

RECENT ADVANCEMENT IN NANO CELLULOSE AS A BIOMASS-BASED ADSORBENT FOR HEAVY METAL IONS REMOVAL: A REVIEW OF A SUSTAINABLE WASTE MANAGEMENT APPROACH

Review paper

UDC:628.316.12

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Abstract:

Rapid industrialization and unplanned urbanization have significantly increased environmental pollution. These human behaviors have resulted in massive waste discharges into the environment. As a result, organic and inorganic contaminants, including heavy metals, have accumulated in surface and groundwater. Heavy metals are highly carcinogenic and deadly. Heavy metal removal from drinking water has always been difficult. Conventional water treatment procedures could be more efficient, wasteful of energy, and generate massive amounts of harmful waste. In this initiative, researchers created a bio-based adsorption technology for removing heavy metal ions from polluted water. Nano celluloses (NCs) as biosorbents have sparked considerable attention due to their unique properties, such as the presence of several -OH groups on their surface, allowing the insertion of chemical moieties, a substantial specific surface area, strong mechanical properties, recyclability, and biodegradability. This review paper goes into great detail regarding the ways of producing Nano cellulose and its essential qualities. Many factors influence the use of NC-based adsorbents in water treatment systems, including synthesis pathways, functionalization of the surface, specific surface area, regeneration capacity, and reusability. Recent advances in bio-sorbent synthesis have prompted using bio-derived NC-based adsorbents in water treatment methods. This study also demonstrates that utilizing the potential for agricultural wastes, specifically sugarcane bagasse (SCB), as a precursor for Nano celluloses represents a sustainable approach, namely the conversion of low-value waste into a specific high-value product and its use in wastewater treatment.

ARTICLE HISTORY

Received: 13 October 2023

Revised: 30 November 2023

Accepted: 16 December 2023

Published: 31 December 2023

KEYWORDS

Sugarcane bagasse, biosorption, nano celluloses, heavy metals, development

1. INTRODUCTION

In recent years, many countries have seen massive development expansion in the infrastructure, industrial, and agricultural sectors. Commercial developments jeopardize technical discharge, drainage, sewage, road, and rubbish disposal requirements. Harmful pesticides and

chemical fertilizers used in agriculture run off and contaminate surface water. As a result, the primary drinking water supplies (groundwater and surface water) are extensively polluted with heavy metals that are potentially hazardous and carcinogenic, much beyond WHO criteria. Because of their carcinogenic and poisonous characteristics, heavy metals in water for drinking are a severe

environmental hazard that requires immediate response [1-3]. Heavy metals are critical water pollutants that have a long-term impact on health and the environment, and it is critical to remove them from water. Lately, many methods have been investigated to produce more affordable and more effective methods of removing pollutants from water.

Among many diverse innovations, adsorption is one of the most dynamic and technically feasible approaches for eliminating heavy metals from water. So far, activated carbon has proved the most effective adsorbent in the wastewater treatment process for heavy metal removal. Nonetheless, its use on an industrial scale is less economically appealing because of this material's high cost and energy-intensive regeneration [4-6]. As a consequence, alternative economical adsorbents have received increased scrutiny in the past few years in attempts to save operational costs. In light of this, using natural bio-based products has gained popularity. There has been much interest in recent years due to its eco-friendly characteristics, lower cost, excellent adsorption capacity, minimum sludge production, possibility for reuse regeneration, and general availability [7].

Among the most recent sophisticated investigations are biomimetic membranes. Aerobic sludge that is granular and has transparent exopolymer particles is linked. Membrane fouling, for example, has been employed as a high-performance water purification method [8-11].

Natural waste materials from agricultural sectors can be acquired and used as biosorbents with high heavy metal absorption potential. Cellulosic nanoparticles have been promoted as the most outstanding examples among numerous natural bio-based adsorbents used in water treatment in the integrated field of biotechnology and nanotechnology. The increasing quantity of research articles and patents registered in scientific databases demonstrates this [12].

Nano cellulose offers a wide range of applications in many fields, including sensor technology, cosmetics, catalysis, energy production, water remediation, paper industries, oil and gas industries, medicinal applications, and so on [13-15]. NCs are a unique material for water treatment operations due to their distinct qualities, such as high specific surface area, exquisite functionalization of the surface property, high aspect ratio, substantial mechanical strength, chemical inertness, and plentiful availability [16].

Furthermore, researchers have discovered that chemically altering the surface of Nano celluloses or reinforcing polymers can boost their efficacy [17-21]. These changes aid in increasing binding affinities and, as a result, adsorption capacities [22]. Although several review articles have highlighted the importance of nanocellulose-based adsorbents in water pollution control, many lack a clear and complete examination of Nano cellulose characteristics, extraction methods, and usage in water treatment technologies. In this review, a variety of strategies used to extract Nano celluloses from lignocellulosic biomass were examined. In addition, we have provided an overview of using sugarcane bagasse as a cellulosic precursor for Nano celluloses, a promising and sustainable biopolymer. Furthermore, we compiled the literature on improved nanocellulose-based adsorbents for eliminating heavy metal pollutants from water. The present obstacles and critical bottlenecks that limit the breadth of Nano cellulose extraction technologies and their practical implementation in water treatment systems are also discussed.

This investigation aims to provide insight into an integrated waste management method by utilizing agro-industrial waste for water treatment, minimizing environmental damage in both directions.

2. HEAVY METAL TOXICITY AND REMOVAL METHODS

Heavy metals occur naturally as materials that are considerably denser than water. Heavy metals are mostly obtained from businesses such as tanneries, pharmaceuticals, chemicals, electroplating, mining, alloy making, fertilizers, and so on [23]. Table 1 depicts the primary sources of heavy metals, their toxicity effects, and the permitted limits in drinking water indicated by WHO standards. Heavy metals (arsenic, mercury, cadmium, chromium, lead, nickel, copper, and so on) can even cause cancer. Despite being present in tiny amounts in drinking water, it poses a significant threat to humankind. Heavy metals from anthropogenic sources reach the environment. As ions that are hydrated or artificial complexes, matrices are formed. It is exceedingly poisonous, carcinogenic, non-biodegradable, and mobile in the environment [24, 25].

There is an urgent need to remove hazardous elemental ions from water used for drinking and residential reasons. Many studies have been

conducted to eliminate these elements due to their high carcinogenicity, which affects cellular organelles and their components involved in metabolic functions such as the formation of new molecules, detoxification, breakdown, and restoration of damage [26-28]. Many conventional technologies for heavy metal removal from water have been introduced recently, including adsorption, coagulation, flocculation, membrane separation, chemical precipitation, electrochemical deposition, and ion exchange. These traditional water treatment technologies have various advantages and downsides, as shown in Table 2,

most of these processes are inefficient, produce excess hazardous sludge, have high operating expenses, high energy and reagent requirements, and frequently result in partial contamination removal, among other things. Contamination with heavy metals in water is critical, particularly in emerging and impoverished nations where water treatment systems are expensive.

As a result, greater attention should be placed on alternative technology that is bio-based, less expensive, biodegradable, and effective in removing harmful heavy metals.

Table 1. Heavy metals, their hazardous effects on people, and the WHO-recommended limits in drinking water, (The WHO-recommended acceptable threshold for heavy metals is based on [29])

Heavy metals	Sources	Toxic consequences	WHO acceptable limits (mg/l)	References
Cadmium (Cd)	Used as an electrode in the manufacture of alkaline batteries and in the production of alloys.	DNA damage, apoptosis or programmed cell mortality, and disorders such as osteoporosis, renal failure, and adenocarcinomas.	0.005	[1]
Mercury (Hg)	Battery manufacturing units, dentists, Thermometers, and automobile industries.	High blood pressure and depression Promote carcinogenic pathways (DNA methylation, histone alterations).	0.001	[2]
Lead (Pb)	Tanneries, transport exhaust systems, lead bullets, metal processing units, and colored paints.	Disrupt metabolism processes such as transcription, translation, and so on, resulting in gene mutations and brain and kidney damage.	0.05	[24]
Chromium (Cr)	Metallurgical fields such as industrial welding and plating, chrome pigment manufacture, Oil well drilling, and fertilizers.	Genetic abnormalities, Anemia (Cr VI), neurological difficulties, breathing problems, and nose ulcers.	0.1	[26]
Arsenic (As)	Veterinary medications, wood preservatives, Paints, and dyes of various colors.	Produces free radicals, which cause cancer, leukopenia, hematological problems, and other diseases.	0.05	[30]

3. DEFINITION OF THE BIOSORPTION PROCESS

In the interests of economic development and environmental safety, scientists have developed an interest in bio-based solutions for water treatment in recent years. Several studies have suggested applying green technology to remove emerging pollutants from drinking water [38-40].

Bio sorption is one alternative strategy that involves the removal of pollutants from water

using biological materials, such as biomass products of microbes, animals, or plants [41, 42]. It is a type of adsorption in which biological materials (biosorbent) collect heavy metals (adsorbate) from effluent [43, 44]. It is a physicochemical process in which the bio sorbent-adsorbate binds with higher affinity through various mechanisms such as chemisorption, oxidation, complexation, reduction, electrostatic interaction, micro precipitation, ion exchange, aggregation, and chelation [45, 46].

Table 2. Comparison of the benefits and drawbacks of conventional approaches for contamination removal

Methods of heavy metal removal	Benefits	Drawbacks	References
Coagulation and flocculation	Coagulation is a chemical reaction that causes colloidal destabilization by opposing the forces that scatter them, while flocculation is the agglomeration of these particles. Straightforward procedure.	Sludge production is dangerous. The consumption of chemicals causes hazardous substances to be displaced into the solid phase.	[31, 32]
Electrochemical deposition	The approach is based on the oxidation and reduction of heavy metal ions in a cell composed of an anode, a cathode, and an electrolyte cell regulated by current. There are no other reagents utilized. Eco-friendly procedure. Low price.	Non-aqueous solution commercialization problems (poor current efficiency). Heat equilibrium in the cell. Corrosion of cell components.	[32, 33]
Adsorption	It is the transfer of a material from the liquid stage to a solid surface through physical and/or chemical interactions. The operation is simple. Low price. A wide pH range is available.	Selectivity is low. Production of waste products. Chemical-based method.	[34]
Ion exchange	It is a chemical process that can be reversed in which ions are exchanged from wastewater to an equally charged ion which is adhered to a stationary solid surface. Metal ion recovery is selective. Sludge volume is reduced. The separating procedure is quite efficient.	Only the separation of charged ions or molecules with poles has limitations. Ion exchangers are rapidly contaminated, resulting in a loss in exchange capacity. The regeneration fluid, in particular, is inefficient in terms of cost.	[35, 36]
Chemical precipitation	The employment of chemical agents to convert metal ions into insoluble solid particles that may be separated by filtration or sedimentation. Simple procedure. Energy saving.	When the metal concentration is low, the efficiency is reduced. Fine particles are formed, which are sluggish to settle by gravity and expensive to remove by centrifugation. A large amount of chemicals are required, resulting in the development of sludge.	[37]

4. SUGARCANE BAGASSE AS A PRECURSOR FOR NANO CELLULOSES

Several biosorbents have been discovered that have proven effective in eliminating toxic contaminants [47-52]. These are more secure, biodegradable, and renewable [53, 54]. Concerning other bio-polymeric substances, cellulosic adsorbents are the most preferred adsorbents. Aside from its unlimited and surplus supply, it also meets all of the characteristics of a good biosorbent [55-57].

The choice of source substance for the cellulose extraction is critical because it must have high cellulosic content and be readily available and accessible. During the production process, agro-

industries involving sugar, tea, coffee, vegetable oil, cotton, paper, and fruit canning generate a large number of waste products such as rice and corn husk, fruits and vegetable peel-offs, sugarcane bagasse, wheat brans, and so on [58, 59]. These wastes are largely composed of lignocellulosic chemicals, which are a good source of cellulose. Several studies have been undertaken using lignocellulosic wastes to remove heavy metal ions [60].

Sugarcane bagasse (SCB) has long been in high demand among lignocellulosic materials because it is a low-value agricultural residue that is abundant, cheaply available, repeatable, and ecologically friendly [60-64]. It is made up of 40-50% cellulose, 20-25% hemicellulose, 15-20% lignin, and the rest

is extractive [65-68]. Furthermore, because sugarcane bagasse is extensively generated in many countries, it has a substantial commercial benefit as a cellulosic precursor. This reduces the cost of bagasse collection and transportation, which will be more significant for other lignocellulosic leftovers.

The United Nations Food and Agriculture Organization (UNFAO STAT, 2021) conducted a statistical analysis of annual agricultural production. Worldwide, sugarcane production is at 1.9 billion tonnes, equivalent to 21% of the total production of crops. Each ton of sugarcane produces around half as much moisture, and over 250 kg of bagasse and almost 200 kg of straw are used to produce both ethanol and sugar [69, 70]. Sugar businesses mistakenly produce vast amounts of sugar. Bagasse is a waste product in large quantities. It is thrown on the roadway or at a landfill near water bodies. Furthermore, bagasse obtained from domestic residences and small merchants is not properly dumped. If waste is not adequately advocated for, it negatively influences the environment.

As a result, wastes must be properly handled such that unvalued wastes result in developing a specified high-value product. Numerous research studies have been conducted to study cellulose as a biosorbent. However, most results are poor [71-73]. Also, when the size is lowered to the Nanometric scale, the efficiency of heavy metal adsorption rises due to increased particular surface area [74-76]. As a result, Nano cellulose was chosen as a top bio-based choice for the adsorption procedure.

5. PROPERTIES AND POTENTIALS OF NANO CELLULOSE AS A BIOLOGICAL ADSORBENT

The growing global desire for green and sustainable biomaterials with minimal environmental impacts has piqued the curiosity of academics worldwide. Biosorbent selection is critical throughout the process since it must be biocompatible, practical, environmentally safe, and have minimal carbon footprints. The optimum characteristics of an effective biosorbent are depicted in Fig. 1. Aside from that, an effective biosorbent's two most significant qualities are its high sensitivity and adsorption effectiveness for developing water contaminants. The physicochemical properties of the adsorbent, such as solubility, charged surface, chemical formula, reactivity, molecular size, and hydrophobicity,

influence the biosorption process [44, 46, 54]. The presence of surface functional groups on the adsorbent such as sulfonate (SO_3), amine ($-\text{NH}_2$), amide ($-\text{CO}-\text{NH}_2$), carbonyl ($-\text{CHO}$, $-\text{CO}$), phenolic, carboxyl ($-\text{COOH}$), esters ($-\text{COO}$), and alcohols ($-\text{OH}$) that can attract and sequester metal ions in water facilitates this [46, 51-53]. Cellulose is a common polysaccharide that can be obtained from lignocellulosic waste products such as wheat bran, sorghum straw, rice husk, corn stalks, sugarcane bagasse, etc. Cellobiose is generated from D-glucose units composed of a linear chain of condensed β (1-4) connected glycosidic linkages. Each cellobiose unit has three hydroxyl groups that create hydrogen bonds with oxygen atoms in the same or surrounding chains, resulting in high tensile strength [77-79]. Native cellulose has a lower adsorption capability than modified cellulose. Despite numerous alterations, the adsorption capacity remains consistent because it is incompatible with hydrophobic polymer matrices and is prone to aggregation formation during composite processing. It is also short-lived and lacks consistency, restricting its application in polymer reinforcement [80, 81]. However, an elementary cellulose-building unit known as Nano cellulose can overcome this [80]. Recent research has established Nano cellulose as a subsequent-generation material and a promising product for various technological applications.



Fig. 1. The essential characteristics of an effective biosorbents

Heavy metals and chemical dyes have been extensively studied utilizing cellulose-based nanomaterials due to their low cost, non-toxicity, adaptability, and sustainability [82-84]. Also, other forms of nanoparticles are used in various industries, ranging from air and water purifying to environmental prevention to healthcare and

beauty products. These are derived through several natural processes, including photochemical reactions, erupting volcanoes, woodland fires, and erosion, and are synthesized from various chemical compounds. There is little evidence to support the health and safety concerns of conventional nanomaterial exposure, although there are few publications on the adverse effects of exposure through inhalation [85, 86]. Another issue is the accumulation of nanotechnology in the natural environment, namely in water bodies, which accumulates and destroys aquatic life. Nano celluloses, on the other hand, are bio-derived nanomaterials with no environmental implications. Fig. 2 shows the distinct characteristics of Nano celluloses.



Fig. 2. The distinct characteristics of Nano celluloses

Furthermore, Nano celluloses are biodegradable and do not harm the environment. In the paper [87], the adsorption mechanisms of NCs-based adsorbents for wastewater treatment were investigated. Natural fibers (NCs) are rod-like, organized, transparent, biodegradable materials derived from cellulose biomaterial via hydrolysis. These are lightweight natural polymers with low density (1.6 g/cm^3), good tensile strength, crystallinity, and thermal stability, as opposed to the bulk shape [15].

The following points describe these properties:

- Linkages that allow for simple functionalization among various functional groups. Etherification, oxidation carboxylation, esterification, surface hydrophobization by silylation or acylation, cationic surface functionalization, polymer grafting, and other methods are included [74, 88, 89]. It aids in binding contaminant ions (adsorbate) to the adsorbent's adsorptive sites through various chemical

reactions, including chelation, complexation, coordination, hydrogen bonding, and so on [14, 15, 90].

- High particular surface area: Because of their Nanometric scale, cellulose nanoparticles have a high specific surface area, typically greater than $30 \text{ m}^2/\text{g}$ depending on the type of Nano cellulose [91-94], whereas native cellulose has only $2 \text{ m}^2/\text{g}$ [95]. Because of the enormous surface area of Nano celluloses, there are more contact places between adjacent fibers, making the product rigid and ultra-superior.
- Aspect ratio (length/diameter): The percolated Nano crystalline celluloses (CNCs) and entangled Nano-fibrillated celluloses (CNFs) network robustly bound by hydrogen bond show a high aspect ratio of Nano celluloses [15, 16]. It improves the adsorbent's mechanical strength. When seen under scanning electron microscopy, Nano cellulose synthesized by a succession of alkali and acid treatment procedures had a more significant aspect ratio [96].
- CNCs are high-grade materials with substantial mechanical stiffness, with Young's modulus of roughly 130 GPa , significantly greater than glass fibers [79, 97, 98]. Aside from that, the particular Young's modulus of the Nano cellulose is 65 J/g , which is greater than steel, which is approximately 25 Jg^{-1} [97].
- Superior crystallinity, NCs have a crystallinity index ranging from 60 to 80%, which enhances resistivity and decreases solubility even in highly polar liquids [99, 100].
- Biological compatibility and biodegradability: NCs are renewable polymeric nanomaterials that do not alter the adsorption process in any way [83].

6. TYPES OF NANO CELLULOSE AND EXTRACTION METHODS

Nano cellulose's superior qualities make it a viable adsorbent in removing heavy metal ions from wastewater. Nano cellulose obtained from agro-industrial waste sources not only aids in heavy metal cleanup but also promotes the improvement of the environment. Nano celluloses are classified into three types:

- Bacterial Nano cellulose (BNC).
- Nano crystalline cellulose (CNC).

- Nano-fibrillated cellulose (CNF). Given their various biomass origins and extraction procedures, all of these types have comparable chemical compositions but vary in particle size, crystallinity, and shape [101-103]. CNCs and CNFs are obtained from mineral plants, animals, timber, or agricultural wastes, whereas BNCs are derived from microorganisms such as *Gluconacetobacter hansenii*, *Acetobacter xylinum*, *Gluconacetobacter xylinus* (BC), *Sarcina ventriculi* [104].

CNCs are needle-shaped crystallized fibrils 5-10 nm in diameter and 150-300 nm in length. These are a combination of amorphous and crystalline domains. Acid hydrolysis of bleached cellulose fibers is used to break down amorphous cellulose (in particular) into the suspension and liberate crystalline regions [105-107]. Acid hydrolysis is the most often used approach for isolating CNCs because the tightly packed cellulose reduces the access of the crystalline areas, rendering them resistant to acid assault [108]. CNFs are entangled cellulose nanofibers with lateral dimensions ranging from 10-100 nm. The extraction method includes high-pressure homogenization, microfluidization, and high-intensity mechanical grinding with a Masuko grinder.

CNFs have a greater surface area due to their long-chain structure and an abundance of hydroxyl groups on their surface [109]. Bacterial celluloses are discharged as exopolysaccharides by bacteria grown in aqueous nutrition media. It is pure since it contains no lignin or hemicellulosic material [104, 110]. BCs have a high molecular weight, crystallinity index, and excellent mechanical stability [110, 111]. Numerous techniques for extracting Nano cellulose (CNCs and CNFs) have been reported to improve its utilization in various plausible applications [112-114]. The diverse extraction procedures affect the kinds, qualities, and morphological aspects of NCs [103]. The extraction technique, as depicted in the diagram, consists of two significant steps:

- Removal of non-cellulosic components such as lignin, hemicellulose, and other polymers using various pretreatment methods--pretreatments can be chemical, physical, or physicochemical.
- Separation of the Nano cellulose from cellulosic fibrils using various extraction procedures. Mechanical, chemical, and biological isolation methods are commonly

used. These pretreatment and isolation procedures for Nano celluloses should be regulated under controlled conditions. Aside from enhancing NC production, it will also aid in getting the desired size and characteristics. Fig. 3 illustrates methods for lignocellulosic biomass synthesis of Nano cellulose.



Fig. 3. Nano cellulose synthesis and pretreatment procedures

6.1 Methods of Pretreatment of Biomass (Lignicellulose Fractionation)

The pretreatment method tries to degrade resistant lignocellulosic biomass to its constituents, lignin, cellulose, and hemicellulose. This stage entails removing non-cellulosic components through various pretreatment methods. This is the first and most important stage in the isolation of Nano celluloses because the compound should be free of non-cellulosic components. Depolymerization of lignocellulosic biomass is accomplished by solubilizing lignin and hemicellulose, followed by bleaching using oxidizing chemicals, which include sodium hypochlorite.

6.1.1 Pretreatment with Acid

The use of concentrated or dilute acids involving sulphuric acid, hydrochloric acid, nitric acid, phosphoric acid, and others to disturb the lignocellulosic components is the most sought-after method for lignocellulose fractionation among developed pretreatment procedures. However, the hydrolysis of polymer utilizing concentrated acid is repulsive because it can result in the development of hydroxymethylfurfural (HMF) and furfural inhibitors, which can be detrimental to subsequent extraction procedures.

Furthermore, corrosion issues and acid recovery constitute a hindrance. Thus, dilute acid is

used to minimize the efficacy of these inhibitors and solve other issues [115]. Dilute sulphuric acid is extensively used for lignocellulosic biomass pretreatment. Pretreatment is accomplished by mixing the biomass and the dilute acid solution simultaneously at a moderate temperature using ordinary heating. This affects the glycosidic bond in hemicelluloses, the lignin bond, and the hemicellulose-lignin bond while increasing biomass porosity. In the paper [116], a dilute solution of sulfuric acid was used to pretreat the bagasse, and the results showed that the bagasse was degraded (80-98% of the hemicellulose was hydrolyzed). Citric acid was used as a homogeneous catalyst for the breakdown of sugarcane bagasse [117]. Citric acid is useful since it is a mild, renewable organic acid.

6.1.2 Pretreatment with Alkali

Alkali pretreatment uses alkaline solutions, including calcium hydroxide, ammonia, or sodium hydroxide, to degrade the structure of lignocelluloses into their constituents. Compared to other treatment processes, alkali pretreatment is performed at moderate conditions, typically lower temperatures and pressures [108, 118]. However, treatment time is measured in hours or days rather than minutes or seconds [119]. Alkali treatment is an environmentally friendly method that uses less energy and does not cause corrosion. The alkaline pretreatment method causes pulp fiber expansion due to the uptake of water, which improves defibrillation inside the fibers. As a result, it lowers hydrogen bonding and the subsequent procedure of microfibrils to microfibrils while consuming less energy [108, 120].

The disintegration of lignin and hemicellulose, as well as the breaking of ester linkages, occur. A shift follows saponification and the redemption reaction in each component's degree of polymerization, surface area, porosity, and crystallinity [108, 121]. It is an efficient treatment, particularly for agricultural leftovers. Alkali pretreatment is less successful than acid or hydrothermal treatments because it primarily increases lignin solubilization, with little effect on cellulose and hemicellulose [115]. When paired with other treatments, alkali treatment is more effective than treatment with alkaline substances alone [122]. In research [123], the alkaline treatment of sugarcane bagasse was combined with the steam explosion method. It increased

delignification by increasing lignin removal and decreasing hemicellulose]. Following alkaline treatment, a bleaching step is required to remove all lignin from the remaining pulp fibers and get cellulose for Nano cellulose manufacturing [124].

The most common bleaching substances are hydrogen peroxide and sodium chlorite with diluted acetic acid. Sodium chlorite in dilute acetic acid enhances cellulose oxidative cleavage or acid hydrolysis, resulting in simple depolymerization. Although the hydrogen peroxide procedure reduces crystallinity, it is the most favored approach for lignin removal because it improves surface area and is free of chlorine-based chemicals, making the process less damaging.

6.1.3 Treatment with Organosolv

Numerous organic or aqueous solvents, such as phenols, alcohols (ethanol, methanol, ethylene glycol, and tetrahydrofurfuryl alcohol), esters amines, ketones (acetone), and others, are used as dissolving agents in organosolv pretreatment to solubilize lignin and some hemicelluloses, with or without the use of a catalyst. However, when no catalyst is used, organosolv pretreatment includes a highly concentrated solvent (almost 60%) and high temperatures (160-220°C); this technique is known as auto-catalyzed organosolv treatment. The underlying benefit of this pretreatment is that it removes about 100% of the lignin content while causing no substantial decrease in celluloses [125]. One disadvantage of this procedure is using solid solvents, which must be completely removed because they are suppressive to subsequent extraction processes. Small-molecule solvents like methanol and ethanol are favored because of their low boiling temperatures and ease of removal [126].

Few research publications advocate using acid-catalyzed organosolv pretreatment, which incorporates an acid catalyst, to improve pretreatment efficiency under less harsh conditions. This method extracts cellulose as a solid, whereas lignin and hemicellulose dissolve in a biological solvent [127]. Research [128] studied the application of a formic acid-catalyzed organosolv method with a low-boiling point acid-solvent system to degrade sugarcane bagasse. This study showed that the elimination of hemicellulose and lignin was improved due to formic acid's increased efficiency and selectivity. Despite its high efficiency in cellulose removal, it is an unsustainable method due to the use and recovery

of organic solvents. Furthermore, it requires much energy and has certain environmental drawbacks [67].

6.1.4 Ionic Fluids

Ionic fluids (IL) are sustainable solvents, entirely ionic (cations and anions) salt-like compounds that stay liquid even at low temperatures. They offer excellent heat stability and perfect non-volatility. Many studies have shown that ILs may effectively extract lignin from lignocellulosic biomass [116, 129]. Several types of ILs have been successfully used in the pretreatment procedures, such as:

- 1, 3-dimethylimidazolium dimethyl phosphate ([Mmim] [DMP]) ($C_7H_{15}N_2O_4P$).
- 1-butyl-3-methylimidazolium bis (trifluoromethane sulfonyl) imide ([Bmim] [NTf2]) ($C_{10}H_{15}F_6N_3O_4S_2$).
- 1-ethyl-3-methylimidazolium acetate ([Emim] [Ac]) ($C_8H_{14}N_2O_2$).
- 1-allyl-3-methylimidazolium chloride ([Amim] [Cl]) ($C_7H_{11}ClN_2$).
- 1-butyl-3-methylimidazolium chloride ([Bmim] [Cl]) ($C_8H_{15}ClN_2$).
- 1-ethyl-3-(hydroxymethyl) pyridine ethyl sulfate ($C_7H_{14}N_2O_4S$).
- 1-ethyl-3-methylimidazolium acetate ([Emim] ($C_8H_{14}N_2O_2$)).

[Ac] is the most often used IL solvent in sugarcane bagasse pretreatment because it is particularly successful in lignocellulose fractionation. Ionic liquids can dissolve up to 60% of the lignin in sugarcane bagasse; however, the main disadvantage is that they reduce the crystallinity of cellulose fibers, resulting in lower cellulose recalcitrance. According to [30], processing of bagasse using an ionic liquid (1-butyl-3-methylimidazolium chloride ([Bmim] Cl)) ($C_7H_{11}ClN_2$) then subjected to high-pressure homogenization in a homogenous media resulted in 90% recovery of Nano cellulose.

6.1.5 Oxidation of TEMPO

Researches [67, 130] has recently demonstrated innovative pretreatment strategies for synthesizing CNFs. TEMPO (2, 2, 6, 6-tetramethylpiperidine-1-oxyl radical ($C_9H_{18}NO_2$)) oxidation stands out as a distinct method among the various chemical processes that incorporate chemicals and require extensive energy input because it enhances the modification of cellulose's

surface charge through the addition of anionic carboxylate groups.

TEMPO is a nitroxyl or nitroxide radical with the formula 2, 2, 6, 6-tetramethylpiperidine-N-oxyl ($C_9H_{18}NO$). It is a stable chemical that is toxic to organic molecules and challenging to break down. In this approach, catalysts such as TEMPO and NaBr can dissolve in the biomass at a higher pH (10, 11), followed by the addition of NaClO as a primary oxidant. The oxidation reaction generates a negative charge (COO⁻) on the surface, boosting Nano fibril repulsion, which is aided by the action of delaminating forces subjected to defibrillation [68, 112]. Used TEMPO-mediated oxidation to produce cellulose nanofibers from SCB without high-energy mechanical treatments [34]. TEMPO oxidation is preferable to acid oxidation because of its high reaction speed, selectivity, and yield. Pretreatment with TEMPO oxidation, in conjunction with other treatments involving ultrasonication and homogenization, aids in forming a highly functionalized structure, resulting in denser carboxylate groups on the particle's surface [51].

6.1.6 Explosion of Steam

Researchers have actively exploited steam explosion in recent years for successful lignin elimination and hemicellulose solubilization from bagasse pulp. The steam explosion involves exposing the biomass to steam that is saturated at high temperature and pressure for a short period, resulting in thermal expansion of the biomass that follows rapid decompression, subsequently decreasing particle size and increasing pore size volume [108].

As a result, it increases the ability of enzymes to enter the cellulose fibrils, disturbing the material and causing delignification and hemicellulose transition due to high temperature [118]. The influence of temperature is more essential than pretreatment time in achieving long-term performance, i.e., optimal depolymerization of LCB biomass. Temperatures as high as 280 °C combined with a short pretreatment time result in maximal cellulose recovery [11, 131]. The steam explosion method isolates cellulose nanofibers off sugarcane bagasse residue at various temperatures and times [132]. To improve the efficacy of this pretreatment procedure, warm water or organic acids are used as catalysts to break down hemicellulose, encouraging the cleavage of glycosidic bonds during stream purification [131]. Because of its

inexpensive cost and great catalytic activity, sulfuric acid is commonly used as a catalyst for steam explosions. Despite being energy-efficient, cost-effective, and environmentally friendly, the steam explosion process can emit harmful compounds.

Using acid catalysts causes acid water autohydrolysis and the production of corrosive chemicals, which depolymerize hemicellulose and partially degrade and solubilize cellulose [133]. A different technique has been devised, notably the hydrothermal technique, which uses liquid hot water. This approach has numerous additional benefits, including a low-cost reactor setup and a lower corrosion potential. It also does not require a catalyst and may remove up to 80% of hemicelluloses [30, 115, 134]. SO₂ and CO₂ gas-phase acid catalysts are also used, and they do not need to be impregnated before the steam explosion process. [135] Conducted comparison research on the pre-treatment procedure, which included supercritical CO₂ explosion or organosolv enhanced by CO₂, with traditional steam explosion and organosolv procedures for treatment. Regarding environmental and energy issues, they discovered that SO₂-catalyzed steam explosions and supercritical carbon dioxide explosions were less financially appealing process designs [135].

6.2 Methods of Extraction

Mechanical, chemical, and enzymatic procedures are required following cellulose recovery to create Nano cellulose from the recovered celluloses. The morphological structure and nanoparticle size correspond to cellulose extraction methods from biomass [15, 101].

6.2.1 Acid Hydrolysis

This process is usually used to create cellulose Nano crystals. The following processes are involved in the creation of CNCs via acid hydrolysis [113]:

- It occurs in a controlled environment, such as acid concentration, agitation speed, temperature, time, and acid-to-cellulose ratio.
- Dilution of the solubilized items, with subsequent washing and subsequent centrifugation of the solid residue.
- Removal of remaining acid by substantial dialysis.

- Mechanical treatment, generally by sonication, to diffuse the nanoparticles and form a uniformly stable suspension.

Many studies have found that CNCs generated from sugarcane bagasse using the acid hydrolysis process have a higher crystallinity index. In research [99], the characteristics of Cellulose Nano crystals (CNCs) were examined after extracting CNCs from sugarcane bagasse using acid-hydrolysis. The results revealed rod-shaped CNCs with a nanometer size range and a high crystallinity index [99]. CNCs derived from plants have a tremendous potential for biological applications.

They are used as nano-reinforcement fillers in bio-nanocomposite composites and as value-added products in commercial applications. Sulphuric acid is a chemical. As may be observed in the literature, the hydrolysis process is commonly employed [93, 100]. The negative charge that accumulates on the particle surface caused by the concentration of sulfate ester groups is one of the essential properties of CNCs derived from sulfuric acid. It increases the aqueous phase stability of nanocrystalline particles [126].

6.2.2 Enzyme Hydrolysis

Although mechanical or acid/alkaline hydrolysis are the most commonly employed procedures, they cannot be called green because they require energy inputs and include acids and alkalis [39]. As a result, an enzymatic hydrolysis approach may be a better option than any chemical catalysis process. Enzymatic hydrolysis is an elaborate process in which celluloses are transformed into NCs using enzymes such as laccase, endoglucanases (endo-1,4- β glucanase), cellulase, and others. Endoglucanases reduce the extent of polymerization by disrupting the cellulose fibers' glycosidic link. Electrostatic interactions hydrolyze the amorphous fraction of the cellulosic component, resulting in a rise in the crystallinity index of nanocellulose [136, 137]. This approach strives for low energy costs, a moderate working environment, and excellent selectivity [138].

The resulting NCs have good thermal endurance and are simpler to functionalize. Because of the intricate mechanism of the enzyme-substrate complex, which includes poorer yield and longer processing time, NC generation via enzymatic hydrolysis has received little attention in the

literature compared to other chemical and mechanical approaches [139]. However, several researchers have countered the process by modifying enzymes with other approaches, including ultrasonic-assisted enzymatic hydrolysis or alternative treatments [140, 141].

6.2.3 High-Pressure Homogenization and Micro Fluidization

More research has been directed toward treatment methods that are simple, efficient, and do not involve many chemicals. High-pressure homogenization (HPH) is an approach that aids in creating CNFs [142, 143].

To disintegrate the amorphous regions and defibrillate the cellulosic fibers, cellulose microfibrils (CMFs) are bombarded through a homogenization chamber under successive applications of high shear pressure (50-2000 MPa). The HPH approach is reported to have good defibrillation efficiency, requires minimal isolation time, and can be scaled up to pilot vary [132]. Furthermore, NFCs isolated via the HPH approach have a huge surface area, a high aspect ratio, and exceptional mechanical properties. Research [143] examined different treatment methods for the synthesis examined different treatment methods for the synthesis of NFCs from sugarcane bagasse,

such as grinding, high-pressure homogenization, and ultrasonication. They concluded that HPH and ultrasonication procedures improved the ratio of sides to sides and size equality of NFCs [143].

Micro fluidization is a capable method that defibrillates or differentiates cellulosic fibers uniformly at low temperatures and high shear rates [112]. In contrast to the homogenization procedure, which uses a constant pressure, micro fluidization is a treatment method that uses a constant shear rate [126]. The cellulosic substrate is transported through a z-shaped channel that sweeps to a significant shear force in this process. The pressure rises to 40,000 psi (276 MPa), causing fiber fibrillation due to strong delaminating forces and collisions against channel walls and colliding surfaces [120]. Micro fluidization has several advantages, including eliminating pretreatment stages, reducing particles with fewer passes, and lowering manufacturing costs. The main drawbacks of adopting HPH and micro fluidization techniques are high energy input and material blockage [16].

Table 3 shows further examples of hydrolysis processes for producing Nano celluloses. Many studies on various Nano cellulose extraction strategies have been proposed, and we have summarized the stages of a few isolation approaches here.

Table 3. The advantages and disadvantages of hydrolysis procedures for the synthesis of Nano cellulose

Methods	Processes	Changes in cellulose microfibrils	Remarks	References
Acid hydrolysis	Sulfuric acid	The presence of a negative charge on the particle surface is a result of sulphate ester group buildup. The CNC crystallinity index has increased.	Primarily utilized in industrial applications. This procedure mainly entails the creation of cellulose nanocrystals (CNCs).	[93, 100, 144]
Enzyme Hydrolysis	Endoglucanases, Laccases	The CNC crystallinity index has increased.	Low energy expenses, a pleasant operation environment, and excellent selectivity	[138, 141]
High-pressure homogenization and micro fluidization	Cellulose microfibrils (CMFs) are battered via a homogenization chamber under high shear pressure (50-2000 MPa) applications.	CNFs have a huge surface area, a high aspect ratio, and excellent mechanical qualities.	This method involves the creation of cellulose Nano fibrils (CNFs). Includes a commercial advantage because this technique may be scaled up.	[132, 143-146]

7. HEAVY METAL ADSORPTION USING NANO CELLULOSE / MODIFYING NANO CELLULOSE

Several investigations have documented the removal of heavy metal contaminants from water using cellulose extracted from agricultural waste residues. However, a few studies have used Nano cellulose produced from SCB as a bio-sorbent. Reducing cellulose to Nanometric dimensions increases adsorption capacity by exposing active sites that bind to contaminant ions [57].

A large number of functional groups, larger surface area, and better reinforcing capabilities are just a few of the essential qualities of NCs as adsorbents [15, 75]. Because of the accessible active sites that interact with the metals, CNFs have an improved capacity for adsorption than cellulose fibrils (CFs) [147]. This is owing to the increased surface area of celluloses in Nano form and the many groups of functions on the surface of CNFs that aid in creating ionic active sites. Optimizing aspects such as reaction circumstances, contact time, pH, temperature, the initial concentration of heavy metal ions, and ionic nature determines proper biosorption performance - adsorbate and biosorbent, for example [148]. However, numerous investigations have indicated that Nano cellulose has a high adsorption capability. Two types of modification approaches are investigated to develop a good NC-based biosorbent. The first is grafting, using monomers to bind various functional molecules to the backbone of Nano cellulose.

The main chain of the cellulose backbone is covalently linked to the side chain monomer in this method to generate a grafted copolymer [88, 97]. The second way is to change the cellulose structure by adding a functional group. It is a direct modification technique in which Nano cellulose hydroxyl groups are connected to functional groups via a range of physicochemical processes such as oxidation-reduction, esterification, halogenation, sulphonation, mercerization, cationization, and so on [149]. Nanocellulose-based composite aerogels based on bio-based polymers may be further designed to improve adsorption capacity for heavy metal ion removal from contaminated water. Incorporating substantial functional compounds from Nano cellulose and surface charges from Nano cellulose composites makes heavy metal ions accessible. In research [21], Nano-bentonite aerogel was presented with Nano cellulose/Chitosan to

investigate its metal adsorption capacity from simulating wastewater [21].

Aerogels are made through freeze-drying, which results in a sponge-like light structure. It is often made up of percolated cellulose nanocrystals (CNCs) and entangled cellulose nanofibers (CNFs) linked together by hydrogen bonding, which increases the mechanical strength of aerogel nanocomposites [75, 106]. The pollutants from aqueous solutions must be removed. Unlike chemical adsorbents, NC-based bio-adsorbents degrade naturally. As a result, it can be disposed of in the soil after use [90].

8. RECENT KNOWLEDGE GAPS AND PERSPECTIVES FOR THE FUTURE

Bio sorption offers a far more significant and immediate application in water cleanup than previous approaches. Natural polymers have been extensively evaluated as starting materials for biosorption due to their explicit properties and minimal environmental impact.

Because of their substantial, heavy metal adsorptive potential, agricultural waste residues are a promising choice for use as a biosorbent.

Furthermore, agricultural waste created by food sector enterprises will offer access in tons per day and be provided continuously. Many researchers hope to develop nanomaterials with excellent efficiency, strength, cheap cost, biodegradability, and renewability. Nano celluloses have the benefits mentioned above but are limited in the market economy due to their high production costs. Nano-cellulose extraction necessitates pretreatment before incorporating it into composites, raising production costs and impeding commercialization and industrialization. The materials used in the preceding Nano-cellulose extraction and additional modification processes cited in this work are unequal and varied. As a result, standardizing product value becomes extremely challenging [150-153]. The most difficult task is maximizing the value of production cost and capacity. Furthermore, there are numerous unexplored methods of Nano cellulose functionalization.

Now that Nano cellulose has the unique property of surface functionalization, it may represent an opportunity or a challenge to modify the material, as the goal of the modification should not affect the efficiency, particle structure, surface morphology, crystallinity, and chemical and physical characteristics of the synthesized final

composite. Because Nano-celluloses in their natural state do not conform to greater adsorptive capacities, future research should focus on enhancement tactics that contribute to a more efficient and adaptable composite [21]. Surface functionalization can improve the binding properties of NC-based biosorbents, but this may increase the overall cost of the technique, equating it to the price of commercial adsorbents such as ion exchange resins, molecular sieve carbon, activated alumina, or zeolites, among others [154].

Nonetheless, surface modifications may only sometimes result in improved biosorption due to unsuccessful conformational changes, steric hindrances, and other causes. The absence of feedstock materials, decreased affinity of biosorbent towards particular metals in a metal combination solution, issues with bio sorbent regenerative capacity, and preventing coinciding heavy metal ions on the adsorption process are all factors impeding the industrial application of biosorption techniques. On the contrary, improving bio-sorbent physicochemical qualities through surface modifications, grafting techniques, immobilization procedures, and composite synthesis is limited to the laboratory scale.

To treat complicated industrial wastewater, large-scale development of hybrid technologies such as electrochemical processes, biomimetic membranes, bio-precipitation, and so on, as well as heavy metal removal from solutions, is required. More effort should be put into developing low-cost, strong biosorbents with good selectivity and affinity. For technological transfer, more study should also consolidate their approach to reusing and regenerating biosorbents, which will reduce process costs.

9. CONCLUSION

Native cellulose is less desired in adsorption due to its smaller surface area, smaller pore size, lower surface-to-volume ratio, etc. As a result of its outstanding adsorption properties, the development of Nano cellulose using agricultural leftovers with a significant amount of cellulose has added a new dimension to the science of environmental engineering.

This work provides a better understanding of the exceptional properties of cellulose nanoparticles, extraction methods, and their application in removing pollutants and hefty metals from water. Using agro-wastes as the basis

for production in biosorption is not only a feasible alternative to chemical-based adsorbents, but it also advocates for a toxic-free environment. Using agricultural waste has several advantages, including high regeneration, ease of processing, and high metal recovery from wastewater. Furthermore, continued access and availability to these resources may be advantageous in future work.

The increasing number of scientific investigations on this subject indicates that researchers are increasingly interested in bio-based products that can be appropriately formulated from garbage. Using sugarcane bagasse to make Nano cellulose for heavy metal recovery provides a two-way benefit, namely waste minimization and water treatment. It, therefore, represents an applicable instance of a loss-to-wealth economy.

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