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Nanocellulose from Agricultural waste–A Concise Insight Into Extraction and Applications

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ABSTRACT

Uncontrolled dumping of agricultural wastes is a cause of concern for the well-being of the environment. Nanotechnology has gained research popularity due to its beneficial applications. Nowadays the separation of nanocellulose from different agricultural wastes is explored in an increasing measure. Agricultural wastes are subjected to various treatments to extract nanocellulose. Nanocellulose extracted from agricultural wastes can be used in the preparation of biodegradable nanocellulose-based materials (for various applications) which are environment-friendly and can promote sustainable economic growth by ensuring the good usage of agricultural wastes.

Keywords: Nanocellulose, Agricultural waste, Nanotechnology, Acid hydrolysis, Biodegradable.

INTRODUCTION

Agricultural production is forecasted to increase further to provide food to the burgeoning world's population as well as to meet the industrial needs and animal food requirements[1]. The manufacture of biofuels had a substantial impact on rising crop consumption between the years 2000 and 2015[2]. The negative social and environmental effects of extracting fossil fuels have stimulated the search for greener alternatives[3]. The recycling of several biomass wastes intended for high-value-added bioproducts Fig. 1 has gained substantial attention recently for minimizing the environmental effect due to the use of fossil-dependent polymers[4].

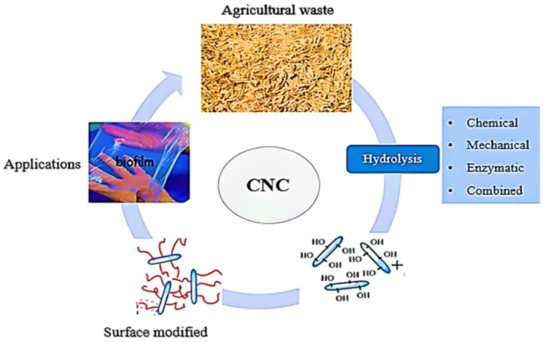

Fig. 1. Environmental benefits of using cellulose nanocrystals (adopted from Chaka, 2022)[4]

The problems associated with the dumping of plastic and agricultural waste must be addressed to protect the global ecosystem[5]. Biomass or agricultural wastes have a significant potential to generate sustainable energy from renewable fuels[6]. Starch from cereals, mostly wheat, and maize, is used as the primary raw material to create ethanol[7]. Agricultural wastes and natural plant fibers find application in composites because of their affordability, sustainability, and environmental friendliness[5]. Fruit peels and husks are potential cellulose reservoirs[8]. Cellulose is the most abundant organic substance and is almost an inexhaustible source of raw material. The most prevalent natural biopolymer is cellulose, which can be found in agricultural wastes (straw, corncobs, corn stover, sugarcane bagasse, wheat and rice husks, coconut husks, maize straw, palm oil waste, and skins of various fruits), tree trunks and dead forest matter (both hard and soft), energy crops, food waste, municipal and industrial bio-waste such as used paper, carton, and wood from the construction sites[9,10,11]. The potential for making cellulose nanocrystals by using different extraction methods from agricultural wastes is very high among biomass wastes[4]. Several types of agricultural wastes Fig. 2 have been used for the extraction of cellulose nanocrystals[4].

(a) coconut husk, (b) cocoa pod husk, (c) corn cob, (d) ramie fiber, (e) enset fiber, (f) kenaf fiber, (g) rice husk, (h) wheat straw, (i) jute fibers, (j) pineapple waste, (k) date pits, and (l) pea hull

Fig. 2. Common agricultural wastes useful for the extraction of cellulose nanocrystals (adopted from Chaka, 2022)[4]

Biofuels and biopolymers are two examples of value-added products that may be made from lignocellulosic biomass[12]. Researchers are actively focusing on cellulose nanofibers, also known as microfibrillated cellulose, cellulose microfibril, and microfibrillar cellulose, because of their encouraging nanocomposite applications[13]. Research trends are focused on finding practical applications for lignocellulose, which is present in huge amounts in agricultural wastes. Biodegradability, low density, and superior mechanical qualities make lignocellulosic materials quite interesting. Research into nanocellulose extraction from lignocellulosic biomass, notably from agricultural leftovers, is extremely appealing because of nanocellulose's remarkable qualities and potential future uses[14]. Recycling agricultural waste helps to keep the planet clean and the value of agricultural waste is increased due to the extraction of nanocellulose[15]. Considering the increased research focus on nanocellulose, an attempt has been made to highlight the nanocellulose extraction from agricultural wastes and subsequent applications in a concise manner with the help of published literature.

Nanocellulose from Agricultural Wastes: Extraction and Applications

Nanocellulose manufacturing began in 2011 in a small pilot facility and gradually nanocellulose is being manufactured on an industrial scale in a variety of countries throughout the globe using innovative techniques[16]. Extraction of cellulose from pre-treatments often forms either nanofibers or nanocrystals, depending on the usage of specific treatment and the majority of the literature discusses methods for extracting and purifying cellulose using acid treatments[3]. Cellulose, lignin, hemicellulose, and other non-cellulosic components are included in lignocellulosic biomass and to ensure that only cellulosic materials are used in the subsequent nanocellulose extraction process, the pre-treatment of biomass is essential[15]. Acid hydrolysis is often performed to get cellulose nanocrystals after the alkaline treatment and purification, whereas cellulose nanofibers are also obtained by mechanical treatments terminating in a high-pressure homogenization procedure[3]. Nanocellulose is often isolated from cellulosic materials using acid hydrolysis and it has been observed that some of the cellulose chains are more organized than others, and acids quickly break down the disorganized regions while leaving the more orderly ones intact[17]. Nanocellulose is extracted from lignocellulosic biomass using the two-stage process in which the pre-treatment eliminates all of the chemicals that are not cellulosic, such as lignin, hemicellulose, and others and subsequently in the next step, cellulose fibrils are processed for removal of nanocellulose[14]. Nanocellulose extraction is a multi-step, costly procedure due to the expensive chemicals, the high expenses related to building and maintenance of the equipment used in acidic conditions, and the difficulties of acid wastewater treatment for operations like acid hydrolysis[18]. Microcrystalline cellulose is a primary by-product of acid hydrolysis and amorphous cellulose is decomposed during acid hydrolysis[19]. Hydrolysis is significantly impacted by factors such as reaction temperature, reaction duration, acid type, acid concentration, and fiber-acid ratio, while different particle sizes, crystallinity, morphology, thermal stability, mechanical characteristics, yields, and degrees of polymerization may be achieved from microcrystalline cellulose depending on various factors[19]. During acid hydrolysis, cellulose, and hemicellulose polymers are broken down into their constituent monomers and the liberated cellulose may then be used in a succession of processes, such as glucose degradation or in some other way[20]. The crystalline domains of the cellulose chains are released from the amorphous areas via an acid hydrolysis process, which is part of the extraction procedure[20]. Formic, sulphurous, phosphoric, nitric, hydrochloric, and sulfuric acids may all be used in hydrolysis however, only hydrochloric and sulphuric acids have been utilized in industrial processes because of their least cost and are less toxic in nature[20]. Though a variety of acids may be used for acid hydrolysis, sulphuric acid (H_2SO_4) is the most commonly used[21]. Sulphuric acid is often used in acid hydrolysis, the sulphate ions esterify the hydroxyl group, allowing the nanocrystalline cellulose to be successfully partitioned and disseminated as a stable colloid system[17]. The separation of cellulose chains and finally the dissolution of cellulose Fig. 3 by the breaking of intra and inter-molecular hydrogen bonds among hydroxyl groups in cellulose can be accomplished with the interactions among sulphuric acid and cellulose[22]. Pre-treatment using concentrated sulphuric acid help in breaking down the crystalline arrangement of the cellulose[22].

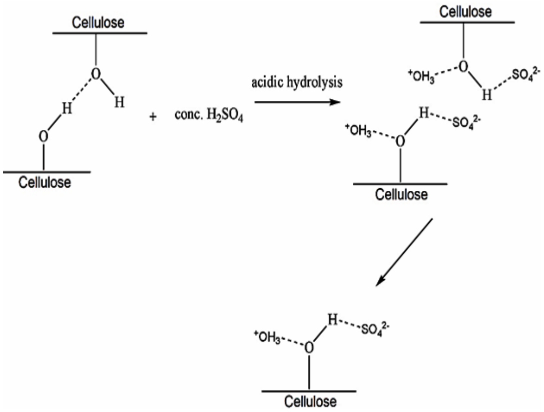

Fig. 3. Cellulose dissolution mechanism (adopted from Kong-Win Chang *et al.,* 2018)[22]

Time, temperature, and acid concentration are important factors in determining the final nanocellulose characteristics while the most important disadvantage of acid hydrolysis is the formation of acid wastewater during washing to enable the pH value neutralization of the nitrocellulose solution[17]. After acid hydrolyzing lignocellulosic biomass, the resulting particles which are thin, rod, or whisker-shaped are known as cellulose nanocrystals are almost pure cellulose particles known by different names such as nanocrystalline cellulose, cellulose whiskers, or cellulose nano whiskers[18]. Purification of the cellulose fibers through a series of chemical processes is necessary to get cellulose nanocrystals[20]. During the esterification process with the hydroxyl groups on the cellulose surface, the anionic sulphate ester groups form a negative electrostatic layer on the surface of the nanocrystals, facilitating their dispersal in water[21]. At 60-70% H_2SO_4 for 10-12 min at temperatures between 40°C and 80°C, acid hydrolysis generally includes 5-15 g of fiber per 100 cm^3 of acid dissolution[21]. The amorphous parts of cellulose are separated from the crystalline sections by hydronium ions penetrating the cellulose chain in an acid process and hydrolyzing the glucosidic linkages between the amorphous areas and it should be noted that based on the plant and extraction procedure, the nanocrystals might have different sizes, shapes, and crystallinities[21]. Isolating microcrystalline cellulose from various natural sources for use in functional goods of high-added value is still a topic of active study and numerous techniques have been documented for isolating microcrystalline cellulose from lignocellulosic wastes, such as the use of sodium chlorite, followed by NaOH and acid hydrolysis, to produce it from sisal fibers; the mercerization of 12% NaOH at normal room temperature for 2 h the acid hydrolysis of the resulting mercerized material; and finally the treatment of the resulting mercerized material with NaOH; and the use of phosphotungstic acid[23]. After washing, the pH of a combination may be neutralized by adding cold water and spinning the mixture in a centrifuge[24]. The pH value can also be neutralized by washing using an alkaline such as sodium hydroxide[24]. For isolating nanocellulose, researchers hydrolyzed biomass using 47% sulfuric acid, and upon completion of the reaction, the acid was washed away using deionized water and centrifugation, the suspension was neutralized using 0.5 N sodium hydroxide, and the whole thing was washed again using distilled water[24]. A catalyst named 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) and the major oxidant, hypochlorite, may transform the hydroxyl group of the cellulose to carboxylates whereby the end product is nano fibrillated cellulose, which is 3–4nm in diameter and a few microns in length, high aspect ratio, and has a carboxylic acid surface[25]. By dissolving TEMPO and NaBr in high-pH water and then adding sodium Hypochlorite to start the oxidation, TEMPO-mediated oxidation can be achieved. The TEMPO/NaClO/$NaClO_2$ oxidation method may also be employed in neutral or somewhat acidic conditions and the application areas for TEMPO-oxidized cellulose nanofibers include flexible and see-through displays, gas-barrier films used for packaging, and nanofiber fillers used for composite materials[25]. The cellulosic fibers must be subjected to a mechanical disintegration procedure that induces fiber delamination separating the nanometric fibers and large amounts of energy are required to ensure the efficiency of the nano-fibrillation process[13]. Many variations of pre-treatments for cellulose fiber before nano-fibrillation are available[13]. The crystallinity and thermal stability of the produced nanocellulose significantly improved when compared with those of the natural wood fibers and when significant shear force was applied to cellulose fibers, the cellulose fibers adhered along their longitudinal axis, isolating the fibrils and producing nano-fibrillated cellulose[26]. High-pressure homogenization, ultrasonication, and ball milling were the most commonly used mechanical procedures[26]. Conversion of cellulose into glucose may be achieved using enzymes that help to catalyze the breakup of the cellulose polymer into small branches of the polymer[27]. Enzymatic hydrolysis is a viable option for producing nanocellulose, but is still a work in progress compared to alkaline and acid methods[27]. Enzymatic hydrolysis breaks down or alters cellulose fibers with the use of enzymes and the biological treatments using enzymes often require only moderate conditions, but a lengthy period of operation therefore enzymatic hydrolysis is often combined with other techniques[26]. The process of enzymatic hydrolysis using laccase was investigated for its potential use in separating the cellulose fibers from the wood chips after pre-treatment with an ionic liquid to increase the available area at the surface[26]. The process of high-pressure homogenization mostly involved the introduction of a cellulose slurry into a pressurized, fast-moving vessel and thereby the cellulose microfibrils were shattered into nanometer-sized pieces through impact force and shear force in the fluid[28]. When high-pressure homogenization was used to separate nanocellulose from sugarcane bagasse, the nanocellulose produced exhibited a size range of 10-20nm and less crystallinity when compared with the starting material[28]. The resulting material had a diameter of about 20nm but exhibited weaker thermal stability and crystallinity in comparison to the raw cotton cellulose when high-pressure homogenization was used to separate nanocellulose from cotton cellulose[28]. The disruption of cellulose's intermolecular and intramolecular hydrogen bonding during the high-pressure homogenization process resulted in reduced crystallinity[28]. When cellulose fiber was subjected to ultrasonication, the hydrodynamic forces of the ultrasound broke down the cellulose fibers so that they could be processed more easily and it was also found that the ultrasonication technique generates mechanical oscillating power by having molecules of a liquid absorb ultrasonic energy, which causes the development, expansion, and implosion of small gas bubbles[29]. A mechanical method called ball milling defibrillates cellulose fibers wherein the shear forces between the balls and the jar's surface, as well as those between the balls themselves, are generated by the centrifugal force generated by the jar's rotation and thereby the cellulose fibrils are broken down to a smaller diameter[29]. The biggest problem of mechanical technique, however, is the considerable energy consumption, and consequently, various pre-treatment methods are often used in conjunction with the mechanical process to reduce energy consumption[29]. The principal conventional extraction procedures are sulfuric acid hydrolysis for nanocrystalline cellulose synthesis and nano-fibrillated cellulose production using mechanical treatment. Envisaging the thermal behaviour of microcrystalline cellulose under thermal stresses may also be done with the use of differential thermal analysis, differential scanning calorimetry, and thermogravimetric analysis and it is very significant to note that the origin and separation method has a significant impact on the diameter of microcrystalline cellulose fibers[14]. Microcrystalline cellulose is a refined wood pulp product30. Microcrystalline cellulose can be used as an extender, anti-caking agent, viscosity-increasing agent, texturizer, emulsifier, abrasive, binder, bulking agent, emulsion stabilizer, slip modifier, and bulking agent[30]. Microcrystalline cellulose may also be used as an alternative to carboxymethylcellulose and in vitamin supplements[30]. Microcrystalline cellulose is known for its effectiveness as a thickener, stabilizer, and emulsifier and therefore has many uses in the cosmetics industry[30].

The size of the particle, capability to absorb moisture, the amount of moisture present, density, compressibility index, powder porosity, crystallinity index, hydration swelling capacity, crystallite size, and mechanical engineering properties like tensile strength and hardness are taken into account when determining microcrystalline cellulose viability for industrial use[14]. Nano cellulose's biodegradability and exceptional qualities (good mechanical strength, excellent thermal characteristics in conjunction with low weight and high transparency) make it a desirable material for use in a wide variety of contexts, including nanocomposite constructions, surface-modified substances, translucent papers with novel features, filler in polymer matrices, create a variety of products including the blade of a wind turbine, lightweight armor, and flexible batteries[31]. Nanocellulose derived from soybeans when added to different types of synthetic polymers showed that the nanocellulose reinforced polymers were substantially stronger and stiffer than the pure base polymers[31]. Nanocellulose has a large area on the surface and a huge concentration of hydroxyl groups making it a valuable material for accomplishing surface modification[32]. Direct chemical alteration or covalent interaction with the surface hydroxyl groups of nanocellulose and polymer grafting from nanocellulose is a common method for modifying the grafted polymers and nanocomposite materials[32]. One of the nanocellulose surface modification applications is the fabrication of amphiphilic surfaces. An amphiphilic surface is resistant to damage from both polar and non-polar liquids and surface features such as self-cleaning, anti-bacterial, anti-reflective, resistance to corrosion, etc., are related to anti-wetting characteristics[33]. Nanocellulose increases the hydrophobization of substrates by chemical means resulting in a wettability shift[33]. Nanocellulose is modified by reacting with its hydroxyl groups for a wide range of uses and qualities, including etherification, silylation, carbonylation, and amidation[33]. Nanocellulose was used in the medical profession because of its non-toxic, renewable, strong biocompatibility, and outstanding physical qualities[34]. Nanofibrillated cellulose extracted from bleached birch pulps was found to be biocompatible with skin transplant donor sites when used in dressing wounds wherein it was observed that the nanocellulose bandage stuck securely to the wound and peeled off easily after the skin was healed[34]. Nanocellulose has the potential to be used in a variety of other medical applications including medication delivery into targeted cells, soft tissue implants, and replacement of blood vessels. Nanocrystalline cellulose having functionalized chains at both ends, like the new hairy cellulose nano crystalloid, has several potential applications35. Scientists were able to isolate nanocellulose from a sheet of softwood pulp with the help of a chemical process that yielded nanocrystalline celluloses with varying carboxyl concentrations in their projecting chains, each of which affected the surface charge[35]. Nanocrystalline cellulose with carboxylated chains is appropriate as the carrier for nanomedicine since it does not clump in serum and may be taken up by different cells[35]. Modifications were attempted to electrostatically stabilize nanocrystalline cellulose to create transparent films. The films, which were composed of carboxyl groups in extended chains of electrostatically stabilized nanocrystalline cellulose had a light transmittance of 87% and when treated with trichloromethyl silane, the clear films also displayed hydrophobicity[36]. The potential uses of biodegradable films are flexible packaging and filtration of copper ions out of wastewater[36]. Researchers used periodate/chlorite oxidation to remove the electrostatically stabilized nanocrystalline cellulose from wood fibers, resulting in nanocrystalline cellulose with decarboxylated projecting chains, which resulted in the electrostatically stabilized nanocrystalline cellulose being effective in removing copper at concentrations as high as 185 mg/g[36]. Another potential use for nanocellulose is in the treatment of wastewater by removing metal ions. Aerogels containing bifunctional hairy nanocrystalline cellulose and carboxymethylated chitosan were effectively produced by cross-linking and the negative charges and high porosity of these biodegradable aerogels allowed them to effectively absorb methylene blue dye at a rate of up to 785 mg/g showing the path for the development of novel nanocellulose based bio-adsorbents[36]. When material strength, flexibility, and specialized nano structuration are required, nanocellulose is a viable material and phosphorylated nanocellulose has a wide range of uses such as a support for catalysts, adsorbents, and bone scaffolds[37]. Nanocellulose has found attention as a nanoscale material for the reinforcement of polymer matrix composites due to its mechanical characteristics, renewability, and biodegradability. Nanocellulose has gained importance on account of its abundant availability, superior mechanical and optical properties, good biocompatibility, and applications in material science to biomedical engineering[38]. Nanocellulose surface chemical functional capability has been explored for the ensuing applications in the key fields of nanocellulose research, including drug administration, bio-sensing/bio-imaging, tissue regeneration, and bio-printing[38]. Researchers are increasingly interested in creating ecologically acceptable extraction methods due to the enormous potential of cellulose nanoparticles[8]. The cellulose nanocrystals exhibit different properties as physical and chemical surface functionalization renders them into different functional groups and as a result, the cellulose nanocrystals have the potential to be used in many applications apart from being used in a variety of applications relating to composites, packing, cosmetics, food, optoelectronic devices, biomedical and pharmacy[4]. Nanocellulose being environmentally friendly and biodegradable can be used as an alternative biomaterial to synthetic materials and cellulose nanocomposites are used in electronics, vehicles, medical, building, packaging, and wastewater treatment applications[39].

Characterization and a good understanding of nanocellulose properties can be obtained using different analytical techniques[39]. The carious nanocellulose characterization techniques are shown in Fig. 4. Dimensional analysis of nanocellulose can be performed using microscopic techniques like field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), and atomic force microscopy (AFM) while it has also been reported that AFM can be used to investigate the topography, morphology and mechanical properties of nanocellulose[39]. Dynamic light scattering (DLS) technique is employed to find the hydrodynamic size and it has been reported that the DLS method gives data about the apparent diameter which can be used in combination with other methods to check the consistency of cellulose nanocrystals and the size change throughout time[39]. Investigations concerning the rheological properties of the aqueous nanocellulose suspension are essential since the rheological properties influence the making, processing, and combination of nanocellulose with different materials in industrial uses[39]. The composition of elemental nature on the surface of the nanocellulose can be studied using the X-ray photoelectron spectroscopy (XPS) method, while the factors relating to the molecular arrangement and purity of the nanocellulose can be ascertained with the help of nuclear magnetic resonance (NMR) method and further data regarding the conformation regarding the presence of the carbon atoms in cellulose can be provided by MNR spectroscopy[39]. It is reported that thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and differential thermogravimetry (DTG) techniques are used to determine the thermal stability of nanocellulose[39].

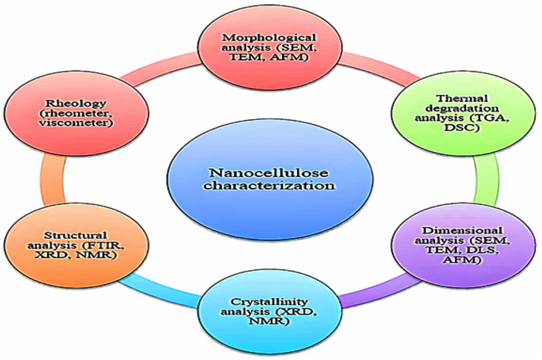

Fig. 4. Nanocellulose characterization techniques (adopted from Kaur *et al.,* 2021)[39]

Conclusion

The word nanocellulose is commonly used interchangeably with microcellulose, both of which refer to specific kinds of cellulosic particles. Biomass is a good source of raw material for nanocellulose synthesis and its high value-added applications. Depending on the structure, cellulosic fibers may be broken down into nanocellulose, which is a material with very precise diameter and length dimensions. Due to the crystalline structure of cellulose, nanocellulose may be extracted from lignocellulosic biomass using methods that break down the cellulose strands into either cellulose nanocrystals or cellulose nanofiber. The most common approaches for generating cellulose nanocrystals and cellulose nanofiber are acid hydrolysis and mechanical treatments, however, both these approaches have problems concerning the environmental and economic aspects such as the energy requirement for the process and also the significant amount of water needed in the neutralization stages. Enzymatic hydrolysis is another viable option for manufacturing nanocellulose, with the benefits of enzymatic treatments providing an ecologically benign and sustainable solution, despite the enzyme cost potentially being an inconvenience. To promote cellulose bundle fibrillation and minimize energy needs for separation operations, research is focused on the use of enzymes coupled together with mechanical treatments. Cellulose nanofibers can be obtained through enzymatic and/or mechanical fragmentation processes. Rod-shaped, highly crystalline cellulose nanocrystals are typically realized with the help of hydrolysis with concentrated mineral acids. Nanocellulose has a wide range of potential uses because of its high-quality mechanical, optical, and thermal qualities. Nanocellulose-based materials are related to biomedicine, packaging of food materials/products, mechanical reinforcement of matrices, membrane filtration, nanocomposites, gels, viscosity transformers, aerogels, films, fibers, barrier layers, foams, and applications on energy. Technological and financial hurdles should be overcome before implementing mass manufacturing. Many additional agricultural wastes may be explored for the extraction of nanocellulose and further preparation of new composite materials for industrial and commercial applications.

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Conflict of interest

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