Nr 2 (73–148) LUTY 2025 Tom LXX

POLIMERY

Functionalization and stability of palm oil biomass nanocellulose – an overview

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DOI: https://doi.org/10.14314/polimery.2025.2.1

Abstract: This article presents a literature review from 2018–2024 on the issues related to the functionalization and stability of nanocellulose from palm oil biomass. The nanocellulose isolation process, methods for assessing stability and ways to increase it, as well as potential applications are discussed in detail. Emphasis is placed on the commercialization possibilities of nanocellulose-based products, with a view to developing engineering of advanced nanomaterials derived from oil palm biomass.

Keywords: isolation process, oil palm biomass, nanocellulose, stability, functionalization.

Funkcjonalizacja i stabilność nanocelulozy z biomasy oleju palmowego – przegląd literaturowy

Streszczenie: W artykule przedstawiono przegląd literaturowy z lat 2018–2024 dotyczący zagadnień związanych z funkcjonalizacją i stabilnością nanocelulozy z biomasy oleju palmowego. Szczegółowo omówiono proces izolacji nanocelulozy, metody oceny stabilności i sposoby jej zwiększenia, a także potencjalne zastosowania. Nacisk położono na możliwości komercjalizacji produktów na bazie nanocelulozy, mając na uwadze rozwój inżynierii zaawansowanych nanomateriałów pochodzących z biomasy palmy olejowej.

Słowa kluczowe: proces izolacji, biomasa oleju palmowego, nanoceluloza, stabilność, funkcjonalizacja.

Malaysia's preeminence in the oil palm sector stems from its vast plantation area and substantial number of operational palm oil mills, encompassing approximately 5.4 million hectares and 423 mills [1], standing as the world's second-largest palm oil producer and the fourth-largest contributor to the nation's Gross National Income (GNI) [2]. The continuous growth of oil palm plantations in Malaysia corresponds with the rising

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worldwide demand for edible oil uses, including cooking oil, home goods (such as detergents), personal care items, nutrition, and fuel for vehicles [3]. There are several oil palm industry organizations that represent the diverse interests of both upstream and downstream producers, including Malaysian Palm Oil Association (MPOA), Malaysian Palm Oil Board (MPOB), and Malaysian Palm Oil Promotional Council (MPOPC) [4]. MPOA consisting of more than 100 members and handling over 1.4 million hectares of oil palm plantations, which dedicated to securing the sustained profitability and growth of Malaysia's palm oil sector. Meanwhile, MPOB also known as Palm Oil Research Institute of Malaysia (PORIM), spearheads industry research and development efforts, regulatory functions and licensing activities [5]. Its research agenda emphasizes increasing oil palm productivity, optimizing biomass utilization to achieve "zero waste" and expanding palm-based products. Lastly, MPOPC plays a crucial role in promoting Malaysian palm oil via marketing communications, technical marketing, and market promotion initiatives, supported by industry contributions to the Palm Oil Promotion Fund based on production volume [6].

Palm oil mills generate approximately 12.54% of crude palm oil (CPO) from mesocarp and palm kernel oil (PKO) from kernel of oil palm fruit, however, the remaining 87.46% is dumped or left as lignocellulosic biomass (LGM) either in plantation or palm oil mill (Figure 1) [7].

The National Biomass Strategy 2020 estimated that Malaysia will generate up to 80 million tons of biomass yearly, with most of that coming from the oil palm sector. As mentioned, one of MPOB's objectives is to optimize the utilization of oil palm biomass to produce commercial goods, energy generation, and recycled inputs for plantations. Biomass leftovers from the palm oil sector now stands out as one the largest sources of non-wood LGM in Malaysia [8]. Although these are often considered waste, the numerous biomasses remain that should be viewed as "opportunities" to value-added products in the production of CPO and in oil palm plantations. Efficient utilization of biomass waste is imperative to mitigate the risk of spreading diseases that afflict oil palm plantations, such as vascular wilt, bud and spear rot, sudden wilt, red ring disease, and basal stem rot disease [9]. Among these, basal stem rot (BSR), also known as Ganoderma disease, stands out as the most significant threat. This ailment is attributable to by the pathogenic basidiomycete Ganoderma boninense, a white-rot fungus prevalent in Indonesia and Malaysia [10]. BSR drastically reduces the productive lifespan of oil palms tree and results in substantial low yield, with Malaysia alone experiencing estimated losses of more than USD 500 million due to an average mortality rate of 3.7% from the disease.

Consequently, Malaysia has taken a leading role as the producer of the oil palm biomass, leveraging advancements in nanotechnology [11]. Researchers have made significant strides in isolating oil palm-based cellulose to produce nanocellulose, thus championing the concept of zero waste. This is because nanocellulose has a high surface area, high aspect ratio, abundant hydroxyl group,

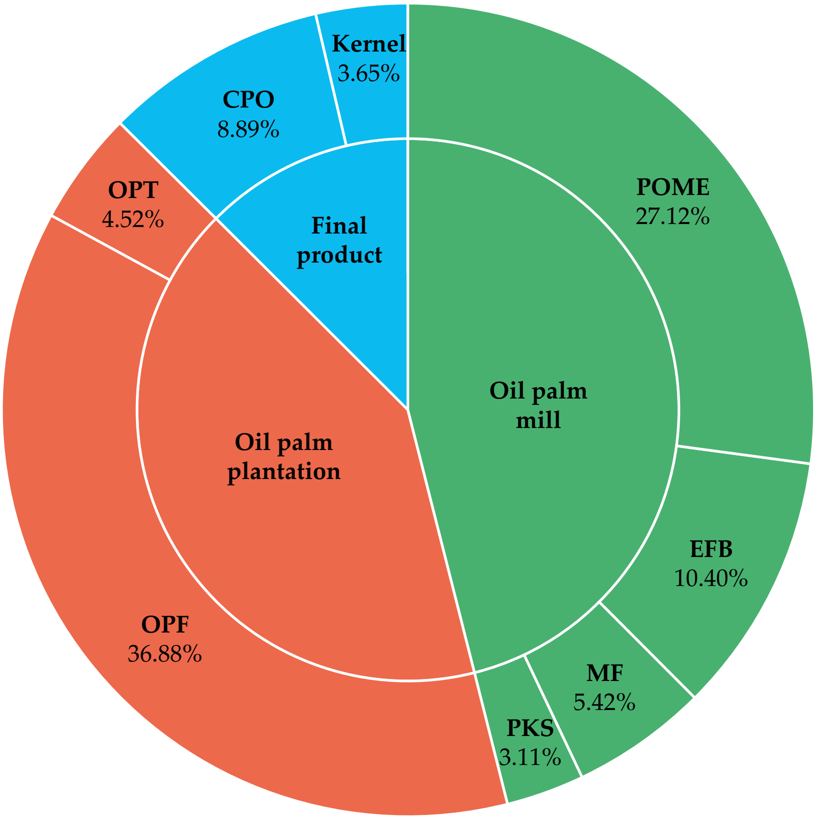


Fig. 1. Palm oil biomass usage by different branches of Malaysian palm oil industry

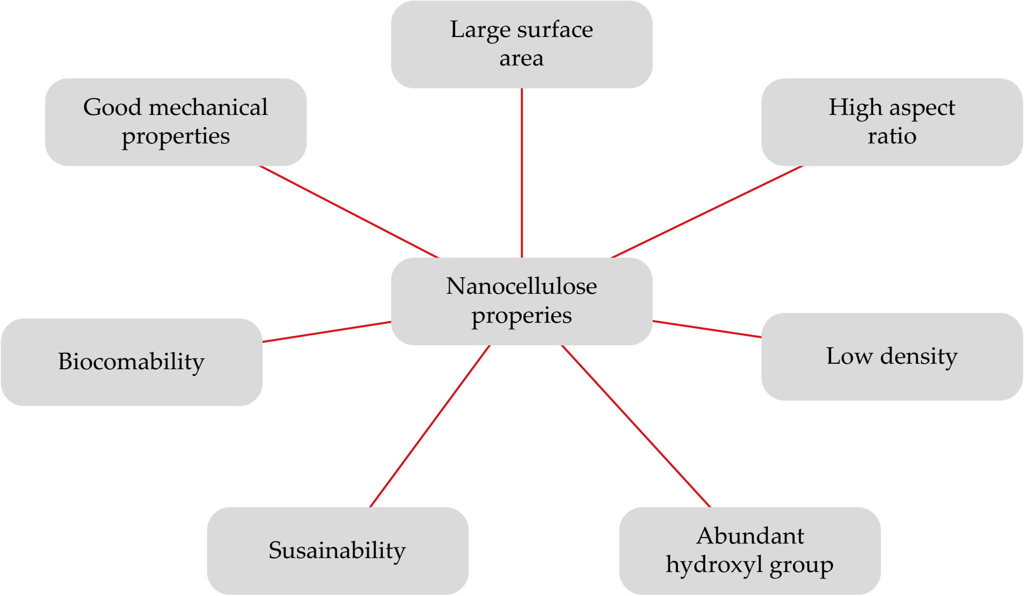


Fig. 2. Unique properties of nanocellulose

low density, suitability, biocompatibility and has great mechanical properties which impressed interest of researchers from variety of industries as demonstrated in Figure 2.

Various high-value applications stemming from oil palm biomass, including nanomaterial-based products like hydrogels, offer superior economic value compared to synthetic alternatives, which potential threats to human beings and the ecology [12]. There are two types of nanocellulose that can be isolate from oil palm based biomass which is known as nanocrystalline cellulose (CNC) and nanofibrilated cellulose (CNF), which is distinct mainly based on the isolation method such as chemical treatment and mechanical treatment respectively [13]. CNC is dominant with crystalline structure and have needle like morphology, while CNF is still consisting of amorphous and crystalline part which look similar as spaghetti-like morphology as shown in Figure 3 [14]. The fibrillation process is caused by mechanical treatments like high-pressure homogenization or microfluidization.

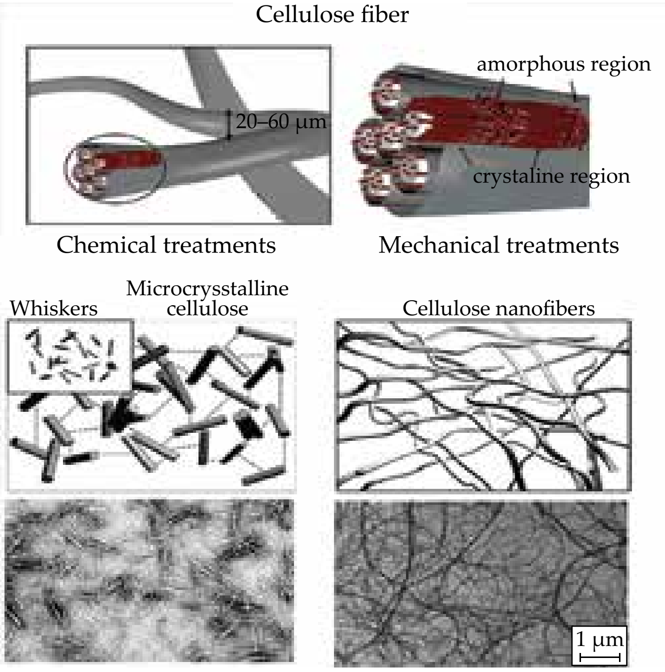


Fig. 3. Difference between CNC and CNF structure. Reprinted from [15]

This review paper gives more focus on the stability of both oil palm-based CNC and CNF. This is because there is a scarcity of information on the stability of CNC and CNF from oil palm-based biomass compared to other biomass materials. Therefore, this review paper uniquely focuses on the stability of the CNC and CNF as it is important properties that should consider when it is applicable in some specific application such as in production of nanofluid or film. Moreover, this review paper gives an overview of the important characterization for the stability evaluation and the ways to overcome this issue to revolutionize industries while concurrently addressing environmental concerns.

SOLID AND LIQUID WASTE FROM OIL PALM MILL

Figure 4 depicts the three basic anatomical parts of oil palm tree: OPF (leaves), oil palm fruit bunch (fruit), and OPT (trunk) [16]. The weight of a typical oil palm fruit bunch ranges from 10 to 40 kg, containing a single seed (palm kernel) covered with soft and oily pulp (mesocarp). After the oil palm fruit bunches are collected from the plantations or estates, they are swiftly transported to the nearest palm oil mills for processing [17]. At the mills, the oil palm bunch undergoes grading and processing and undergoes sterilization to deactivate enzymes as an initial step. After the CPO and PKO extraction, OPEFB, MF,

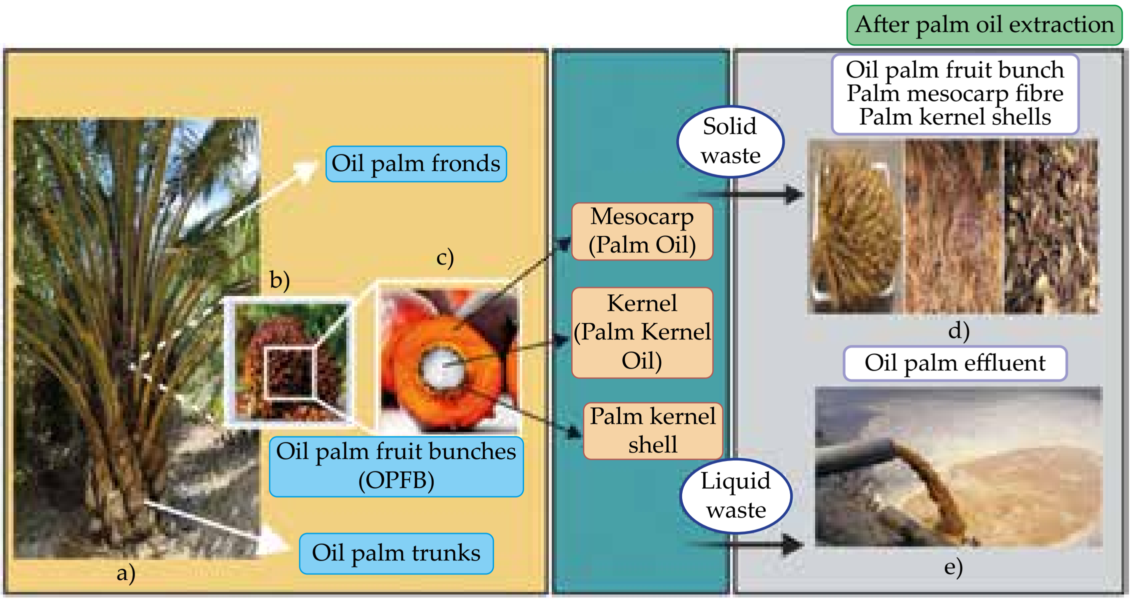


Fig. 4. Anatomy of a palm tree and waste generated after palm oil extraction: a) palm tree, b) oil palm fruit bunch, c) oil palm fruit, d) solid waste from OPF in the milling process, e) liquid waste [mill effluent (POME)] from the milling process. Reprinted from [11]

and PKS are the leftover solid waste that obtain from oil palm bunches in palm oil processing mill. While, POME is a liquid waste that produce during the palm oil extraction, typically repurposed as organic fertilizer in plantations after treatment [18]. Besides that, OPF and OPT are examples of leftover solid wastes in plantation field.

Potential of oil palm solid waste to nanocellulose

In recent decades it has been proved that solid waste produced by this industry holds significant potential for conversion into nanocellulose, offering diverse applications [1, 16]. OPEFB are particularly noteworthy, being regarded as one of the most economically viable natural fibers with desirable properties. They present a cost-effective alternative raw material, especially in industries where traditional wood plants are financially burdensome. Furthermore, OPEFB is classified as a fibrous crop waste, or lignocellulosic residue, and may be converted into pulp, which commercially available product in Malaysia. This pulp boasts a high cellulose fiber content of approximately 35%, rendering it suitable to produce value-added goods. Moreover, OPEFB comprises two distinct fiber types, known as stalk and spike, as illustrated in Figure 5 after being press-shredded OPEFB [19]. The bunch consists of a primary stem known as the stalk and plenty spikelet on its surface.

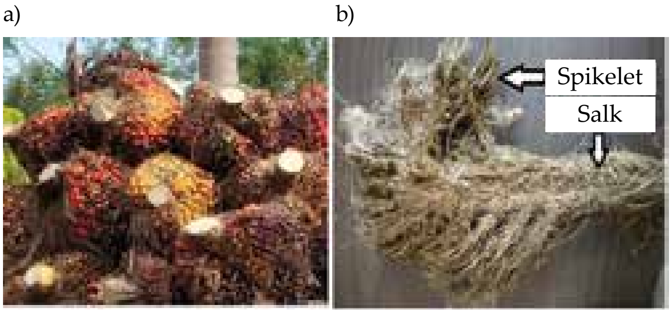


Fig. 5. Images: a) oil palm fruit bunch, b) oil palm empty fruit bunch composed of spikelet and stalk. Reprinted from [20]

A research by Reneta Nafu *et al.* [21] discovered that spikelet fiber is particularly well-suited as reinforcing fibers due to its high strength and resistance. Conversely, stalk fiber holds potential as a feedstock for nutrient recycling or bioenergy, owing to its high potassium content and less recalcitrance. Another study also been clearly stated that the spikelet fibers are can be used as reinforcing materials in the cement mortar and show better mechanical properties than stalk fibers [20]. Besides that, according to the research on the chemical and physical properties of the residual oil in OPEFB by Yunos *et* al. [22] indicates that the oil amount in the stalk is lower (2.04%) than in spikelet (7.39%). Moreover, pikelets exhibited high lignin contain which around 23.50%, but on the other hand, stalk have low lignin content around 11.50%. Meanwhile, stalk have high cellulose content (26.90–28.80%) than spikelet (20.60%) [22]. As a result, precaution action is needed in selection of correct parts of LGM to get high yield of cellulose content to turn into nanocellulose and not only that more research is needed to correctly measure the percentage of stalk and spikelet in an OPEFB.

Furthermore, solid waste such as PKS (which protects the kernel), MF and OPF also have emerged as viable candidates for nanocellulose isolation, owing to their high cellulose content. PKS can have several shapes, including uneven, spherical, angular, flaky, smooth, or irregular shell. PKS denotes as the shell fractions that persist after the crushing of the nut in the palm oil mill [23]. Moreover, it also refers to fibrous materials treated in bulk from production to end user. The mixture included dust, fiber, and clusters of tiny shells. Next, PKS absorbs water at a lower rate, ranging from 11–13% [23]. Furthermore, when oil palm tree reaches the end of its lifecycle, which usually lasts 25 to 30 years, OPF decomposes naturally in the field. However, it is noted that OPF exhibits low density and high porosity, rendering it unsuitable for structural applications [24]. The structure is made up of two primary components, namely the petiole (stem) and many long leaflets arranged on both sides of the stem. The upper two-thirds of the frond possess the most nutrients, and the lower third is abundant in cellulosic materials and sugars, crucial to producing biofuels and bio-based substances. Moreover, OPF consists of juice (liquid fraction) and bagasse (solid fraction). In this article a solid fraction was focused on converting it into nanocellulose [25]. Frond trimming of oil palm trees has a substantial impact on the yield of fruit bunches [26]. Trees that do not undergo frond trimming yield smaller and lighter bunches in comparison to trees that undergo major pruning below 32 fronds. Consequently, the regular industrial frond trimming procedures result in a widespread supply of OPF throughout the year.

STABILITY OF NANOCELLULOSE

Even though there are many articles on the isolation of nanocellulose (either CNC or CNF) from oil palm-based biomass, there are only a few that focus on the stability or dispersibility of oil palm based nanocellulose. Stability is also a very important property that need to consider during the isolation of nanocellulose, otherwise can give negative effect on the final product. Because the formation of aggregation hampers uniform dispersion and can result in sediment formation during certain applications such as in film production and many more in long term usage. As mentioned before, nanocellulose's strong intermolecular hydrogen bonding that occurs within and between nanofibers as shown in Figure 6 and due to high surface energy leads to a high tendency to form bundles or agglomerations [27, 28].

Nanocellulose, which is hydrophilic in nature, poses challenges in its dispersion in hydrophobic substances, including low polar and non-polar chemical solvents, and also in the majority of polymer matrices used for composite application [29]. The dispersion of nanocellulose in some highly polar solvents, like water, is facilitated by the strong contact between the surface hydroxyl groups and solvent molecules. To date, the successful dispersion of nanocellulose in different mediums remains a significant obstacle in the advancement of commercial cellulose nanoparticles. The effective enhancement of nanocellulose dispersibility may be achieved by appropriate surface modification that considers the properties of the medium. This modification aims to alter the surface hydrophilicity and enhance compatibility.

Besides that, colloidal dispersion and the electrical double layer are the important key features for controlling stability. The traditional DLVO theory well describes the stability of charged particles in electrolyte-rich aqueous solutions [30]. Based on the theory, the interaction energy between two colloidal particles is equal to the sum of the attractive van der Waals and repulsive electrostatic interactions as shown in Fig. 7 [31]. For instance, the amount of salt used gives little or no effect on the van der Waals interactions, which are mostly caused by variations in transient dipoles in the electron clouds of atoms and molecules. While repulsive electrostatic interactions are caused by the overlap of electrical double layers around charged particles, and it can be modified by the incorporation of salts. Hence, stable colloidal suspensions is achieved when there is low amounts of salt, whereas unstable suspension formed when the amount of salt

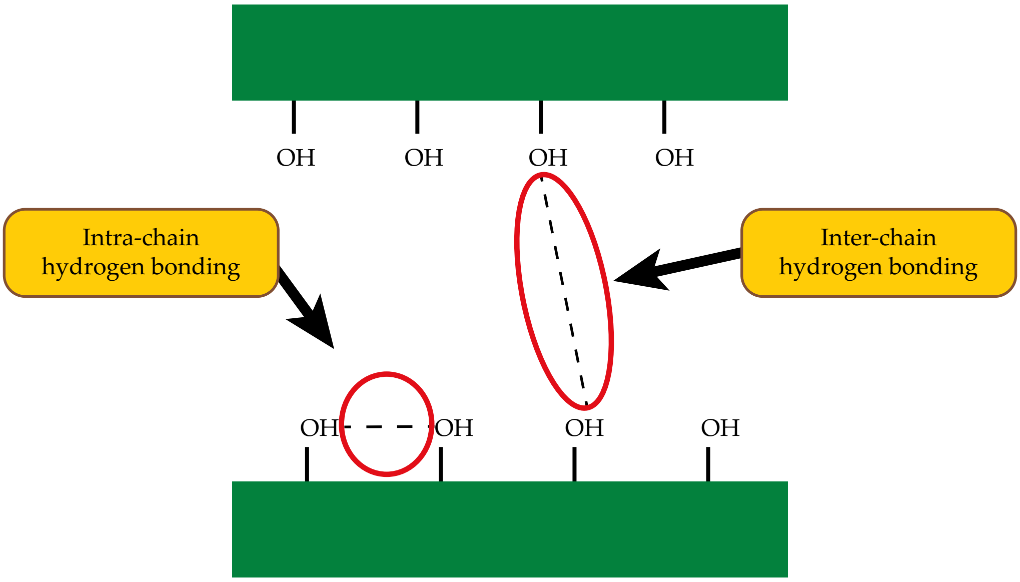


Fig. 6. Scheme of intra- and inter-chain hydrogen bonding in nanocellulose. Reprinted from [28]

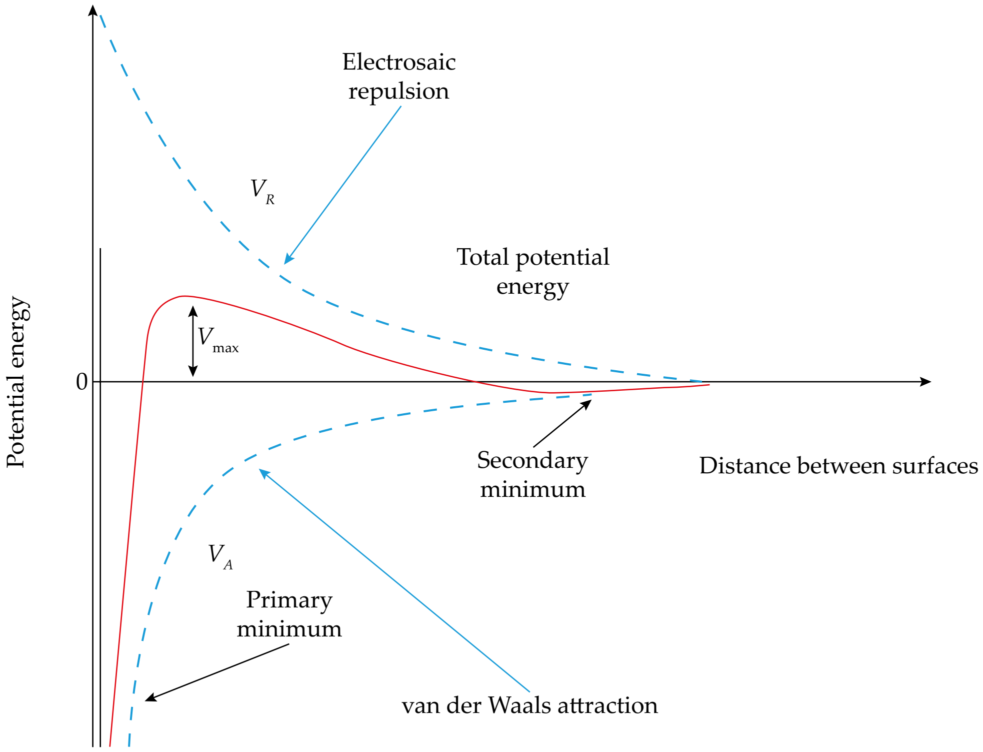


Fig. 7. The DLVO theory

rises [32]. The sudden transition between these two states occurs at a particular concentration referred to as the critical coagulation concentration (CCC) [32]. In this section, ways to overcome this issue are discussed.

Functionalization and surface modification of nanocellulose

The modification on the surface of CNCs depends on the availability of sufficient OH groups on their surface, which provide a desirable platform for covalent surface functionalization or chemical modification of CNCs. Currently, a number of alternative modification approaches have been proposed with the purpose of modifying the hydrophilic nature of CNCs is to improve the distribution of them in diverse solvents and polymer matrices [33]. By means of surface modification of CNCs, it is possible to optimize their hydrophilicity and hydrophobicity, as well as enhance their dispersibility and compatibility [34]. Novel functionalities can also be incorporated and could lead up to new route for sophisticated applications of CNCs. Chemical modifications like esterification, etherification, oxidation, sialylation, polymer grafting and many more have been explored in order to utilize the inherent benefit of having a large number of hydroxyl groups on the surface of nanocellulose [27]. Chemical functionalization have mostly been performed to immobilize stable negative or positive electrostatic charges on the surface of nanocellulose to enhance dispersion of nanocellulose produced by H_2SO_4 hydrolysis contain labile sulphate groups and to adjust the surface energy properties of nanocellulose to enhance compatibility, particularly when combined with nonpolar or hydrophobic matrices in nanocomposites [35].

Functionalization of nanocellulose is one of the ways to overcome the stability issue. For instance, Whba *et al.* [36] synthesized a stable nanocellulose by modifying its surface with polyethylene glycol (PEG) to enhance its biocompatibility and structural durability in biological applications. This technique is desirable due to its non-toxicity, low immunogenicity and stability which effectively minimize protein absorption and opsonization. This surface modification of nanocellulose with PEG results in the establishment of covalent bonds between the hydroxyl group on the nanocellulose surface and the functional group on the PEG molecules as shown in Figure 8. A successful surface modification of nanocellulose was achieved using the hydrolysis process. This was proved by the colloidal stability and negative surface charge that were observed in the nanocellulose -PEG/NaOH sample.

The zeta potential of this modifies nanocellulose is -25.6mV. A negative zeta potential is more favorable as it can counteract aggregation by causing charges to repel one another rather than cluster together. Furthermore, biocompatibility evaluations demonstrated that nanocellulose-PEG/NaOH is a safe and non-toxic material, proving its potential as an MR imaging contrast agent. This study's findings indicate that nanocellulose can serve as a coating agent to develop a novel dual-contrast composite material for diverse applications, particularly in the medical sector [36]. Moreover, they are many other

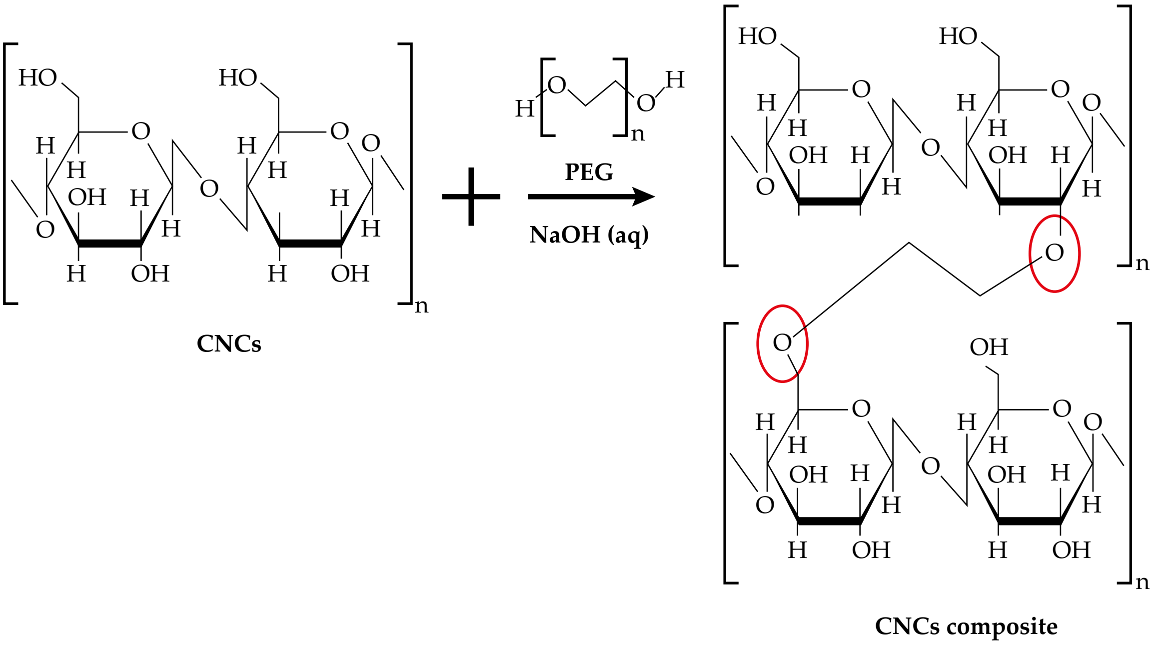


Fig. 8. Reaction mechanism between CNC and PEG and NaOH to produce surface-modified CNC. Reprinted from [36]

surface modified nanocellulose that have been used in the biomedical industry as review by Long *et al.* [37].

Besides that, Song *et al.* [38] creates multi-carboxyl group decorated on the nanocellulose surface by using HCl as catalyst and ascorbic acid and citric acid (CNC-*g*-AA-*g*-CA) as branching group which result in various sequential grafting of ascorbic acid or citric acid as shown in Figure 9. The resultant nanocellulose shows superior properties such as high thermal stability (T_{max}=359.3), high carboxylic group of 4.073 mmol/g, high zeta potential (47.7mV). These properties led to functionalizing nanocellulose can be utilized as absorbent, efficient flocculants, and good stabilizing Pickering emulsion. The excellent coagulation-flocculation capability to kaolin suspension with turbidity removal rate is 91.07% and 87.8% cationic dye (methylene blue) removal. Functionalized nanocellulose is a cost-effective and eco-friendly replacement to conventional flocculants derived from petroleum-based synthetic polymers to treat wastewater. This is because they are non-biodegradable and can potentially contaminate soil, leading

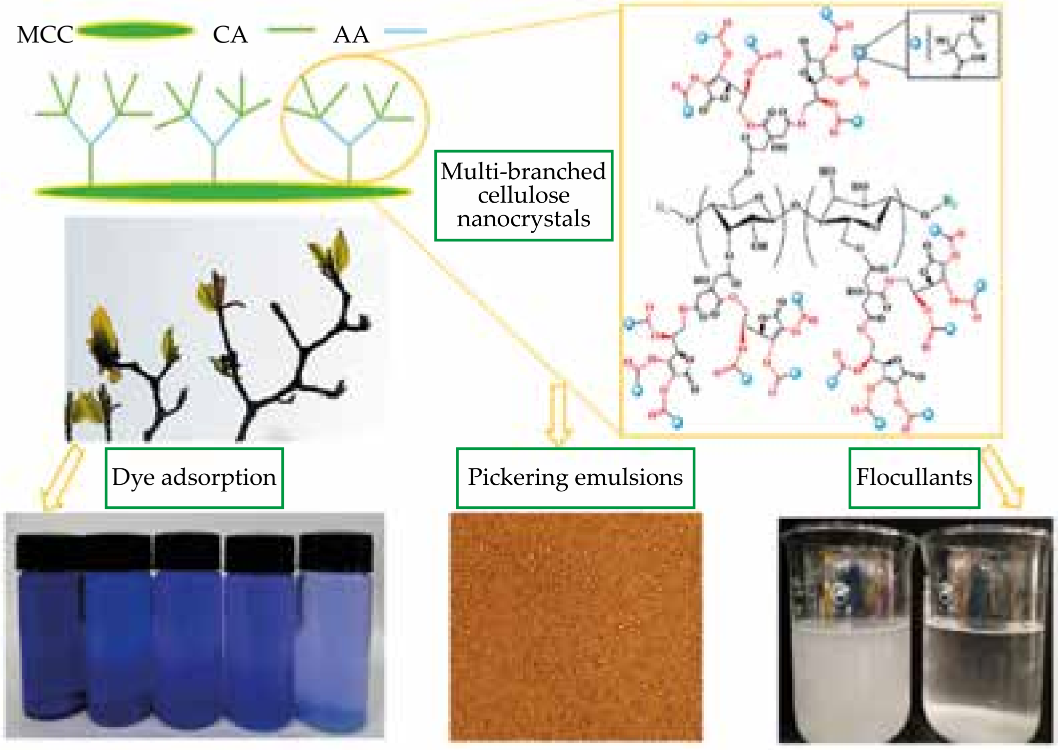


Fig. 9. Application of nanocellulose functionalized with ascorbic acid and citric acid. Reprinted from [38]

to secondary environmental degradation. Moreover, the more carboxyl group on the surface of the functionalized nanocellulose led to a slight increase in length compared to unmodified nanocellulose and good dispersion was absorbed based on morphology test which resulted from the stronger electrostatic repulsion between the nanocellulose.

Besides that, Abdelgawad *et al.* [39] introduced carboxymethyl group on the nanocellulose, which was used as a reducing agent or capping agent for silver nanoparticles (AgNPs) to produce cryogels for medical application, especially in wound care field. The purpose of the AgNPs loading is to acquire antimicrobial activity. Based on the TEM it has been clear that the surface modification led to the well distribution of spherical shape AgNPs without agglomeration attributable because of its strong reduction capability and the dual function of carboxymethyl nanocellulose as both a reducing and stabilizing agent.

Surfactant/creating steric barriers between nanocellulose particles

As mentioned before, nanocellulose produced via H_2SO_4 has high colloidal stability in aqueous environment, credited to the electrostatic repulsion that results from the anionic sulfonic group. However, it demonstrates poor thermal stability because of the sulfate group. As a result, in some cases HCl can be used to isolate nanocellulose. This way is easy, safe, lowers the use of acid, lowers consumption of water, and has good thermal stability compared to nanocellulose produced using H_2SO_4. However, it has lower stability (very easy to agglomerate in aqueous solutions), because it contains low negative charge group on nanocellulose surface. This statement also been proven worked done by Espinosa *et al.* [40], who isolate the nanocellulose using 3 different type of mineral acid: H_2SO_4, HCl, and H_3PO_4. The results indicate that even phosphoric acid has low charge density related to the small number of surface phosphate groups [43], it still stables in polar solvent such as DMSO, DMF and water like nanocellulose. Isolated from sulfuric acid, but nanocellulose produced from HCl shows poor stability due to low surface charge. However, among examined acids, nanocellulose isolate from the HCl shows the highest thermal stability followed by phosphoric and sulfuric acid nanocellulose. The author proposes, that the challenge of low stability in HCl-based nanocellulose may be addressed through the incorporation of ions, surfactants, as well as ultrasonication [41].

As a solution, Shang *et al.* [42] add hexadecyl trimethyl ammonium bromide (CTAB) after the HCl acid hydrolysis, which result in nanocellulose with well-dispersed CNC. This is because effective steric hindrance provided by the long alkyl chain of CTAB at low concentration, aids in minimizing CNC aggregation even after 7 days of storage which was proven based on TEM and particle size distribution of CNC as shown in Fig. 10. However, at high concentration of CTAB can caused the CNC aggregation due to "bridging" effect of CTAB. CTAB is a cost-effective and consist of tetra-substituted ammonium cationic surfactant with positively charged quaternary nitrogen and a long alkyl (C16) chain [42].

Same goes to Pawcenis *et al.* [41], who investigate the stabilizing effect of HCl hydrolyzed nanocellulose by the addition of the various type of surfactant (CTAB, sodium dodecyl sulfate (SDS), Pluronic F-127 (PF-127)) which are cationic, anionic and non-ionic surfactant respectively and also with the effect of pH value. Based on the result, it can be concluded that, the highest negative values (-44mV) were observed in the case of nanocellulose hydrolyzed for 180 min and stabilized with PF-127 without aggregation [41]. Furthermore, it was demonstrated that nanocellulose samples hydrolyzed for at least ten minutes, with a pH maintained between 5 and 7, exhibited excellent colloidal stability. The increase in length observed after introducing PF-127 to the nanocellulose solution after hydrolysis, may be attributed due to the aggregation or interaction between nanocellulose and either PEO (poly(ethylene oxide)) or PPO (poly(propylene oxide)) of PF-127. Significantly, the concentration of PF-127 employed in this work is lower than its CCC to prevent the micellization of PF-127, which might hinder the interactions between nanocellulose and PF-127. The interactions between nanocellulose and PF-127 are characterized by short-range hydrophilic and hydrophobic interactions, rather than electrostatic interactions [43]. Previous investigations have shown that PEO establishes hydrogen bonding due to the oxygen in the monomer and hydroxyl groups on cellulose. As a result, PPO can generate loops between PEO blocks that are adsorbed on the nanocellulose surface [41]. Hence, the observed elongation of carbon nanofibers (CNFs) that are stabilized with PF-127 may be attributed to the binding of multiple nanocellulose by PF-127 molecules.

Besides the addition of surfactants, there is also a study that adds salt which is Na^+ and Ca^{2+} ions to understand the CNC dispersion. The study indicates that the incorporation of salts and electrolytes may influence the quantity and distribution of charge on particle surfaces, as well as the hydrophobic/hydrophilic ratio, contingent upon the specific type of salt and surfactant used. Moreover, they also undergoes study relating the zeta potential to the engineering outcomes for nanocellulose, roughness analysis using the Atomic force microscopy (AFM) measurement [44]. Based on the AFM investigation of the surface roughness of films produced from CNC dispersions with varying zeta potentials using ionic salts reveals a negative correlation between zeta potential and film roughness [44]. This provides a straightforward approach to manipulate the visual characteristics of CNC films. In contrast, altering the zeta potential using a hydrophobic surfactant had no impact on surface roughness, suggesting the presence of distinct factors influencing film development. The findings indicate that the selection of ions and electrolytes function as an efficient method for

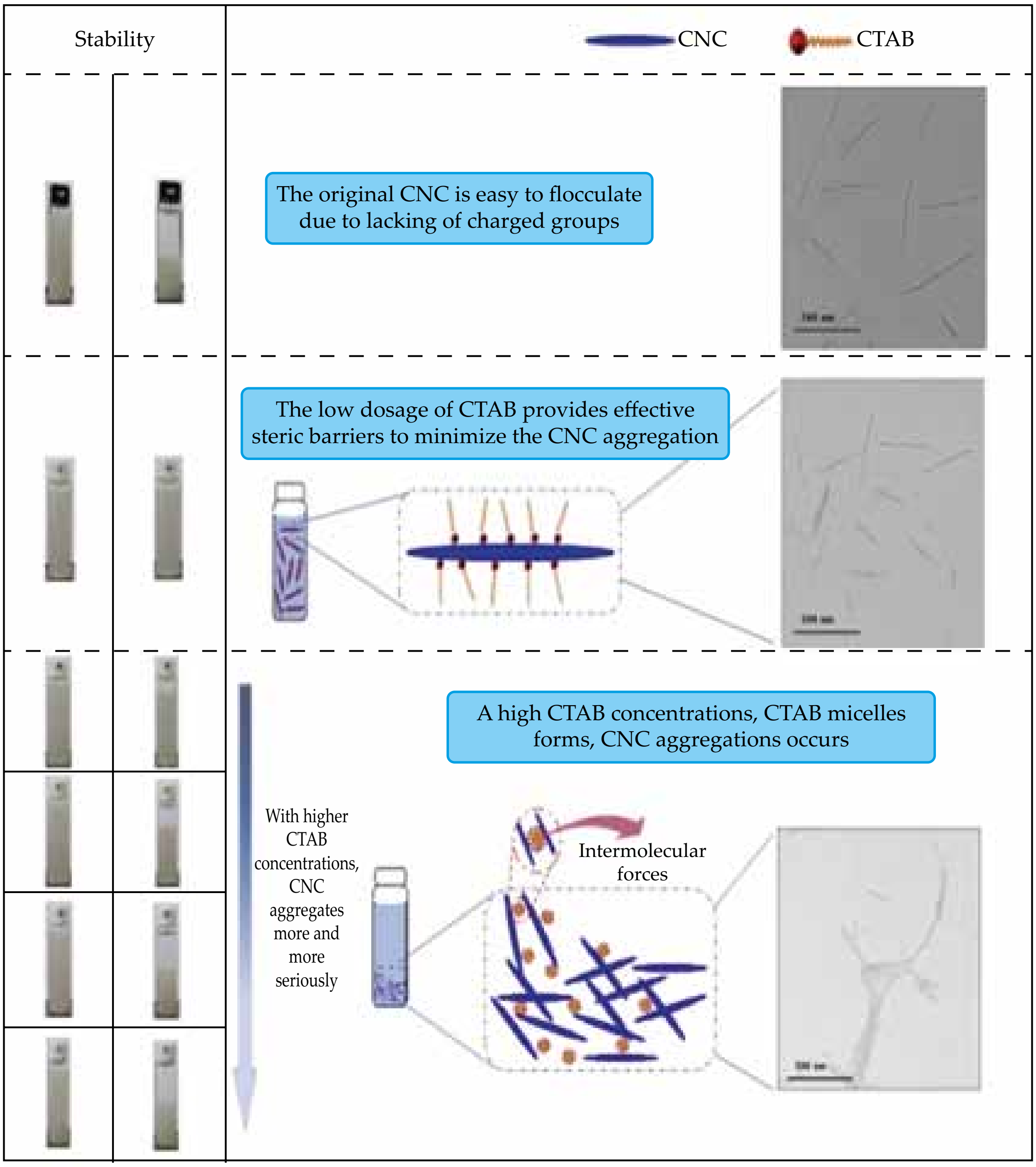
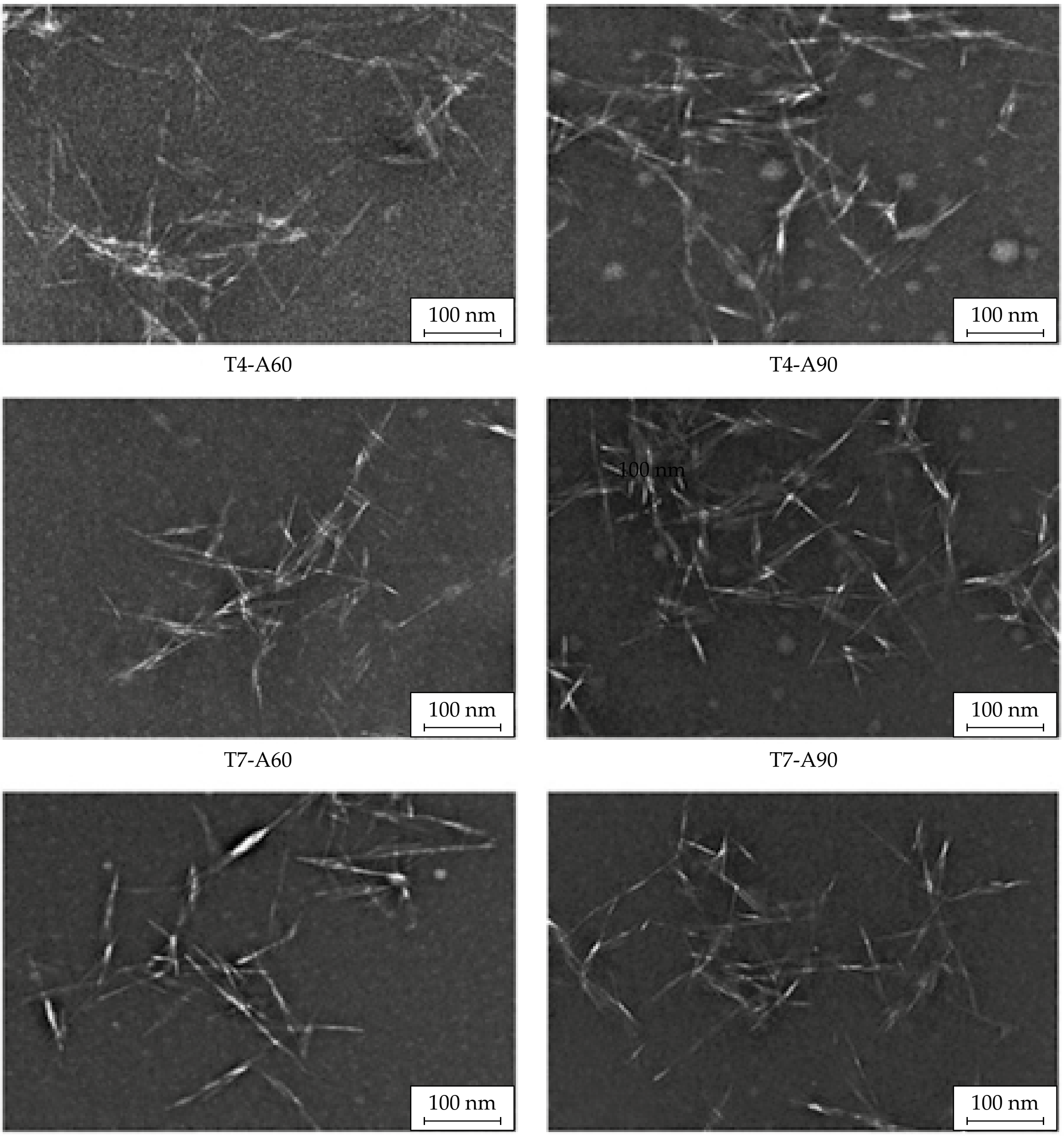


Fig. 10. The stability of CNC at different CTAB concentrations. Reprinted from [42]

regulating the CNC zeta potential, since it is closely correlated with the stability and quality of films derived from nanocrystal dispersions.

Furthermore, Cao *et al.* [32] have utilized monovalent salt (LiCl, NaCl, KCl, and CsCl) , divalent salt ($MgCl_2$, $CaCl_2$, $BaCl_2$) and trivalent inorganic salt ($LaCl_3$, $AlCl_3$, $FeCl_3$) which contain same anion (Cl^-) and different cations charge with the nanocellulose to monitor the colloidal stability in aqueous solution. As the concentration of monovalent salt increases, it leads to a reduction in negativity resulting from the compression of the double layer caused by charge screening. Electrophoretic mobility at a certain salt concentration decreases in the sequence $Cs^+ > K^+ > Na^+ > Li^+$. The extremely hydrated Li^+ cation exhibits the most substantial negative surface charge and CCC among the analyzed cations, attributable to its tendency to stay in the bulk solution with minimal adsorption on the CNC surface. Conversely, weakly hydrated Cs^+ cation adheres strongly to the particle surface, yielding the lowest CCC [32]. Additionally, as the concentration of the divalent salts increased, the electrophoretic mobilities rapidly become less negative. Furthermore, particularly within the elevated salt concentration range, the electrophoretic mobility at a specific salt concentration decreased slightly or became less negative in the order $Ba^{2+} > Ca^{2+} > Mg^{2+}$ [32]. Next, electrophoretic mobility of CNCs decreased in negativity as the concentration of $LaCl_3$ increased, which can be related to the effective charge screening by the trivalent La^{3+} counterions. The significant adsorption of aluminum and ferric hydrolysis species resulted in charge neutralization and charge reversal [32].



T4-A60 T4-A90

T7-A60 T7-A90

T10-A60 T10-A90

Fig. 11. TEM images of PVA/CNCs prepared at different times (T part) and amplitudes (A part) of sonification. Reprinted form [46]

SONICATION TIME AND POWER

Among several mechanical pre-processing methods, ultrasonication is commonly used as a effective approach to improve the dispersion of nanocellulose in polymer blends [45]. The ultrasonication technique involves applying sonic waves to a nanocellulose suspension to disintegrate and disperse the nanocellulose bundles. The sonication process involves the application of ultrasonic waves to specimens at frequencies over 20 kHz, corresponding to 20,000 cycles per second. As the frequency rises, the intensity of the agitation also increases [46]. In the context of nanocellulose, sonication is a technique recommended by the Joint Research Centre (JRC) Guidance to mitigate agglomeration/aggregation. Research has demonstrated that sonication is effective in dispersing individual fibers prior to microscope imaging and biological exposure. The sonication process is defined by the amount of energy delivered to the suspension, which is dependent on the sonication power, amplitude, duration, and probe surface area [46]. Variability in results is more likely to occur when ultrasonic treatment is applied inconsistently. Although sonication is commonly employed to improve the dispersion of nanofillers in different solvents, there is a scarcity of research examining how sonication parameters, such as power and duration, affect the dispersion of nanocellulose in an aqueous medium. Nanocellulose can disperse in water, however the stability of the dispersion is somewhat limited by the hydrogen bonding between hydroxyl groups

[27]. Several physical methods including thinning, ultrasonic dispersion, and high-pressure homogenization have been employed to reduce the strength of hydrogen bonding, thereby promoting the dispersion of nanocellulose. Hamid *et al.* [47] have reported that the low sonication power only able to broken the weak bonds of amorphous cellulose portion, while the cellulose interlayers are partially disentangled and partially delayers [47]. However, at elevated sonication power, the strong glycosidic bonds within the crystalline region of cellulose can be disrupted, along with the breaking of the interconnected hydrogen bond network. This leads to the structural opening, commonly referred to as defibrillation. Moreover, ultrasonication also help in size distribution of colloidal nanocellulose [47].

Besides that, Hastati *et al.* [48] obtain a self-aggregations and irregular shapes TEM image of nanocellulose. They explain that this phenomenon occurs due to the strong hydrogen bonding between the nanocellulose and due to the van der Waals forces attraction. In addition to this reason, this phenomenon also can be occurred due to the low intensity of ultrasonication can cause self-aggregation in water even after it has been sonicated. It is most likely owing to insufficient sonication force to efficiently distribute the nanocelluloses in distilled water [48]. Thus, optimization of sonication power and time is very important to get high stable and well dispersed nanocellulose without any formation of aggregation. Furthermore, Shojaeiarani *et al.* [46] also agreed that the sonication can help in the dispersion of the nanocellulose and they also agreed that the sonication time and amplitudes can affect the nanoparticles length or size and dispersion. The primary cause of nano scale nanocellulose is the breakage of fibers caused by particle agitation. Figure 11 demonstrates that the presence of single CNC particles in the PVA-CNCs suspension indicates enhanced dispersion of CNCs in T7-A90 [46]. Specifically, the longer duration of sonication in T10-A60 and T10-A90 corresponds to a higher ultrasound energy input, leading to a better homogeneous dispersion of CNC in the polymer matrix [46]. Yet, prolonged, and harsh ultrasonic treatment may deteriorate the structure of nanofibers, therefore affecting their crystallinity and mechanical characteristics. The optimization of the ultrasonication treatment process is crucial for achieving consistent distribution of CNCs and regulating their formation.

DRYING PROCESS

Dispersibility of nanocellulose in water is significantly influenced by the employed drying technique. The redispersion of the nanocellulose obtained through water evaporation is exceedingly challenging, whereas the freeze-dried nanocellulose can be successfully redispersed in water. Drying of cellulose materials is known to cause irreversible reorganization of hydrogen bonding in cellulose, thus leading to the difficulties with dispersion of dried cellulosic material [41]. This rearrangement arises from the elimination of water during drying, which reduces the space between individual nanocellulose particles [41]. The agglomeration process at high temperature drying is defined as hornification, characterized by creating an irreversible (or reversible) bond between the hydroxyl groups in nanocellulose structure. There are some types of drying that have been utilized for the nanocellulose which is solvent evaporation, lyophilization or freeze drying. Supercritical extraction and also spray drying which has its own advantages and disadvantages [15]. Freeze drying is the most used method among researchers, as it is convenient and cheaper than supercritical extraction. Freeze drying involve ice sublimation, where pressure used is significantly below the triple point of water [49]. By eliminating condensation, it is possible to reduce the self-aggregation of nanocellulose that can formed during the freezing process. A solid-phase condensation (desublimation) of the solvent vapors occurs when sublimation is conducted in a high enough vacuum (residual pressure is approximately (515 Pa) and at temperature between -20°C and 50°C [50].

STABILITY EVALUATION

Understanding the stability of oil palm biomass based-nanocellulose, whether through chemical or mechanical treatment, is a significant area of interest that has caused extensive research among researchers. The stability of the nanocellulose can be evaluated using methods based on the examining of the morphology and topography of nanocellulose using transmission electron microscopy (TEM) and field emission scanning electron microscopy (FESEM). Furthermore, zeta potential techniques are utilized to determine the zeta potential of nanocellulose, which directly give information about the stability of the nanocellulose. Not only that, birefringence observations also can be done to evaluate the stability of the nanocellulose. These analytical methods are fundamental for characterizing the stability of nanocellulose and each characterization technique offers unique insights into the properties of the analyzed sample. Therefore, it is imperative to thoroughly evaluate the isolated nanocellulose to comprehend its properties before its application in various fields. This section will delve into the typical characterization techniques employed for nanocellulose isolation derived from various oil palm biomass sources.

Morphological analysis for stability of oil palm-based CNC

Morphology stands out as one of the most crucial parameters for understanding particle distribution, nanocellulose diameter, and detecting any occurrence of agglomeration. Moreover, morphology analysis also can be used to determine the type of nanocellulose synthesized, whether CNCs or CNFs. Morphology is changed

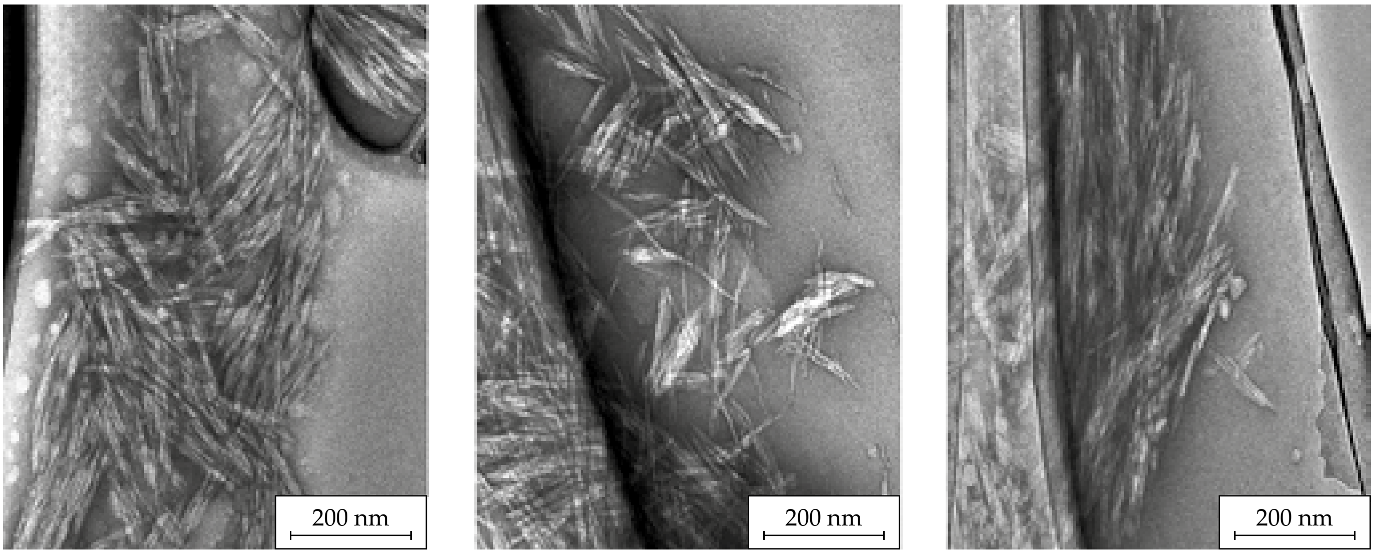


Fig. 12. TEM images of CNC with little aggregation. Reprinted from [29]

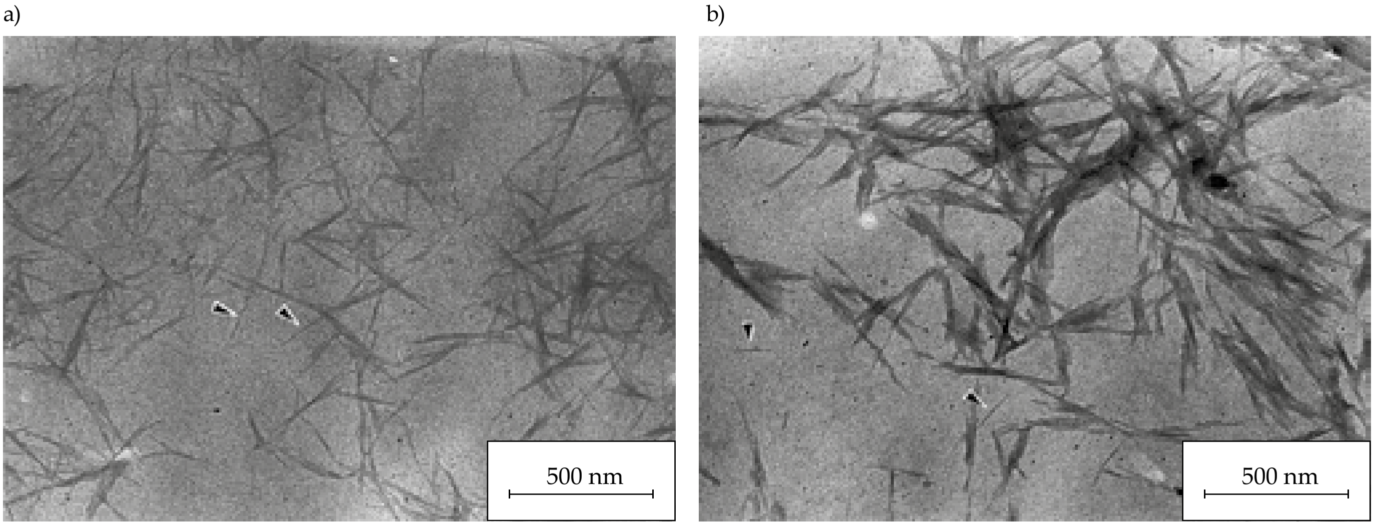


Fig. 13. Dispersibility of CNC isolate using a) H_2SO_4, b) HCl. Reprinted from [57]

by various aspects including source material, extraction methods, reaction time, and isolation method. Various microscopic techniques, such as FESEM, TEM, or AFM (atomic force microscopy), are employed to investigate nanocellulose morphology. During analysis, the sample is coated with a thin coating of platinum or gold to mitigate the charging effect. Braun *et al.* [29] has isolated the functionalized CNC using single step method (HCl hydrolysis process simultaneously with the esterification) and used TEM to examine morphology and understand the dispersibility of the CNC. Figure 12 indicates the presence of a clear needle-like or crystal-like structure of nanocellulose, with minimal aggregation observed in all samples. This phenomenon occurs because the presence of an ester group on the surface during hydrolysis decreases the hydrogen bonding and promotes the individualization of CNC [29]. Consequently, the integrated one-step approach offers both surface functionalization and reduced embedded energy demands.

Besides that, isolation of nanocellulose using diverse types of acid can also influence the stability or dispersibility of nanocellulose in water. For example, CNC prepared by hydrochloric acid will have lower stability compared to CNC prepared by acid hydrolysis. This is because they have lower charge on functional group. The presence of strong acid groups in the H_2SO_4 treated CNC is due to the esterification of hydroxyl groups by sulfate ions, which are not found in the HCl-treated sample [51, 52]. That presence of sulfate groups (SO_4^{2-}) resulted in a negative surface charge on CNC, which facilitates its dispersion in water. Figure 13 shows the drastic changes on the dispersibility of the CNC isolate using H_2SO_4 versus HCl.

Septevani *et al.* [53] used H_2SO_4 and phosphoric acid to isolate the nanocellulose from OPEFB (oil palm empty fruit bunch). In the surface morphology examination, CNC made by phosphoric acid has a harder surface area and more aggregates than CNC isolate by H_2SO_4 as shown in Figure 14. The agglomerated fiber of the CNC made by

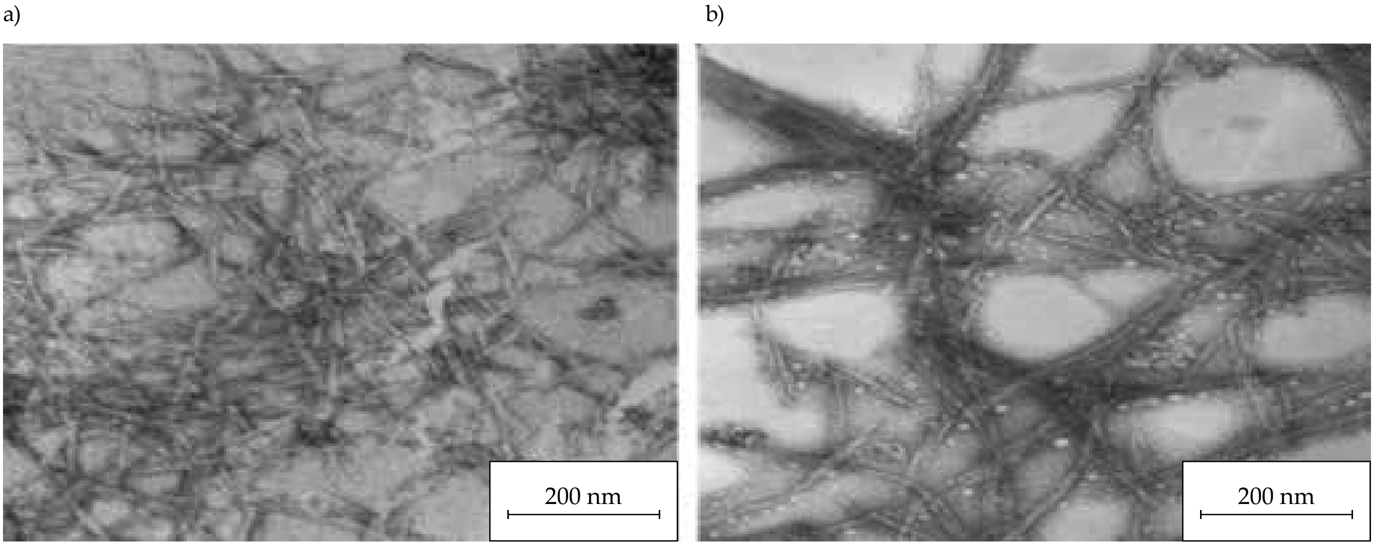


Fig 14. Morphology of CNC isolates produced using different acids: a) H_2SO_4, b) H_3PO_4. Reprinted form [53]

phosphoric acid structure was very clear because each long, twisted fiber was made by mild acid hydrolysis, and an agglomeration of microns was seen after the drying process [53].

The nanocellulose isolation using H_2SO_4 shows the better stability and good colloidal stability of nanocellulose suspension than other acids. However, in the reaction time parameter, H_2SO_4 concentration and temperature must be considered in producing stable nanocellulose [50]. Fahma *et al.* [55] have synthesized CNF from OPEFB using the acid hydrolysis at different reaction time ranging from 15 to 90 min. In the results, scientists mentioned the importance of optimizing the reaction time during the nanocellulose isolation on the dispersibility. Moreover, they also relate the reaction time of acid hydrolysis with the sulfate content. As the reaction time increased from 15 to 90 min, the sulfate content increased from 0.24 to 48.85 mmol/kg. Figure 15 shows

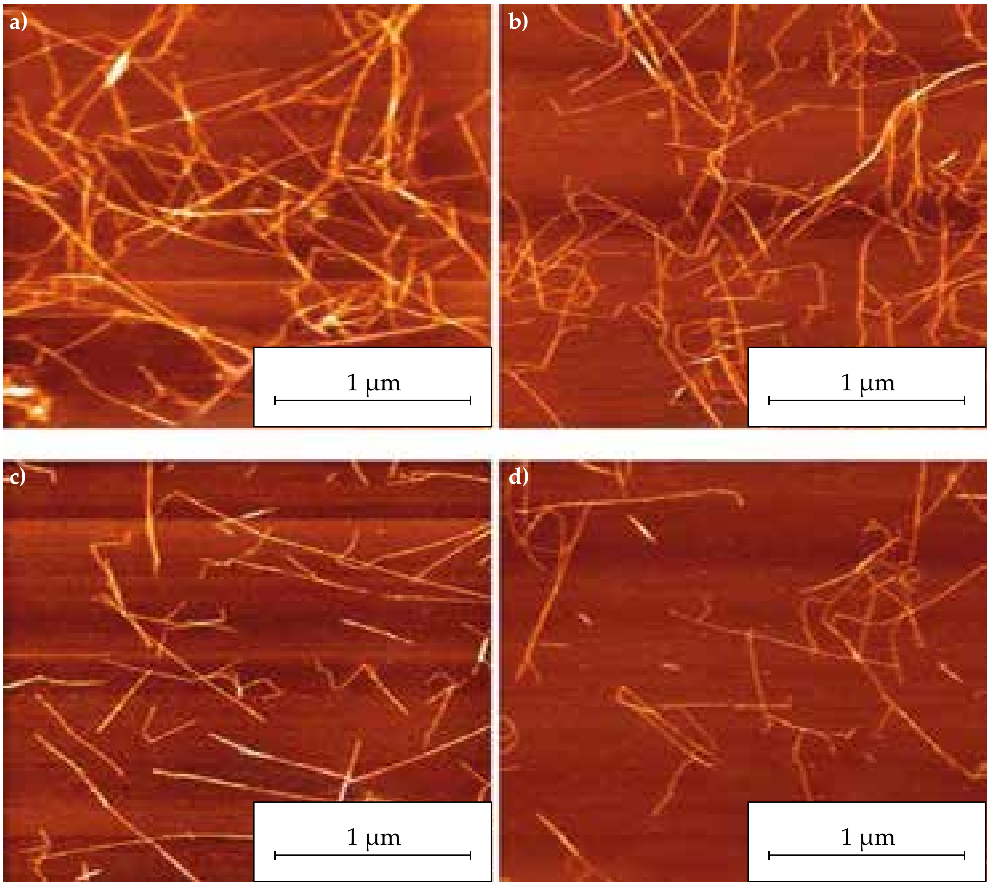


Fig. 15. Dispersibility of CNF at different acid hydrolysis reaction time: a) 15 min, b) 30 min, c) 60 min, d) 90 min. Reprinted from [55]

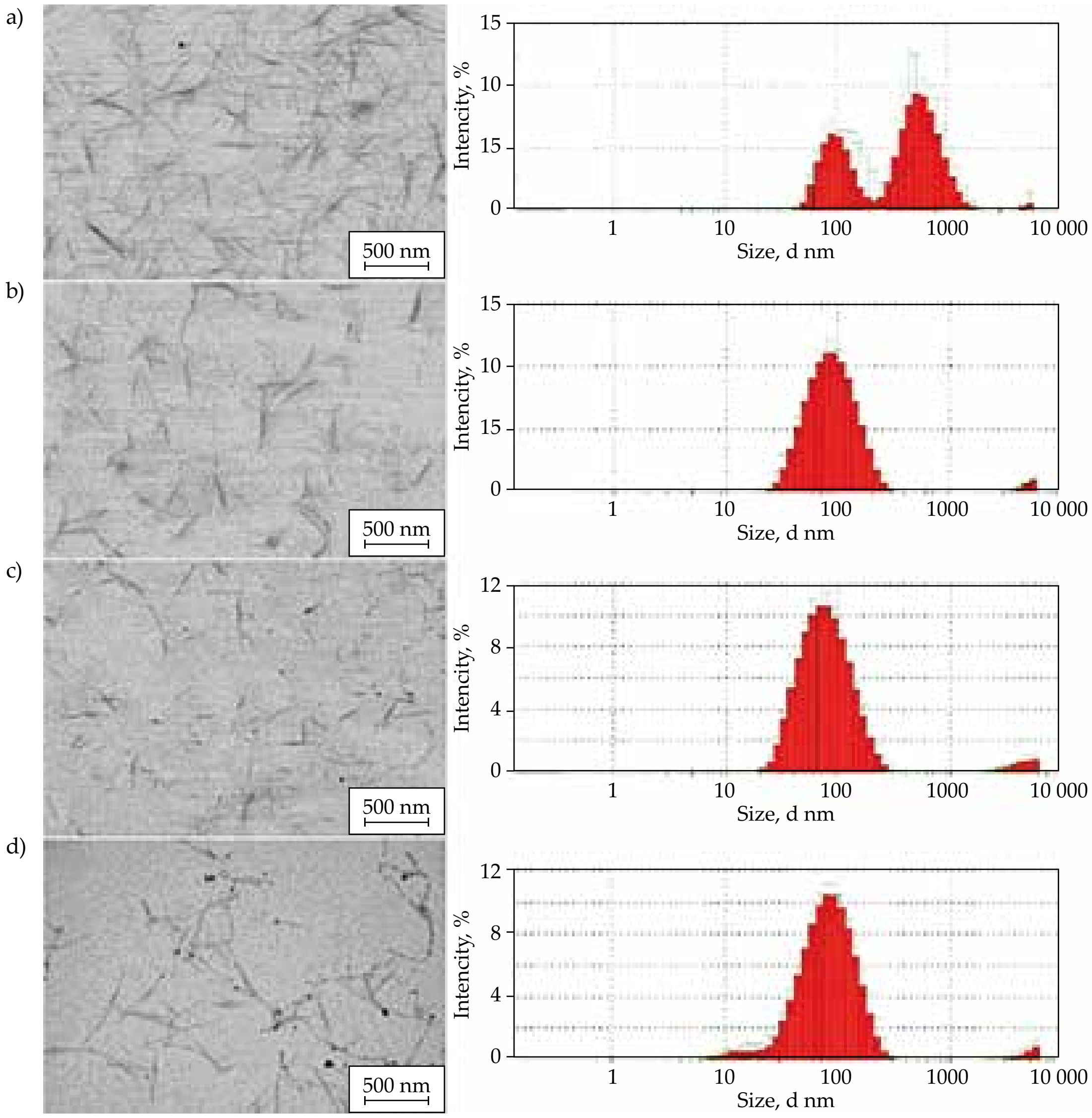


Fig. 16. SEM images and particle distribution graphs of CNCs produced from: a) 56 wt% of H_2SO_4, A/P 15, 30 min, b) 58 wt% of H_2SO_4, A/P 15, 45 min, c) 60 wt% of H_2SO_4, A/P 15, 30 min, d) 57.88 wt% of H_2SO_4, A/P 20, 60 min. Reprinted from [56]

the CNF prepared by H_2SO_4 hydrolysis at different reaction time ranging from 15–90 min. CNF isolation at 15 and 30 minutes demonstrated several bundles of aggregate, but when prolonging the hydrolysis time to 60 min and 90 min, the total number of fibril aggregate bundles reduces. This is because, the hydrolysis of sulfuric acid in the context of CNF typically targets the amorphous regions of microfibrils, leading to a transverse cleavage, that reduces fiber diameter from microns to nanometers which enhances the dispersibility of CNF [55].

Foo *et al.* [56] have combined the SEM image and aspect ratio distribution histograms to provides valuable information about the distribution of the nanocellulose. For instance, Figure 16 illustrates that hydrolysis yield of nanocellulose will be low, either due to the increased in acid concentration or the reaction duration. Fig.16b demonstrates a narrow particle size distribution compared to Figure 16a, where particles tend to agglomerate.

Zeta potential for stability of oil palm-based CNC

Nanocellulose dispersity depends on surface charge density. Zeta potential serves as a crucial parameter indicating the tendency for stability or aggregation in a dispersion [57]. A nanocellulose suspension is deemed stable when the zeta potential value exceeds ±30 mV or ±25 mV, as nanoparticles possessing sufficient charge repel each other, thereby ensuring better stability and preventing aggregation. Conversely, suspensions with extremely low zeta potential are prone to instability, leading to flocculation and aggregation. The utilization of H_2SO_4 during hydrolysis introduces negatively charged sulfate groups onto the nanocrystal surface, forming a negative electrostatic layer that envelops the nanocellulose. This layer contributes to stabilizing colloidal suspension through strong electrostatic repulsion. The adsorption of hydrophobic anions leads to a tendency for more negative zeta potentials, while the adsorption of hydrophobic cations

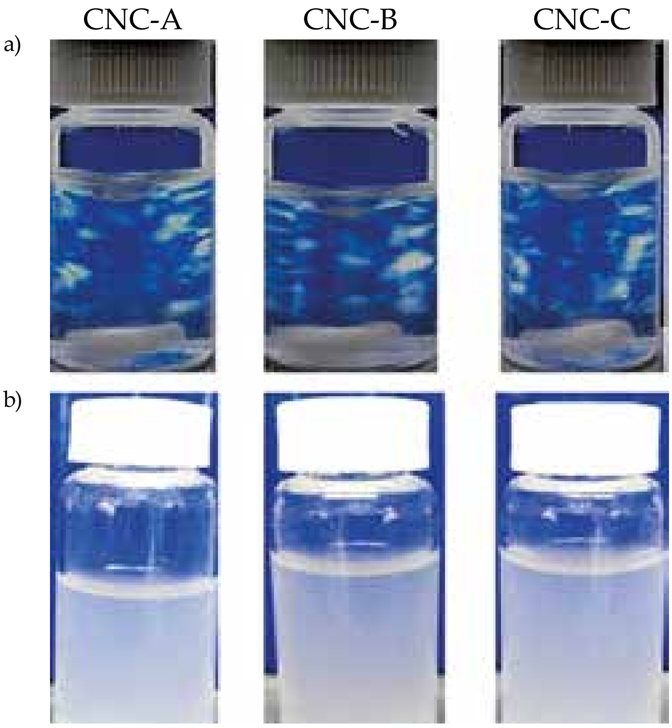


Fig. 17. Birefringence observations of CNC: a) before 6 months, b) after 6 months. Reprinted from [58]

results in more positive zeta potential values [51]. Anionic groups, including sulfate, carboxyl, and phosphate groups, are often added to the surface of nanocellulose to enhance surface charge repulsion. During the isolation of nanocellulose through sulfuric acid hydrolysis, sulfate groups are incorporated into the C6 position of cellulose through esterification, leading to a marked enhancement in the negative charge density on the surface [51] which can create stable aqueous suspensions primarily composed of single fibrils with minimal aggregation.

Birefringence observations

Birefringence observation is used to evaluate the nanoscale dispersibility of CNC in water as shown in Figure 17. When CNC is nano-dispersed in specific solvents, shear birefringence between crossed polarizers is observed under stirring. CNCs isolated by hydrochloric acid hydrolysis will have poor dispersibility, due to lack of charged functions such as sulphate ester to induce electrostatic repulsion, which fail to display birefringence. Nonetheless, the CNC suspensions derived from OPEFB in the present investigation by hydrochloric acid hydrolysis, succeeded by ultrasonication exhibited clear birefringence, confirming the achievement of nano-scale ordered dispersions [58]. Furthermore, these suspensions remained stable with no sedimentation even after standing for 6 months [58].

CHALLENGES AND OPPORTUNITIES

The demand for palm oil is on the rise, accompanied by a proportional increase in solid and liquid waste in the palm oil industry, posing significant challenges. Improper disposal of solid waste from the oil palm industry can pose substantial environmental risks. Consequently, researchers are intensifying efforts to utilize oil palm biomass for various applications, particularly in producing nanocellulose, whether in the form of CNCs or CNFs. Valuing oil palm biomass waste represents a novel approach with vast potential for ecological and economic benefits. Utilizing nanotechnology to produce oil palm based nanocellulose offers numerous advantages over conventional disposal methods like burning and composting. However, the chemical composition varies depending on the type of oil palm biomass, necessitating different pretreatment procedures for nanocellulose isolation. The challenge lies in developing universal pretreatment theories, despite numerous investigations into different techniques. The economic feasibility of the oil palm biomass type and subsequent procedures post-pretreatment play crucial roles in selecting an appropriate technique.

Therefore, research should explore multiple pretreatment methods and combinations to achieve greater yields and high stability. Additionally, testing the same pretreatment method on different oil palm biomass types is essential due to chemical composition variations. Factors like H_2SO_4 concentration, reaction time, and temperature significantly influence end properties, requiring precise control for high-yield, homogeneous, and highly crystalline nanocellulose synthesis. Furthermore, the hydrophilic characteristic of nanocellulose poses limitations in certain applications, prompting researchers to modify its surface. Despite five types of solid waste produced from the oil palm industry (OPEFB, OPT, OPF, MF, and PKS), studies focus on utilizing OPEFB, with limited data on other residues. Hence, future research should explore utilizing various oil palm biomass types for valuable products in diverse applications including biomedicine, biometrics, automotive, and structural fields.

Life cycle assessments should encompass detailed analyses from raw material extraction to final disposal to ensure nanocellulose's environmental and human health safety, an aspect lacking in many studies. Additionally, integrating atomistic and analytical modeling with innovative 3D simulation software supported by machine learning and Industrial Revolution 4.0 technologies can enhance understanding of the structure-property relationship in nanocellulose-based materials, guiding their design and synthesis. In the future, achieving completely automated production processes of advanced nanocellulose-containing materials using artificial intelligence, the Internet of Things, and big data analytics holds promise.

CONCLUSIONS

It is evident that solid waste from the oil palm industry, including OPEFB, OPT, OPF, MF, and PKS, can be transformed into valuable products like nanocellulose like CNCs or CNFs, utilizing different pretreatment methods tailored to specific application requirements and suitability. The growing interest in nanocellulose production is driven by its unique characteristics, including non-toxicity, renewability, biodegradability, lightweight nature, superior mechanical properties, and abundance of OH groups for potential surface modification. However, nanocellulose does present some limitations, such as high surface energy and hydrophilicity, leading to particle agglomeration and aggregation, thereby posing challenges in direct applications [59]. The most employed method for nanocellulose isolation involves is by using sulfuric acid which can result in the good dispersion of nanocellulose in water due to the sulfate group, but it has low thermal stability. But nanocellulose isolate by the HCl has low stability but have high thermal stability. Both situations can be overcome with surface modification, utilization of surfactant, optimization of sonication time and power and the drying method also can increase the dispersibility of the nanocellulose.

Various functionalization and modification techniques have been used, such as amidation, esterification, etherification, silylation, carbamination, polymer grafting, and others. These modifications change hydrophilic nature into hydrophobic nature, providing optimal dispersion of nanocellulose in solvents, composites, blends, films, gels, and other media. Therefore, focusing on the stability of nanocellulose is very important to widen the application, such as in film production or in medicine. Moreover, there are many ways to evaluate the stability of nanocellulose, which are based on the morphology study, zeta potential, or birefringence observations. Therefore, more research is needed to explore the potential applications of oil palm biomass not only OPEFB but also OPT, OPF, PKS, and MF in various fields, in line with the zero-waste concept and aiming to reduce the accumulation of oil palm waste.

ACKNOWLEDGEMENT

Special thanks to the Ministry of Higher Education (MOHE) for funding under the Fundamental Research Grant Scheme (FRGS) (FRGS/1/2023/STG05/USM/02/3). The authors would like express gratitude for the financial support received from the Universiti Pertahanan Nasional Malaysia.

Authors contribution

M.M. – investigation, validation, visualization, writing; M.N.N. – research concept, writing, methodology, investigation, validation, visualization; M.H.M.K. – visualization; M.R.A. – visualization; N.A. – validation, visualization; M.N.F.N. – research concept, writing, methodology, investigation, validation, visualization.

Funding

This research was funded by Fundamental Research Grant Scheme (FRGS) (FRGS/1/2023/STG05/USM/02/3).

Conflict of interest

The authors declare no conflict of interest.

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Received 9 XI 2024.
Accepted 5 I 2025.

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