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Nanocellulose from Pineapple Leaf and Its Applications towards High-value Engineering Materials

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Pineapple is one of the most widespread crops in the world. High volume of pineapple leaf (PL) waste which is burnt directly on the farm or landfill site after harvest leads to negative impacts on the environment. However, PL residue can be considered an important potential source for renewable materials due to its significant cellulose content. As means to reduce waste and enhance cellulose-based materials, nanocellulose gains high attraction due to its special properties such as non-toxicity, biocompatibility, biodegradability, and 100 % environmental friendliness. Because of nanometer scale, nanocellulose becomes a promising material, which expresses high mechanical strength, extremely large surface area, good modifiability and results in various value-added applications, namely packaging, regenerative medicine, electronics, etc. Aiming to sustainability and feasibility, focusing on environmental-friendly resources like nanocellulose to generate high-value materials efficiently is one of the progressive actions. To summarize the common methods approached to utilize PL as raw materials to produce high-value engineering nanocellulose and its potential applications, this review focuses firstly on chemical and mechanical pre-treatment of PL to enrich cellulose content; secondly on the preparation of nanocellulose via conventional acid hydrolysis, enzymatic hydrolysis, and newly mechano-hydrolysis and its physicochemical properties as well as characteristic structure; finally on the sustainable applications of nanocellulose in production of renewable materials such as aerogel, hydrogel, nanocomposite, and other promising fields.

1. Introduction

Pineapple is the second-largest harvested fruit globally, occupying almost 1 M ha of worldwide plantation area (Kengkhetkit et al., 2018). The world production of pineapple in 2019 was around 28.18 Mt, as stated by the Food and Agriculture Organisation (FAO) (Shahbandeh, 2021). Instead of proper treatment, most pineapple wastes are currently disposed by landfilling in fields or burning, affecting the environment and health (Do et al., 2020b). When incinerating pineapple residues in open-air, toxic contamination compounds spread out, causing dangerous health issues and negative impacts on global warming (Do et al., 2020a). Thus, optimizing the use of raw materials is necessary for the current trend of sustainable agriculture development that brings useful products and fewer wastes per unit mass of raw material. The first step is to convert the enormous number of agricultural residues into value-added products (Santos et al., 2013).
Furthermore, the high cellulose content in PLs (36.3 ± 3.8 %) can be used for the isolation of cellulose and nanocellulose to produce high economic value materials or chemicals (Santos et al., 2013). PLs are common raw materials in cellulose recovery, paper production (Sibaly and Jeetah, 2017), textiles (Low et al., 2019), and fabrication of composites (Maniruzzaman et al., 2012). Nanocellulose exhibits vast attractive properties, including low toxicity, biocompatibility, biodegradability, eco-friendliness, high mechanical strength, large surface area, and good modifiability. These products are appliable in various value-added applications, namely packaging, regenerative medicine, electronics, etc. (Du et al., 2019).
This mini-review not only demonstrates existing common methods for the isolation of cellulose and nanocellulose from pineapple leaf wastes but also provides a comparison of the characteristics and the

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effectiveness of these methods. In addition, the review represents current and potential applications of pineapple leaf nanocellulose, especially in composite materials, the medical field, and aerogels.

2. Nanocellulose and its superior properties

In recent years, nanocellulose has been considered to be one of the most outstanding green materials for its excellent properties, such as high aspect ratio, high surface area, better mechanical properties, renewability, and biocompatibility (Sarno and Cirillo, 2019). The numerous hydroxyl functional groups allow a wide range of chemical reactions, creating various high-performing materials (Gan et al., 2020). Nanocellulose is classified into three main types based on morphological characteristics, including cellulose nanocrystals (CNCs), cellulose nanofibrils (CNFs), and bacterial nanocellulose (BNC) (Klemm et al., 2011). The morphological characteristic, size, and variability of each nanocellulose layer depend on the origin, the isolation, pre-treatment, and different processing conditions (Phanthong et al., 2018).
Nanocellulose shows many remarkable properties, such as chemical inertness, high strength, dimensional stability, low density, low coefficient of thermal expansion, and ability to modify its surface chemistry and low thermal expansion coefficient (Gan et al., 2020). Nanocellulose has been introduced as reinforcement in many studies, for example, natural rubber nanocomposites, polylactic acid nanocomposites, epoxy nanocomposites (Yan et al., 2019). The exceptional reinforcement of nanocellulose is due to its lightweight, high rigidity, and outstanding mechanical strength (Reshmy et al., 2020). However, the source of raw material, pre-treatment methods, and isolation process may affect the properties of nanocellulose (Liu et al., 2016).

3. Nanocellulose extraction from pineapple leaves

Various agricultural residues, such as coconut, wheat, and especially pineapples, can be a valuable source of renewable materials. The chemical components of the biomass used are shown in Table 1.
Due to containing many impurities such as lignin, hemicellulose, ash, etc., PLs need to be pre-treated to produce high purity nanocellulose. The higher the concentration of cellulose is, the more suitable it is for being used to extract nanocellulose. The nanocellulose obtained from pre-treated PLs has three main categories depending on synthesis method: (i) nanocrystals by chemical hydrolysis, (ii) nanofibers by advanced grinding, and (iii) bacterial nanocellulose.

Table 1: Chemical compositions of biomass.

Biomass	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Ref
Pineapple leaves	36	23	28	3	(Santos et al., 2013)
Coconut fiber	32	-	38	-	(Cerqueira et al., 2017)
Wheat straw	30	50	20	-	(Saha, 2003)

3.1 Pre-treatment of pineapple leaves

One of the most promising pretreatment technologies for industrial manufacturing is acid hydrolysis. The hydrothermal process uses water in the liquid or vapor phase, does not require catalysts, and does not lead to significant corrosion, making it a gentle approach. The alkaline treatment removes lignin and increases cellulose content. Compared to acid processes and hydrothermal, mild alkaline pretreatment can reduce the solubility of hemicelluloses, prevent the generation of inhibitory chemicals, and can be carried out at lower temperatures. The oxidant methods consist of using peroxide, ozonolysis, and wet oxidation. The application of oxidants for pre-treating lignocellulosic biomass to gets disruption the linking between carbohydrates and lignin but these methods can be decreased cellulose crystallization. Oxidative methods consist of alkaline peroxide pre-treatment, ozonolysis, and wet oxidation. Furthermore, combining moistened oxidation with alkaline substances minimizes the risk of creating furan and phenolic aldehydes (Jönsson and Martín, 2016).

3.2 Methods for isolation of nanocellulose from pineapple leaves

Depending on the extraction method, the shapes and properties of obtained nanocellulose from PL can be different. There are three typical processes for the extraction of nanocellulose: acid hydrolysis, enzymatic hydrolysis, mechanical methods.
The steam explosion process is one of the mechanical methods to isolate cellulose nanofibrils from pineapple leaf fibers (PLF). The alkaline treatment by NaOH in an autoclave causes the degradation of lignin to form hydroxyl, carbonyl, and carboxylic groups. After that, the bleaching procedure also aids in the increase of cellulose content that nearly all lignin and hemicellulose have been eliminated by using a mixture of sodium hypochlorite and sodium acetate buffer. Then, the fibers were extracted with oxalic acid 11 % at 20 lb of pressure in an autoclave. Whiskers have regular dimensions of 5 to 60 nm in diameter and 200 to 300 nm in length after

the steam explosion in oxalic acid solution. The steam treatment effectively increases the content of cellulose to 93.45 %. The crystalline cellulose increases from 35.9 % to 73.6 %. The unique morphology of web-like nanofibers can be created by acid coupled steam explosion treatment and adding the high-pressure defibrillation. As a result, it raises the crystallinity index, increases surface area, and reduces the degree of polymerization (Cherian et al., 2010).

Chemical treatment is the method that utilizes sodium chlorite and alkali medium to remove components other than cellulose. After that, nanocellulose from PLs is extracted using acid hydrolysis under vigorous and constant stirring. For a reaction time of 30 min, the narrow and long CNC shows high starting decomposition temperature stability (225 °C), high crystalline index (73 %), the mean size range of 249.7 ± 51.5 nm in length, 4.45 ± 1.41 nm in diameter, and aspect ratio of about 60 (Santos et al., 2013). Using acid hydrolysis with stirring is easy to do in the lab and requires less equipment than steam explosion. Curauá - an Amazonian pineapple plant is used to generate fibers by alkali and bleaching treatments. Enzymatic processes and sonication are then applied to obtain nanofibers. Endoglucanase, which consists of hemicellulases and pectinases, is a novel method for the synthesis of nanocellulose. The obtained fibers nano size with their diameter and length of 2-12 and 84-300 nm (de Campos et al., 2013). Despite being less effective than acid hydrolysis, enzymatic hydrolysis does not release toxic residues and can be conducted under room temperature and pressure for energy-saving purposes. To limit the usage of chemicals in pretreatment, a combination of high-shear homogenization and ultrasonication is used. One-hour sonication is enough to get high purity nanocellulose with a size average of 68 nm in diameter and less than 88-1100 nm in length. The thermal stability of nanocellulose after ultrasonication reaches 320 °C higher than raw PL (215 °C) (Mahardika et al., 2018). Accordingly, the mechanical treatment uses fewer chemicals than the alkali and bleaching methods, but they excessively destruct fiber structure and require more energy than others. Pressurized acid hydrolysis procedures are used to extract nanocellulose from PLs. Alkali and bleaching are used to pre-treat PLs, followed by acid hydrolysis and pressurized acid hydrolysis to extract rod-like nanocellulose with diameters of 130 and 117 nm. When CNCs are extracted by acid hydrolysis and pressured acid hydrolysis, the crystallinity index (CrI) is 92.95 % and 91.24 % (Chawalitsakunchai et al., 2019). Pressured acid hydrolysis can reduce the fiber size but disrupt the structure of nanocellulose leading to the decrease in CrI.

The chemo-mechanical method is a new method to reduce the demand for comprehensive chemical pre-treatment. Only chemical treatment, sonication, and milling are used to extract nanocellulose. The pre-treatment aim is to eliminate lignin, hemicellulose, and others substances. After a single bleaching step, acid hydrolysis is used to isolate nanocellulose with a high degree of purity. This procedure is said to be less harmful to the environment than previous repeated bleaching procedures. Furthermore, ultrasonication can be used to depolymerize cellulose, which is a very successful method. Milling is useful to obtain high purity nanocellulose fibers with an average diameter of 25-68 nm and a length of less than 120 – 1,700 nm (Gadzama et al., 2020). This treatment reduces both the expense of chemicals and the environmental impact.

4. Applications of nanocellulose from pineapple leaves

Nanocellulose is an "invisible" material with intriguing qualities such as a large specific surface area and great mechanical strength, making it ideal for a wide range of nano-applications, for instance, the preparation of transparent coatings/film (Fang et al., 2019); the resistance to a variety of solvents, namely, water, toluene, methanol and dimethylacetamide (Österberg et al., 2013); barrier applications, especially for packaging (Wang et al., 2018); the reinforcement (Sanchez-Salvador et al., 2020). The higher aspect ratio and fiber imbroglios of CNFs lead to higher strength and modulus in comparison with cellulose nanocrystals. However, they exhibit a lower level of strain-at-failure due to their moderately large fiber clusters (Xu et al., 2013). Nanocellulose extracted from PLs has been utilized in varieties of functions which can be divided into three main regions: composite materials, the medical industry, and aerogel materials.

4.1 Nanocellulose from pineapple leaves in composite applications

Nanocellulose could form good intermolecular bonds with the polymer matrix owing to the various interactions with other components via van der Waals' forces, covalent bonds, molecular entanglement, and mechanical interlocking. In addition, the plenty of hydroxyl groups on nanocellulose helps it to be functionalized because of easy interaction with other substances (Reshmy et al., 2020). Owing to various special properties of nanocellulose in combination with the abundance and eco-friendliness of PLs, extracted nanocellulose has been attracted scientists' attention to be used in biocomposites. Great mechanical properties and high stiffness of nanocellulose make it possible for reinforcement (Gan et al., 2020). The addition of nanocellulose in the composite matrix results in the improvement in the mechanical properties of the material. For example, chitosan films added with nanocellulose between 1 to 5 % showed higher tensile strength compared to a bare one with the highest value at nanocellulose concentration of 3 % (Amalia et al., 2018). The addition of nanocellulose improved the tensile strength of the poly (lactic acid) matrix by 5.4-22.7 % (Shih et al., 2017). The compressive strength of biobased polyurethane foams increased by 15 % with the presence of 2 parts per hundred rubbers nanocellulose (Zhou et al., 2018). The storage module of chitosan-starch nanocomposite was enhanced up to 90 % with only 0.7 % of nanocellulose (Almendárez-Camarillo et al., 2020). Thanks to its extremely "tiny" size, nanocellulose is nearly unobserved in the composites which can be applied for the fabrication of transparent film (Amalia et al., 2019). The strong interactions between nanocellulose or nanocellulose and other polymers, such as polylactic acid, poly hydroxybutyrate, polyvinyl alcohol, etc. create a well-built network to obstruct the movement of the molecules that are highly recommended for barrier applications (Nair et al., 2014). For example, when nanocellulose content in Tapioca/Polyvinyl alcohol composites increases from 1 to 3 %, the water vapor transmission rate decreases from 49 to 39 g/(m^2.day) (Listyarini et al., 2020). Furthermore, nanocellulose can improve the thermal stability of the material, for example, the melting temperature of the polyvinyl alcohol-based film rises from 320.15 to 325.49 ^{o}C when the nanocellulose content increase from 10 to 30 % (Wahyuningsih et al., 2016). Recently, many progressive studies have been made to take advantage of nanocellulose for the preparation of efficient and economical biocomposites, especially the production on the industrial scale (Reshmy et al., 2020).

4.2 Potential application of nanocellulose from pineapple leaves for the medical industry

For medical applications, the materials used should have biocompatibility, nontoxicity, high chemical stability, good mechanical properties, and cost-effectiveness (Gumrah Dumanli, 2017). Several studies have been studied on nanocellulose isolated from PLs for utilization in medical applications. For instance, pineapple leaf nanocellulose is mixed with polyvinyl alcohol and *Stryphnodendron adstringens* bark extract to synthesize bio-nanocomposite used for tissue engineering (Costa et al., 2013). Nanocellulose from PLs is also combined with polyurethane for the fabrication of medical implants like heart valves and vascular grafts (Cherian et al., 2011). In recent years, high surface area and high polymerization of nanocellulose are superior features indicating its potential use in drug delivery systems carrying bioactive components (Hasan et al., 2020). Hence, more studies in this region should be carried out to exploit the outstanding properties of nanocellulose from PLs.

4.3 Potential application of nanocellulose from pineapple leaves for the fabrication of aerogels

Nowadays, there are many reports regarding the utilization of cellulose for the fabrication of ultra-lightweight aerogels. Compared with cellulose-based aerogels, nanocellulose-based aerogels express a higher surface area and contain more active sites because of the "tiny" size of nanocellulose (Gopakumar et al., 2019). Therefore, aerogels originated from nanocellulose have been studied for a large range of applications including adsorption (Gu et al., 2020), drug delivery (Bhandari et al., 2017), and thermal insulation (Duong and Nguyen, 2016). There has been no research on using nanocellulose from pineapple leaves for aerogel fabrication. More research must be conducted on the utilization of nanocellulose from PLs for the synthesis of high-value engineering aerogels.

5. Conclusions

This short review is to summarize some typical methods for the production of nanocellulose from PLs and their broad applications. Both conventional and advanced methods such as acid hydrolysis, mechanical method, chemo-mechanical method, enzymatic hydrolysis have been applied to isolate nanocellulose from PLs. In terms of production cost and purpose, cost-effective methods should be considered to produce nanocellulose and its value-added materials from PLs. Shortly, a simple, effective, and economical method will be highly paid attention to diminishing the cost and the waste to the environment due to the multi-step chemical treatment. Application prospects of nanocellulose from pineapple leaf are diverse from reinforcement to biomedical applications. Numerous research investigations related to nanocellulose extraction from pineapple leaves have been conducted to optimize the properties, the production effectiveness, the economic efficiency of nanocellulose, and its potential in various regions that can deal with many challenges of modern society. Therefore, the studies on nanocellulose extraction from biomass sources, especially PLs, play a noticeable role in material science around the world in the future.

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