments (to reach neutral pH). However, the recollection or reuse of water was not considered. There was no indication of how the alkaline or acidic water was discarded. Further, in most cases, the yield % of the process was not reported, thus the process efficiency cannot be justified. In some cases, the yield % was very low, such as 19.38% only (Sun et al. 2020) and 33%

(after bleaching) (Pakutsah and Aht-Ong 2020). This was because of the chemical treatments that led to uncontrolled hydrolysis of the cellulose polymer chain (Salimi et al. 2019). However, with the use of mechanical processes, these can be improved as in that case, high mechanical shear defibrillates the large cellulose aggregates and turns them into nanocellulose. For instance, a yield of 83.1% was reported where mechanical processes (blending and grinding) were involved and no chemical treatment was carried out to prepare the nanocellulose (Tanpichai et al. 2019). These results suggest that the cellulose-rich and low content lignin of WH can be a useful resource to extract nanocellulose in a more sustainable way.

Comparison of nanocellulose derived from WH and other resources

Table 5 shows the diameter and crystallinity of nanocellulose reported from different lignocellulosic waste and wood compared to that of WH nanocellulose.

Table 5 A comparative scenario of nanocellulose diameter and crystallinity of isolated nanocellulose from different agricultural wastes reported in the literature

Source	Type of nanocellulose	Extraction method	Nanocellulose diameter (nm)	Crystallinity (%)	References
Wheat straw	CNFs	Acid hydrolysis (HCl)	3.91	79.87	(Kaushik and Singh 2011)
	CNCs	Acid hydrolysis (H ₂ SO ₄)	5	76.43	(Nehra and Chauhan 2022)
Rice straw	CNFs	TEMPO-mediated oxidation	1.52	70.8–71.5	(Jiang et al. 2013)
	CNCs	Acid hydrolysis (HCl)	4.8	56.12	(Razali et al. 2021)
Rice husk	CNCs	Acid hydrolysis (H ₂ SO ₄)	10–15	82.8	(Nang An et al. 2020)
	CNCs	Acid hydrolysis (H ₂ SO ₄)	11.7–48.3	58.8–76.6	(Rashid and Dutta 2020)
Coconut husk	CNCs	Acid hydrolysis (H ₂ SO ₄)	10–15	76.6	(Nang An et al. 2020)
	CNFs	TEMPO-mediated oxidation	72.1	49.2–73.5	(Hassan et al. 2021)
Corn husk	CNCs	Acid hydrolysis (H ₂ SO ₄)	26.9	83.51	(Yang et al. 2017)
	CNFs	TEMPO-mediated oxidation	10.48	72.33	(Yang et al. 2017)
Corncob residue	CNCs	TEMPO-mediated oxidation	3.8	48	(Liu et al. 2017)
	CNCs	Acid hydrolysis (H ₂ SO ₄)	5.4	56	(Liu et al. 2017)
	CNCs	Acid hydrolysis (H ₂ SO ₄)	43.8	70.9	(Ditzel et al. 2017)
Wood	CNFs	TEMPO-mediated oxidation	3	–	(Isogai and Zhou 2019)
	CNCs	TEMPO-mediated oxidation	5–10	–	(Isogai and Zhou 2019)
	CNCs	TEMPO-mediated oxidation	4.2–5.4	60.4–66.9	(Li et al. 2018)
Hemp hurd	CNCs	Acid hydrolysis (H ₂ SO ₄)	16.18–27.99	–	(Zhang et al. 2022)
	CNFs	Hydrothermal process and grinding	45–98	30–40	(Tyagi et al. 2021)
Water hyacinth	CNCs	Acid hydrolysis (HCl)	10–40	73	(Asrofi et al. 2017a)
	CNCs	Enzymatic hydrolysis	15.6–29.4	71	(Juárez-Luna et al. 2019)
	CNFs	TEMPO-mediated oxidation	5–10	68.6	(Tanpichai and Wimolmala 2022)
	CNFs	High-pressure homogenization	19	71.5	(Tanpichai et al. 2023)

The extraction processes are also listed to provide a reliable comparison.

The morphology of nanocellulose is commonly observed through microscopic techniques, such as SEM (scanning electron microscopy), TEM (transmission electron microscopy) and AFM (atomic force microscopy), where the diameter perceived is considered a key characteristic. The morphology of CNFs prepared by TEMPO-mediated oxidation mostly showed a finer diameter, such as 1.5 nm from rice straw (Jiang et al. 2013), 3 nm from wood (Isogai and Zhou 2019), 10.48 nm from corncob (Yang et al. 2017). This was also consistent with the diameter of CNCs obtained using TEMPO-mediated oxidation, such as 3.8 nm from corncob (Liu et al. 2017) and 4.2–5.4 nm from wood (Li et al. 2018). In this regard, the diameter of WH nanocellulose was reported in

a similar range, i.e., 5–10 nm when using a similar technique (Tanpichai and Wimolmala 2022). However, through the HCl hydrolysis, the diameter of WH nanocellulose ranged 10–40 nm (Asrofi et al. 2017a), which is much higher than the diameter of nanocellulose reported from wheat straw (3.91 nm) (Kaushik and Singh 2011) and rice straw (4.8 nm) (Razali et al. 2021). Further, nanocellulose diameter at a low range was also frequently reported by sulphuric acid hydrolysis of different waste resources, such as wheat straw (5 nm) (Nehra and Chauhan 2022), corncob residue (5.4 nm) (Liu et al. 2017), rice husk and coconut husk (10–15 nm) (Nang An et al. 2020). Overall, the morphology of WH nanocellulose showed a moderate diameter range. Among the methods used for WH, TEMPO-mediated oxidation appeared to have a

better impact on reducing the diameter, and producing nanocellulose comparable to other resources.

The crystallinity of WH nanocellulose was mostly comparable to that of the literature which can be observed from Table 5. From some resources, a lower crystallinity of nanocellulose was perceived, such as hemp hurd (30–40%) (Tyagi et al. 2021), corn-cob (48–56%) (Liu et al. 2017), rice straw (56.12%) (Razali et al. 2021), but nanocellulose from WH showed consistently higher crystallinity index from all reports (68.6–73%).

Apart from the morphology and crystallinity, the thermal stability of WH nanocellulose was also found satisfactory compared to that of the literature. For example, nanocellulose prepared by TEMPO-mediated oxidation of WH showed the main thermal degradation peak at 319.4 °C (Tanpichai and Wimolmala 2022). This was observed higher than nanocellulose prepared using the same method from many different common resources, such as aspen pulp (224 °C) (Li et al. 2018), kraft pulp (230 °C) (Zhong et al. 2020), hemp stalks (245 °C) (Kassab et al. 2020), denim waste (250) (Zhong et al. 2020), lemon seeds (256.1 °C) (Zhang et al. 2020), denim waste (250 °C) and bleached cotton (255 °C) (Zhong et al. 2020), corn husk (279 °C) (Yang et al. 2017), the spruce pulp (293 °C) (Li et al. 2018), olive chips (298 °C) (Sánchez-Gutiérrez et al. 2020), and rice straw (269–302 °C) (Jiang et al. 2013).

Overall, the results of morphology, crystalline and thermal properties of WH nanocellulose in comparison to that of other cellulosic resources indicate a good justification for selecting WH as a nanocellulose resource.

Applications of WH nanocellulose

To date, most of the applications proposed for WH nanocellulose are as reinforcement in different composite preparation. These include starch-based biocomposites (Syafri et al. 2019b), composites with synthetic polymers and natural rubber. The starch-based composites were prepared through the solution casting method and mainly suggested as eco-friendly food packaging materials, such as packaging bags (Syafri et al. 2019b). An increase in the mechanical strength in composite by WH nanocellulose inclusion was evident from different reports. For example,

Syafri et al. reported a tensile strength of 10.23 MPa in the composite, improving from 3.77 MPa from the starch film (Syafri et al. 2019a). This was confirmed by another study where the tensile strength was increased up to 480% by the inclusion of only 1% of WH nanocellulose (Asrofi et al. 2018b). Another relevant study showed the strength of starch/WH nanocellulose composite can be further increased to 185% by 60 min of ultrasonication of the solution before film preparation (Asrofi et al. 2018a, c). The mechanical and thermal characteristics were found comparable to that of commercial plastics currently used for food packaging (Asrofi et al. 2018b). Aside from starch as the matrix, WH CNFs were used with recycled paper pulp, which was also found to enhance the thermal and mechanical properties of the final paper produced (Sahlie et al. 2022). CNFs from WH were also proposed as fillers in epoxidized natural rubber using compression molding (Tanpichai et al. 2023). This was found effective in oil resistance and resulted in enhanced tensile strength (80%) and modulus (39%), compared to pure epoxidized natural rubber.

Nanocomposites from WH were recently reported for reinforcement in the synthetic polymer matrix, such as ethylene vinyl acetate (EVA) and polyvinyl alcohol (PVA). A good interfacial interaction between CNFs and EVA was claimed which increased the tensile strength (9.6%) and modulus (29.1%) of the composites, without changing the thermal stability and transparency (Tanpichai and Wimolmala 2022). Besides, WH CNCs were found to reduce water absorption, moisture uptake and vapor permeability when reinforced in PVA/gelatin nanocomposite film for possible food wrappers (Oyeoka et al. 2021).

Apart from the use in composites, some other uses of WH nanocellulose were reported in recent years. These include wet spinning, preparation of conductive aerogel, battery separators, nano paper and adsorbent for removing metal ions. Wet-spun WH nanocellulose filaments were claimed to have a greater tolerance to elongation (14.5%) compared to cellulosic filaments (5–7.5%) (Kusnan and Setiadi 2020). A conductive aerogel was reported by Ewulonu et al. by covering WH CNFs with polypyrrole and jelly polyvinylpyrrolidone. The aerogel showed a conductivity ranging from 0.1 to 6.23 S/cm and was suggested to be useful for microelectronics devices. Tanipichai et al. developed a vacuum-filtered heat-pressed WH CNFs-filled nano paper which showed a tensile strength of

73.3 MPa (Tanpichai et al. 2019). Overall, the nano paper reported from WH showed limited thermal expansion compared to some common plastics, e.g., polycarbonate, polyethylene terephthalate, polyether sulfone, as well as nano paper produced from other natural sources, such as chitin and kenaf.

Despite some studies showing an improved hydrophobicity by WH nanocellulose inclusion, the opposite, i.e., better wettability was also reported when CNFs from WH were used in the battery separator. For instance, the contact angle of a liquid drop was reported as 51.84° when using 95% CNFs which was claimed to help efficient transportation of aluminium ions (Beg et al. 2021). WH nanocellulose (both CNFs and CNCs) was also used as a filtration medium for heavy metal (Pb) adsorption from textile wastewater treatment (Ramos-Vargas et al. 2020). Results showed a maximum Pb removal capacity of 87.1 and 30.36 mg/g by CNFs and CNCs, respectively when a moderate temperature (45 °C) was applied.

Such a wide range of applications and promising results observed from WH nanocellulose have opened many new opportunities to be explored in future which are discussed in the following section.

Future scope

Despite WH nanocellulose being proposed in diverse sectors, a wide array of opportunities is yet unrevealed. For example, the composites prepared from WH nanocellulose were suggested for environmentally friendly packaging (Asrofi et al. 2018c), solar cell panels, display electronics (Tanpichai and Wimolmala 2022), textile, automobile, and medical devices (Tanpichai et al. 2023). The true applicability of WH nanocellulose in the suggested applications needs to be tested. Besides, WH nanocellulose showed conflicting effects in different reports, i.e., better wettability (Beg et al. 2021), low moisture absorption (Asrofi et al. 2018c), and improved hydrophobicity (Oyeoka et al. 2021). A better wettability is suitable for the efficient transportation of metal ions in battery separators (Beg et al. 2021) as well as an efficient removal process of metal ions from wastewater (Ramos-Vargas et al. 2020). In contrast, hydrophobic behavior could be useful for composite preparation for packaging applications (Oyeoka et al. 2021). This is unclear whether this was related to the

difference in the nanocellulose extraction methods or the combining mechanisms and it is important to know for future uses.

The tensile strength reported for WH CNF nano paper was 73.3 MPa (Tanpichai et al. 2019), which is lower than the tensile strength (150 MPa) of randomly oriented CNF nano paper from wood resources. With a better orientation, the tensile strength of wood CNF nano paper can further reach 430 MPa (Jakob et al. 2021). Since the mechanical characteristics of cellulose nano paper depend on cellulosic resources and preparation methods, there is a great scope for improving the mechanical properties achieved from WH till now by improving the alignment of fibrils by proven methods, such as dynamic sheet forming or drawing in wet state (Jakob et al. 2021), and selecting suitable chemical treatments that are more beneficial for cellulose structure (Tardy et al. 2021).

Some of the proposed applications of WH nanocellulose are at the preliminary stages and the numbers of studies are low to justify the findings. For example, in one study, WH CNFs and CNCs were found suitable as adsorbents for heavy metals (Ramos-Vargas et al. 2020), but there is no following study to rationalize it. Similarly, despite some promise of WH nanocellulose in preparing battery separators (Beg et al. 2021), aerogel (Ewulonu et al. 2020), and wet-spun fibers (Kusnan and Setiadi 2020), there is no following study to take these benefits to the next phase.

In recent years, CNCs and CNFs derived from different natural resources have been extensively proposed for wound healing, drug delivery, and tissue engineering (Bacakova et al. 2019). Nanocellulose derived from other plants, such as kenaf (Bhatnagar et al. 2021) and sugarcane bagasse (Lam et al. 2017) have been reported to be used in wound healing and fabrication of biocompatible tissue engineering scaffolds, respectively. Wood-derived CNFs (Skogberg et al. 2017) and CNCs (Mo et al. 2015) were also frequently proposed for skin tissue engineering. However, WH nanocellulose has not been tested for biomedical applications yet. Another interesting area of using CNCs and CNFs from WH could be in the construction and building industry, where they can be used as rheological modifier and additives. Use of nanocellulose fillers from other resources (such as kenaf and oil palm) have been proven effective for reducing internal and surface porosity in building materials (Mocktar et al. 2020).

Fig. 10 An illustration depicting various potential applications of WH-derived nanocellulose



Given a good crystalline property of WH nanocellulose, this area deserves to be explored further with WH. The possible future areas for utilizing CNCs and CNFs derived from WH are illustrated in Fig. 10.

An important future aspect of preparing CNCs and CNFs from WH should be process optimization. Current processes involve multiple steps of chemical treatment that are, time-consuming (up to 72 h) and possibly low yield. In the case of industrial production, in the near future, it is imperative to develop a simple or one-step extraction method with reasonable process yield. Acid hydrolysis could be an ideal approach for WH nanocellulose, given a comparatively better yield found in previous studies. But the need for concentrated acid, high water consumption, and hazardous effluent are the main disadvantages of this method. Enzymatic hydrolysis might alleviate some disadvantages of acid hydrolysis, but the lower yield, difficult optimization of reaction conditions, and risk of disruption of the crystalline portion of nanocellulose make this process challenging for mass production. Therefore, a balanced approach is required to get an optimized quality of WH CNCs and CNFs and maintain sustainable manufacturing. Further, a thorough life cycle analysis is necessary for WH nanocellulose to understand the true impact of the entire process. In addition, only the *Eichhornia crassipes* species of WH have been studied for nanocellulose extraction till now, and the impact of

different WH species on mechanical, thermal, and crystallographic properties of CNCs and CNFs is unknown.

Economic prospects

WH is a prevalent source of cellulose and can be economically advantageous for CNC and CNF production. Proper use of WH can also save a huge cost associated with its disposal in many countries. For example, China is counting more than 12.35 million USD to control WH every year (Lu et al. 2007). Between the years 1975 to 2013, Louisiana spent 124 million USD to control this water weed (Wainger et al. 2018). In Sudan, 500 men are required to manually clean a hectare WH per day. Nigeria spends 750,000 USD annually to control WH (Lwasa and Mwanje 2006). In a study regarding cost-effectiveness evaluation on the different processes of controlling WH, it was found that it would require about 106,000 USD per month to control the WH mechanically. Further, it would require 350,000 USD, 623,000 USD, and 890,000 USD to control this aquatic weed manually, biologically, and chemically, respectively (Lwasa and Mwanje 2006).

WH is often freely available in tropical areas. Since the global market for nanocellulose is expected to rise rapidly (Jordan et al. 2019), the

demand for low-cost raw materials for nanocellulose like WH is also growing. Considering WH as a cheap and accessible source at least in the regions where its growth is favorable, this problem could turn into a profit in future with proper initiatives.

Environmental prospects

Almost all industries use wood as the primary cellulose source for nanocellulose extraction (Ribeiro et al. 2019). However, rapid deforestation and climate change might force industries to look for alternative cellulose sources. To solve this future cellulose source crisis, WH could become an ideal candidate for large-scale nanocellulose production, especially in tropical and sub-tropical countries.

Besides, considering the water pollution caused by WH, effective utilization of this biomass is important. This water weed is affecting the lake water quality and quantity, and habitation of the fish as it decreases the oxygen level of water. Human health, irrigation, navigation, agriculture, transportation, tourism, hydroelectric facilities, and many other aspects of human life are affected by WH. Hence, using it as a resource in producing necessary materials will eliminate at least some of the problems. Added to that, current sources of nanocellulose are mainly wood pulp, and some other sources like bamboo, cotton, and bast fibers, while using WH for CNC and CNF extraction will reduce cutting down trees and help maintain a greener environment. Nanocellulose membranes from natural resources including WH can also filter wastewater by removing dyes, heavy metals and contaminants including microbes (Reshmy et al. 2021; Nama-sivayam et al. 2022). Additionally, the accumulated heavy metals can easily be recovered from WH and the water weed can be used as a biofertilizer. Moreover, phytoremediation and metal reclamation, are also eco-friendly approaches for utilizing WH (Harun et al. 2021).

However, adjustment is required to the current CNC and CNF production processes reported from WH to ensure more environmentally friendly production and use of more sustainable chemicals. It is also important to consider how the effluent from nanocellulose extraction will be discarded, and what impact it would bring on the environment.

Conclusions

WH grows at an accelerated rate in waterways and troubles the environment. However, due to its low lignin and high cellulose content, this abundant plant can be effectively used for nanocellulose extraction as a low-cost alternative to current resources, such as wood, bamboo, cotton, and bast fibers. This could bring economic and environmental benefits to the WH-infested areas. Researchers have put forth several strategies to apply WH plant-based nanocellulose in bio-composite, packaging, textiles, and filtration applications. Extraction of nanocellulose from WH should require less intensive chemical treatments due to a lower amount of lignin, yet earlier studies did not take this advantage to tailor the manufacturing process more sustainably. Energy-intensive mechanical treatments, use of concentrated chemicals, and high-water consumption remain some of the drawbacks of this process, restricting mass production of CNCs and CNFs from WH. Recent interest in WH nanocellulose and the reported properties and uses for different applications indicate its promise to become a valued source, rather than just an invasive weed. However, keen attention is required in the process design to reuse water or restrict wastage, use more advanced and sustainable chemicals, and sensible use of mechanical treatments consuming lower energy. The authors expect WH may become one of the potential resources of nanocellulose in near future at least in the tropical and sub-tropical areas where this plant is abundant.

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Declarations

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