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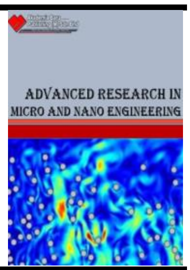


# A Mini Review on Recent Nanocellulose Production from Natural Fibers and Lignocellulosic Biomass via Acid Hydrolysis

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**ABSTRACT**

This review examines the recent advancements in the synthesis of nanocellulose using acid hydrolysis techniques and natural fibres. It focuses on the optimisation of reaction conditions, innovative hydrolysis methods, and the integration of environmentally sustainable and economically viable practices. Through the analysis of contemporary literature, this paper highlights the improvements in yield and quality of cellulose nanocrystals (CNCs) derived from various lignocellulosic biomass sources. Key studies demonstrate that precise control of reaction parameters such as acid concentration, temperature, and duration can significantly enhance CNCs yield. Additionally, technological advancements have facilitated more sustainable and cost-effective production processes, suggesting a promising future for applying nanocellulose widely across various industries.

## 1. Introduction

Among the nanofillers, nanocellulose has attracted many researchers' interests because of its excellent features, including high tensile strength, low density, and biodegradability, which are attributed to the natural cellulose source. These characteristics have placed nanocellulose in a position to be used for biomedical, environmental as well as industrial purposes. In the study by Noremylia *et al.,* [1], the authors indicated the versatility of nanocellulose in different fields with a focus on application in water treatment and biomedical sector inclusive of dressing and regeneration of tissues. The paper's assessment is thorough, this work supports the premise of nanocellulose being friendly to the environment despite its argument in some sections requiring more real life data evidence. The role of nanocellulose in the use of various industries, and medicine, indicates that the mechanical characteristics of nanocellulose and biodegradability that has been described as being able to replace synthetic polymers [2-6].

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The processes of synthesizing nanocellulose can be in several ways with each of them being characterized by certain strengths and weaknesses. The common approaches include; mechanical methods, chemical methods, and enzymatic methods. Further, detailing the same, Moyo *et al.,* [7] pointed out that mechanical approaches, being efficient, are normally associated with high energy usage. Chemical approaches particularly through the process known as acid hydrolysis produce high purity nanocellulose but come with what is considered an eco-unfriendly process. Enzymes on the other hand are environment friendly but slower and more expensive than other methods of deterging. These techniques are contrasted in the review and the benefits and drawbacks of each are stated clearly. Moreover, the study done by Perera *et al.,* [8] has provided a similar stimulus in addition to discharging the efficiency of chemical methods in the production of good quality nanocellulose. But it also reveals the worrying problem of pollution and the requirement to work on this issue more actively. Their comparative analysis gives a middle of the road view of the current state of the synthesis techniques, but the research could expand on the category of green technologies that is currently rather shallow. Also, Ahsan *et al.,* [9] have surveyed several synthesis techniques to showcase that each must be selective depending on the application requirements and resources.

## 2. Acid Hydrolysis Methods

Acid hydrolysis is another important technique in deriving nanocellulose where potassium hydroxide (KOH) solution is used to dissolve cellulose and then, sulfuric acid ($H_2SO_4$) or hydrochloric acid (HCl) is used to hydrolyse the cellulose chains and obtain cellulose nanocrystals (CNCs). The chosen method of sulfuric acid hydrolysis is highly effective due to the introduction of new sulphate groups, which allows improving the nanocellulose dispersibility in aqueous media. This process though creates major environmental impacts such as the production of hazardous waste and enormous water consumption in the neutralization of acids and washing [10]. Despite hydrochloric acid hydrolysis creating CNCs with dissimilar surface characteristics and improved thermal stability, it does not have the surface charges as in the case of sulfuric acid, thus aggregation becomes a problem [11]. Additionally, weaker acids such as phosphoric acid have been used, which while not as effective as hydrochloric acid, produces nanocellulose which has less crystallinity and undesirable characteristics for some applications [12]. Authors Mondal *et al.,* [13] stress the relevance of selecting the proper conditions for carrying out hydrolysis, namely, the concentration of the acid used, temperature, and time of the reaction with respect to the effectiveness and the impact on the natural environment. These are important improvements that can help make the method of acid hydrolysis more feasible for the making of nanocellulose. Furthermore, Burhani *et al.,* [14] explain that nanocellulose can also be used in environmental applications like microplastic removal and proves thus the essence of improving the hydrolysis procedures for producing eco-compatible nanocellulose.

### *2.1 Sulfuric Acid Hydrolysis*

Sulfuric acid hydrolysis is greatly preferable for the preparation of CNCs since it effectively hydrolyzes the amorphous part of the cellulose while not working on the crystalline part. In this method, new functional groups containing sulphate ester are created on the surface of the nanocellulose also improves the water dispersibility of nanocellulose and the stability of colloidal suspensions. Another successful attempt of extracting nanocellulose from waste tobacco stalks was confirmed by Jiang *et al.,* [15] in their study, they used sulfuric acid hydrolysis in combination with

TEMPO-mediated oxidation. However, this process is associated with the following limitations; formation of acidic wastewater, corrosion of equipment, and requirement of many washing steps to eliminate acid residues [16]. Thus, various modifications and optimisations have been proposed and investigated by researchers to overcome these problems. For example, Neenu *et al.,* [17] focused on the oxalic acid pre-treatment method before the sulfuric acid hydrolysis of biomass and concluded that improved efficiency in the hydrolysis takes place along with some decrease in using the environmental effects.

The subsequent developments have been directed at enhancing the characteristics and uses of sulfuric acid-hydrolysed nanocellulose. Thakur *et al.,* [18] studied further treatment of nanocellulose with other acids, such as phosphoric acid, to improve the thermal stability of nanocellulose and to control properties for certain applications. Also, Zhang *et al.,* [19] showed that in their study there is a need to incorporate steps like TEMPO-mediated oxidation before hydrolysis, as it also opens up other sites functionalization for the material that will be prepared for biomedical as well as environmental usage. These works prove the effectiveness of sulfuric acid hydrolysis as a general approach to the production of nanocellulose, with further development focusing on increasing yields, ecological friendliness, and the scope of potential applications. Furthermore, another group of researchers, Awang *et al.,* [20] focused on the employment of HCl hydrolyse in isolation of CNCs from waste office paper, which clearly explicates that waste materials can be effectively used as a source of nanocellulose (Figure 1). Such an approach allows for solving various problems concerning waste management while also using waste as a valuable material for multiple applications.

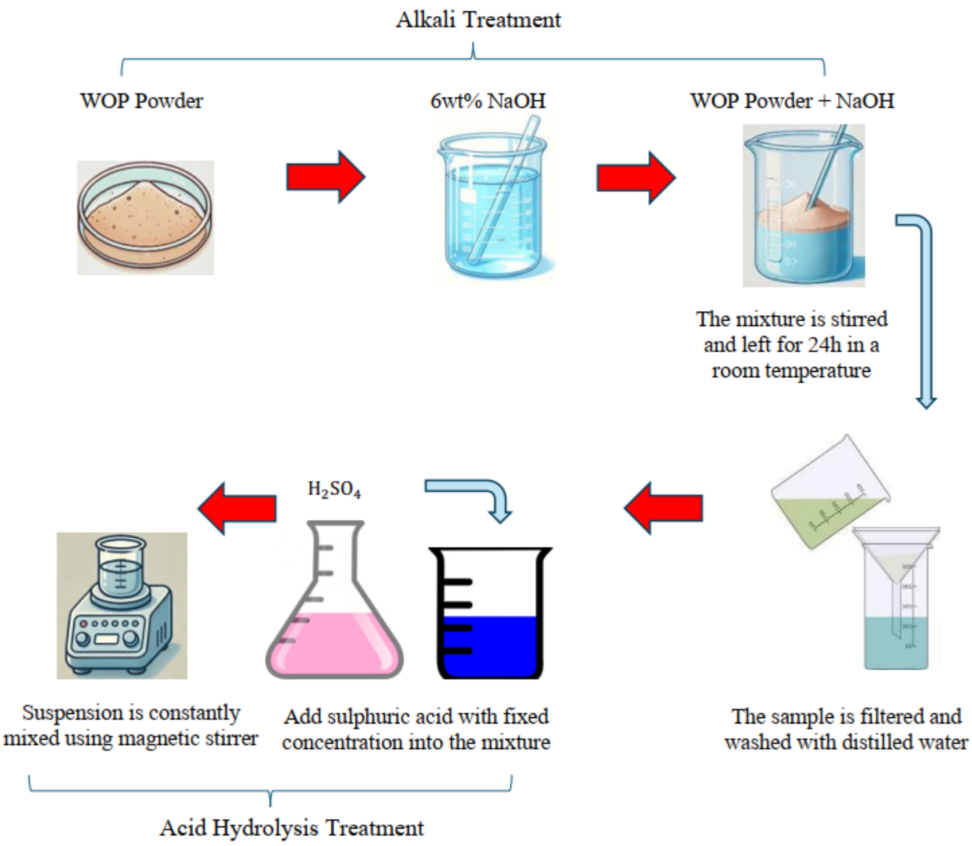


**Fig. 1.** Schematic illustration of sulfuric acid hydrolysis procedure, adapted from Awang *et al.,* [20]

### *2.2 Hydrochloric Acid Hydrolysis*

Since hydrochloric acid (HCl) hydrolysis has been reported to effectively cleave the glycosidic bond in cellulose to generate nanocellulose with high crystallinity, it is widely used in the preparation of CNCs. Wang *et al.,* [21] reported on the effect of HCl in hydrolysis and found that the use of HCl to produce CNCs provides increased thermal stability and possesses different surface chemistry as compared to CNCs which are produced by sulphuric acid hydrolysis as it introduces sulphate groups. These surface properties can be useful in specific applications such as in composites where there is a need for improved thermal stability. Shang *et al.,* [22] studied regression of hydrolysis time on the properties of nanocellulose and observed that hydrolysis of CNCs for greater periods increases crystallinity and enhances the mechanical characteristics of the nanofibrils, although it can also envisage bulky depolymerization of cellulose.

Subsequent advances with HCl hydrolysis have been directed towards improving the reaction parameters with improved yields but with consideration of the rate of degradation of the environment. Pawcenis *et al.,* [23] tried to identify how the different hydrolysis time could determine an important decrease in the environmental influence but with a valuable quality of the produced CNCs. In this regard, this study shows that it is vital for the process parameters to be optimized with the aim of improving both the sustainability and efficiencies in nanocellulose production. Moreover, Munawar *et al.,* [24] pointed out that further improvement in the quality and multifunctionality of CNCs may concern the combined application of HCl hydrolysis with other methods of pretreatment, including alkali treatment, which may open brand new opportunities for their usage, particularly in biomedical industry as well as in environment protection (Figure 2).

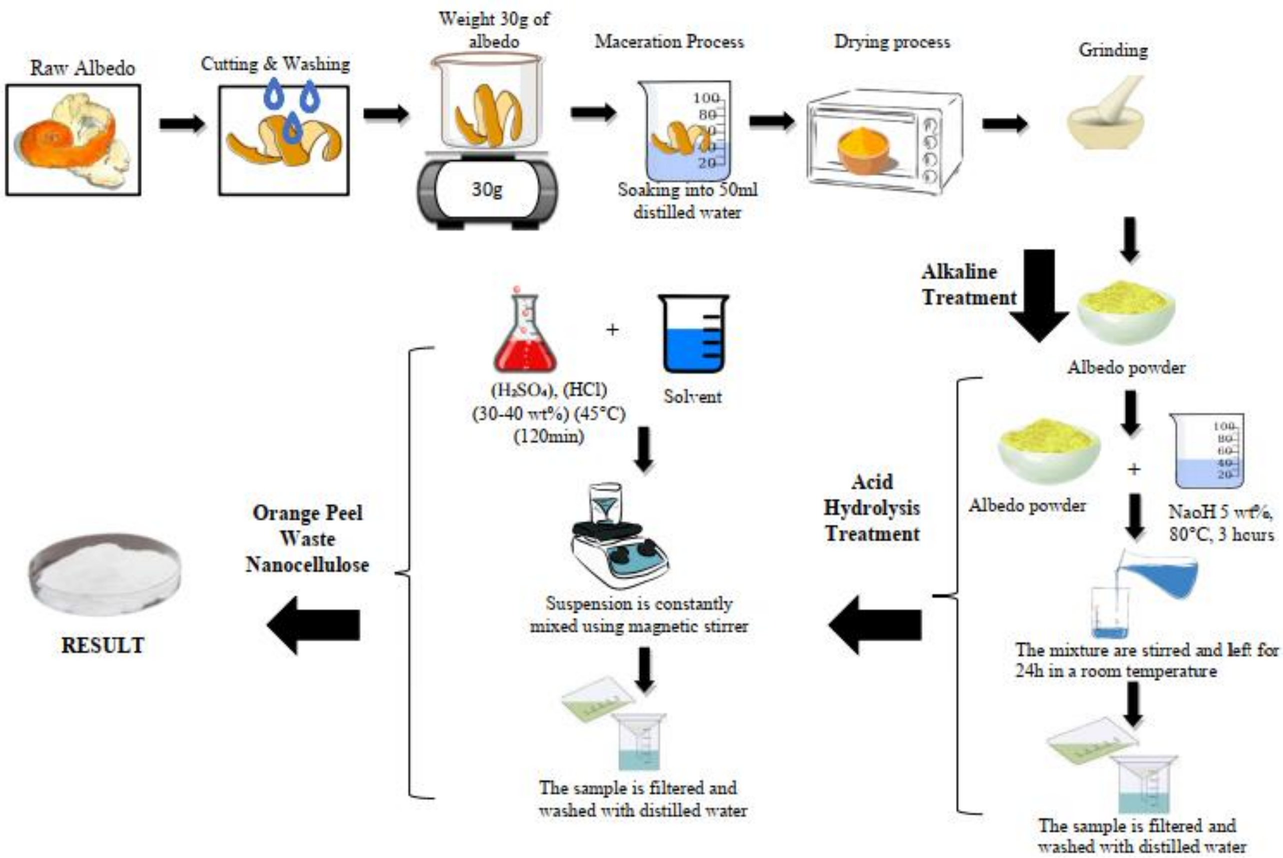


**Fig. 2.** Schematic illustration of hydrochloric acid hydrolysis procedure, adapted from Munawar *et al.,* [24]

### *2.3 Other Acid Hydrolysis Methods*

Analyzing other acids for hydrolysis besides the conventional ones, which are sulfuric and hydrochloric, shows the different possibilities and issues in nanocellulose processing. The use of phosphoric, oxalic as well as citric organic acids has been revealed to be capable of giving proper isolation of cellulose nanocrystals (CNCs) as well as cellulose nanofibrils (CNFs) with specific features. For example, in the case of interaction with zirconium phosphate, phosphoric acid has better effectiveness as less amount of acid is needed to extract nanocellulose and overall cost effectiveness increases. This method not only reduces the environmental effects of the product but also improves its equipment's life cycle [25]. This method of cellulose esterification and hydrolysis using oxalic acid in a solvent-free reaction has also been accomplished with commendable yields of surface-functionalized cellulose nanocrystals [26]. On the other hand, Citric acid hydrolysis has been pointed out for highly carboxylate nanocellulose as it advanced dispersion and functionalization in numerous matrices. These organic acids, however, normally require the use of relatively higher concentrations and longer time of reaction as compared to that of mineral acid which has a major drawback for large scale industrial applications [27].

The comparative analysis of different methods of acid hydrolysis presents the major differences in the rate of the process, the quality of obtained nanocellulose and the yield of the product. Sulfuric acid hydrolysis, nevertheless, widely used in producing nanocellulose, causes degradation in the thermal stability of the cellulose by the introduction of the sulphate groups in its structure, which reduces the thermal decomposition temperature of cellulose [28,29]. The use of phosphoric acid hydrolysis, which combines it with co-catalysts, gives better thermal stability of nanocellulose with less frequency of using acid [25]. The advantage of this method is that it has higher thermal resistance for a particular purpose. In the case of nanocellulose production via oxalic acid hydrolysis, the yield and particles uniform size were the highest, but the process requires complex equipment control to achieve the best reaction parameters and lower energy consumption [27]. In the study by Alhaji Mohammed *et al.,* [30], the different parameters of acid hydrolysis that affect the properties of the nanocellulose obtained from almond shells have been comparatively discussed. The study methodologically envisages the influence of the type of acid (sulfuric, perchloric, methanol ic sulfonic) and its concentration, reaction time and temperature on the yield, particle size and thermal characteristics of the extracted nanocellulose. Thus, tweaking of factors like acid concentration, reaction time, and temperature as well as the employment of mixed or organic acids are factors that should be considered as important in improving the yield and quality of nanocellulose. These technologies will yield improved and hydrogen selective hydrolysis, more environmentally friendly but at the same time realistic for factory use [31].

## **3. Natural Fibres and Lignocellulosic Materials**

The analysis of significant natural fibres and lignocellulosic materials presents major developments in the synthesis and employment of nanocellulose which include the multiple sources of natural fibres. Nanocellulose which is been obtained from lignocellulosic biomass possesses remarkable characteristics including high mechanical strength, thermal stability and biodegradability. New research investigations have focused on the production of cellulose nanocrystals (CNCs) and cellulose nanofibrils (CNFs) from various sources such as woody and non-woody plants, agricultural residues, and other lignocellulosic materials. These nanocellulose forms are synthesized through processes like acid hydrolysis and oxidation by using TEMPO after which the material has different levels of crystallinity and different mechanical properties that are crucial when using it in biomedical, packaging and composite fields [32-35].

The sources of natural fibres for nanocellulose production are numerous; they include familiar woods and cotton as well as typical agricultural residues such as corn husks and pineapple leaves. These fibres present an opportunity for the availability of a wide source of raw materials and enhance the sustainable use of nanocellulose. For example, corn husk and Juncus plant, the former which is an agricultural waste product and the latter which is a common plant all over the world have been efficiently used for the production of cellulose nanocrystals which exhibit mechanical characteristics that are at par with both conventional natural fibres like cotton and jute [36,37]. Thus, the utilization of agricultural residues will help in the fight against pollution of the environment by providing cheap raw materials for industrial uses. Further, when these natural fibres are incorporated into the composite materials, the resultant material gets better mechanical properties to find use in automobiles, construction, paper industries, and consumer products. Thus, the creation of environmentally friendly nanocellulose composite composites is recognized as a positive contribution to humankind in the area of material science [38-40].

## 4. Advancements in Acid Hydrolysis for Nanocellulose Production

In the current literature, enhancement of the unique techniques in the process of nanocellulose production, particularly acid hydrolysis has been deemed important. Such developments achieve the desirable conversion conditions for high product yield and quality, and low environmental pollution. There is strong evidence suggesting that Thakur *et al.,* [41] established that by controlling factors like; concentration of acid, temperature and time, the yield of CNCs can get as high as 90%. This study employed response surface methodology which brings out the potential of achieving high levels of Precision in the hydrolysis process with regard to the different factors affecting it depending on the level and range of the factors under consideration. In the same way, Sarangi *et al.,* [42] claimed that the relationship between cellulose fibres and acids should be in an appropriate proportion; high or low concentration-levels negatively influence the hydrolysis yield. These studies compiled together elucidate that it is critical to optimize the reaction conditions to achieve optimal yield with minimal compromise on quality with respect to nanocellulose fabrication.

New developments in acid hydrolysis include attempts to incorporate the aspects of protecting the environment and the cost aspect. A techno-economic study done by Penloglou *et al.,* [43] explicated that although there are high capital costs of the optimised hydrolysis processes, the operational costs and the effects on the environment can considerably be minimized. The study focused on different cases and concluded that the application of more sustainable practices like the use of enzymes in the process of hydrolysis is possible to bring costs even lower and make the process less harmful. Also, the economic aspect of these processes becomes more favourable as there are technological developments implemented to decrease energy intake, and other losses [44,45]. All these innovations depict a future with more probable opportunities for the extensive use of nanocellulose boosted by technological advancements and economic benefits.

## 5. Conclusions

Nanocellulose produced through acid hydrolysis has recorded tremendous progress in synthesis with the improvement in the reaction conditions and their integration with new approaches. Studies also show that fine tuning of factors such as the concentration of acid used, temperature and time of the reaction can significantly enhance the yield and characteristics of the cellulose nanocrystals

(CNCs). However, the incorporation of environmentally friendly procedures and the incorporation of the economic aspect has become paramount when it comes to the current trends in hydrolysis. Current research indicates that such improvements reduce the effect of nanocellulose on the environment hence the economic feasibility of its production and thus give the audience an assurance of the sustainability of the production. As evident from the current research, these methods are steadily being advanced toward affording scalable and cheap nanocellulose, which has the potential to be adopted widely in fields including but not limited to biomedical, packaging, and composites. Continuous research and development are still crucial in the achievement of these goals as a means of increasing the utilisation and parametrisation of nanocellulose from natural fibre and lignocellulosic materials.

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