



Synthesis of Nanocellulose from Groundnut Husk in Ionic Liquid Medium and its Characterization

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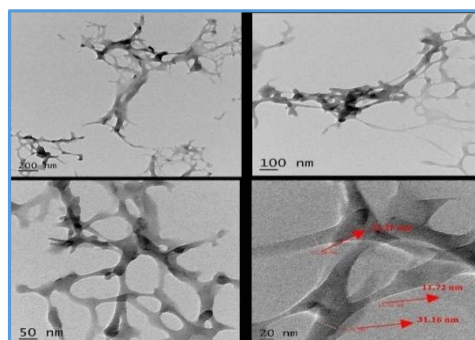
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ABSTRACT

Cellulose creates a lot of interest because it is obtained from bio-renewable sources and possesses the properties such as considerable price, toughness, biocompatibility, and thermal stability. In this study, nanocellulose was extracted from the agricultural biomass Groundnut Husk. The alkali-treated cellulose was then bleached with sodium chlorite in order to expel lignin and hemicellulose. Ionic liquid 1-butyl-3-methylimidazolium chloride ([Bmim] Cl) solvent was used to synthesize the nanocellulose. The functionality of nanocellulose was then characterized by FTIR spectra. X-ray diffraction studies reveal the crystalline or semi-crystalline nature of synthesized nanocellulose. The surface architecture and size of the nanocellulose obtained are represented by SEM monographs. TEM images record the size and exact morphology of synthesized nanocellulose and this shows the size of synthesized is between 11.72 and 31.16 nm. Sonication leads to a substantial reduction of the nanocellulose as depicted by TEM. The thermal stability of the obtained nanocellulose was evidenced using TGA/DTA. The thermal studies insight that the synthesized nanocellulose samples possess appreciable degradation temperatures up to 467.0°C.

Graphical Abstract:



TEM Images of IL-GHNC

Keywords: Groundnut husk, Cellulose, Ionic liquid, Nanocellulose.

INTRODUCTION

Nanoscience and technology are interdisciplinary fields that have evolved rapidly in the last decade, with nanomaterials as cornerstones. Nano-scale materials have at least one nanometer-scale dimension [1]. Nanomaterials are used in many applications due to their optical, magnetic, electrical, and other properties. In recent years, different nanomaterials, including carbon-based nanoparticles, ceramic nanoparticles, and metal nanoparticles, have been manufactured and utilized for various applications. But these materials still need nonrenewable sources of energy, like fossil fuel, to be made. Environmental science research is still looking for materials that are renewable, compatible with living things, and less toxic [2]. Nanoparticle extraction from renewable resources such as lignocellulosic material, microorganisms, and tunicate is of interest to researchers.

Because of its practical applicability in commercial products, countries such as the United States, Europe, Canada, and Australia promote nanocellulose production [3-5]. Nanocellulose is highly dependent on its parent cellulose in terms of structure, size, and function. Recent years have witnessed the expansion of nanotechnology, which is intertwined with multiple other scientific disciplines and affects all forms of life [6]. Nanotechnology is a branch of science that focuses on the creation, modification, and application of materials with dimensions on the nanoscale scale. Nanotechnology is crucial to the study of nanoparticles due to its wide range of applications [7]. Nanoparticles of cellulose and lignocellulose serve a crucial role in nanotechnology. The most popular biological raw materials are cellulosic materials, which can self-assemble into well-defined designs ranging in size from nano to micro. Moreover, cellulose is a versatile raw material that may substitute for a number of non-renewable chemicals [8].

It has been determined that Groundnut husk (GNH) contains 40-45% cellulose, that it is a soft material, and that it is commonly recommended as a superior starting material for nanocellulose manufacturing [9, 10]. It has a shorter duration and 60-100 day growing cycle. This offers it a combination of tremendous strength, stiffness, and thinness that is unparalleled. Numerous plants, such as the husk of coconut rice and maize, hemp, sisal, flax, sugar cane bagasse, etc., have been studied for the extraction of cellulose and Nanocellulose [11]. Because of its high strength and stiffness, it is recommended for the production of Nanocellulose. Considering the availability of raw materials and the viability of the process, GNH is strongly recommended for the synthesis of nanocellulose. Due to the physical features of GNH, the aforementioned raw material is suitable for the manufacture of nanocellulose. Two popular nanocellulose manufacturing procedures have been developed: the chemical method, which involves acid hydrolysis, and the oxidation method with the aid of TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl).

Due to its unique features such as its lightweight, high surface area to volume ratio, and outstanding mechanical capabilities, nanocellulose has attracted a lot of attention from researchers over the past decade [12-15]. Nanocellulose also has properties that can be changed because it has a lot of hydroxyl groups. It is also good for the environment because it is biodegradable and compatible with living organisms [16, 17]. Due to these properties, nanocellulose can be utilized in a wide range of applications, including biomedical applications (including drug delivery, tissue scaffolds, wound dressings, and separation of biomolecules and cells), reinforcement and fillers in composites, optical and electronic devices, and the mobile field [17-23].

Acid hydrolysis, mechanical process, enzymatic hydrolysis, and hydrolysis utilizing ionic liquids are among the ways used to produce nanocellulose from various cellulose sources [24-27].

Ionic liquids (ILs) have piqued the curiosity of researchers not just as promising new solvents for replacing existing solvents in synthesis, but also as new liquid materials. [28, 29]. Recent developments suggest that the new features of certain ILs are of greater interest than their use as green solvents in conventional organic chemistry. The re-discovery of ILs as brand-new materials with numerous remarkable qualities, the majority of which have yet to be uncovered, represents a significant milestone in the field. ILs are distinguished by their compositions, which consist of molecular ions as opposed to molecules found in conventional solvents. In other terms, ILs or molten salts are described in general as liquids composed of ions at room temperature or increased temperatures (below 100°C). In addition to ionic and covalent interactions, ILs have relatively weaker interactions such as H-bondings and π -stacking, which are not usually encountered in conventional solvents [30, 31].

1-butyl-3-methylimidazolium chloride (BmimCl), 1-allyl-3-methylimidazolium chloride (AmimCl), and 1-ethyl-3-methylimidazole acetate are all used ionic solutions for dissolving cellulose [32]. The principal advantage of adopting IL as a pretreatment is the ability to reproduce insolvency recovery with minimal loss. More than 90 percent of BmimCl's activity can be regained by reusing it four times without losing potency [33]. According to previous research, the use of IL as a nanocellulose surface modification medium has applications in nanomedicine and drug delivery [34]. Recent analyses suggest IL as a reaction medium for the homogeneous synthesis of cellulose. According to certain research, homogenous cellulose acetylation can be achieved in AmimCl without the use of catalysts, resulting in cellulose acetates with a broad substitution degree range [35]. The nanocellulose finds valuable applications in the field of Adsorption of dyes, optoelectronics and paper semiconductors, and nanocomposites as strengthening materials [36-39].

This study focuses on the production of nanocellulose from GNH and the evaluation of its characteristics using an ionic liquid, as shown in figure 1. The effects of ionic liquid treatment on dissolution, architecture, thermal strength, crystallinity, and size were investigated using FT-IR, X-ray diffraction, Scanning Electron Microscope, Transmission Electron Microscope Images, Thermogravimetry and Differential thermal analysis. In comparison to conventional research, IL does not add to acid waste and is a more environmentally benign way of isolating nanocellulose [40].

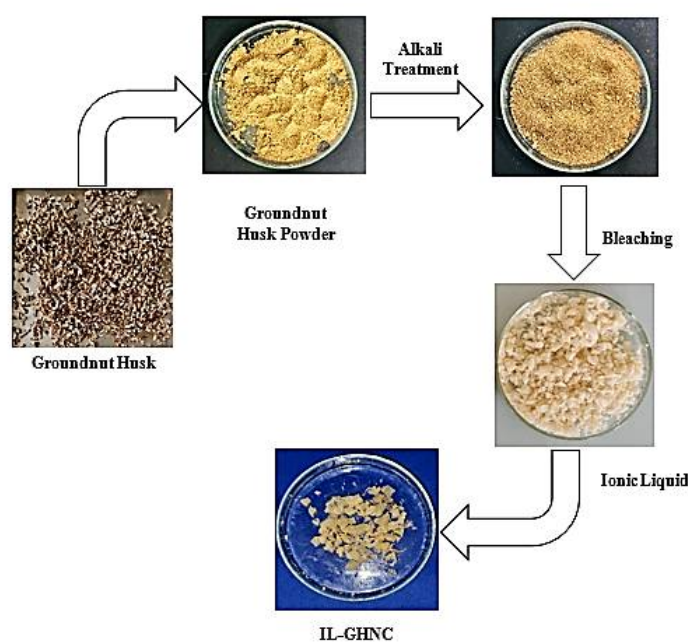


Figure 1. IL Assisted synthesis of Nanocellulose

MATERIALS AND METHODS

The Lignocellulosic material Groundnut husk was collected from farmlands in and around Davanagere, Karnataka, India. The chemicals such as Chlorobutane (C₄H₉C) and 1-methylimidazole (CH₃C₃H₃N₂) were bought from Sigma Aldrich. NaClO₂, NaOH, and CH₃COOH were purchased from Merck and Qualigen Chemicals. All chemicals have a purity of 98 to 99% and are used as such without purification.

Alkali treatment: 10g of finely ground, sieved groundnut husk powder treated with a 5% NaOH solution for 2 h at temperatures ranging from 85 to 100 degrees Celsius to remove hemicellulose. The obtained mass was filtered by repeated rinsing with distilled water until it reached a pH of 7. After that, the product is oven-dried for a day or until it reaches a constant weight. This process makes fibers more susceptible to bleaching, acid hydrolysis, and chemical modification.

Bleaching treatment-Pulping: After being treated with alkali, the sample is bleached to remove any remaining lignin. The alkali-treated was then treated with a sodium chlorite solution containing approximately 5% sodium chlorite. The mixture was refluxed using a thermomagnetic stirrer between 85-110°C for 3-4 h by drop-wise addition of acetic acid to maintain acidic pH. Repetitive washing with distilled water eliminates residual lignin. The synthesized cellulose was filtered by deionized water washing until it reached a neutral pH. The obtained mass was oven-dried for one day until it yields constant weight and stored for further process.

Synthesis of Ionic Liquid- (1-butyl-3-methyl-imidazolium chloride): 60 cm³ of 1-methyl imidazole (0.61 mol) and 80 cm³ of chlorobutane (0.76 mol) were taken in a two-necked round-bottomed flask immersed in an oil bath to ascertain constant temperature. The mixture was kept for overnight refluxing at about 85°C-115°C with a magnetic stirrer until it gives yellow coloured transparent phase. After cooling the reaction mixture, the pure product in the form of an oil or solid was obtained.

Nanocellulose synthesis in 1-butyl-3-methylimidazolium chloride ionic liquid solvent: 10 g of 1-butyl-3-methylimidazolium chloride ionic liquid into a 100 cm³ dry two-necked flask fitted with a water-cooled condenser and carries a calcium guard tube at the outlet of the condenser. It was then placed in an oil bath. 1 g of finely ground and dried Groundnut Husk Cellulose (GHC) was added individually and slowly into a flask containing above the ionic liquid and refluxed on a thermomagnetic stirrer at 115°C to 125°C for 1-2 h. By dissolving cellulose in ionic liquid, a pale yellow solution is obtained. This reaction mixture was then appeased by adding 50 mL of ice-cold distilled water. The resulting nanocellulose precipitate was filtered, washed 5-6 times with cold distillate water followed by sonication for 45 minutes and centrifuged at 2000 rpm for 30 minutes then filtered and dried, stored as IL-GHNC. The yield obtained by this method was found to be 90%.

Characterizations: The FTIR of Cellulose, IL-GHNC was recorded with Thermo Nicolet iS50. After being mixed with KBr powder, the samples were pressed into thin pellets. The sample's wavelength range was measured to be between 4000 and 400 cm⁻¹. The XRD data were recorded using a Bruker D8 Advance Diffractometer. The crystalline and amorphous zone peak heights were measured, and the crystallinity index (CI) was calculated using Scherer's formula with a 0.02 step size. The Jeol 6390LA/OXFORD XMX N instrument used to capture SEM (Scanning Electron Microscopy) images had an acceleration voltage range of 0.5 to 30 kV. A secondary electron (SE) detector was used to acquire the images. TEM images were recorded using a 200 kV, LaB₆ electron gun with a 0.23 nm point resolution and 0.14 nm lattice resolution. TGA (Thermogravimetric Analysis) and DTA were performed using the Perkin Elmer STA 6000 instrument (Differential Thermal Analysis).

RESULTS AND DISCUSSION

FTIR analysis: FTIR was used to explore the chemical functional groups of cellulose. Figure 2 shows the FTIR of IL-GHNC. Due to -OH stretching vibrations induced by hydrogen bonding, the cellulose-I structured absorption peaks between 3331.89 and 3332.35 cm^{-1} . C-H stretching vibrations are represented by the peaks at 2893.39 to 2900.60 cm^{-1} . The H-O-H deformation of absorbed water and conjugated C=O stretch vibrations cause peaks between 1636.04 and 1641.23 cm^{-1} . The absorption region is between 1390 and 1428.27 cm^{-1} was attributed to asymmetric C-H deformation, which changed to a low wave number and weakened due to an interaction with IL at higher temperatures. C-O antisymmetric bridge stretching represents the breakage of H-bonding at O-6. The peak at 1157.25 cm^{-1} represents the breaking of H-bonding at O-6. The skeletal vibration of the C-O-C pyranose ring has peaks ranging from 1029.98 to 1066.28 cm^{-1} . In cellulose, the peaks at 897.09 cm^{-1} correlate to glycosidic linkage. Absorption peaks moving to higher wave numbers showed the transition from cellulose I to cellulose II. The result shows that there was no other reaction in the middle of the dissolving process.

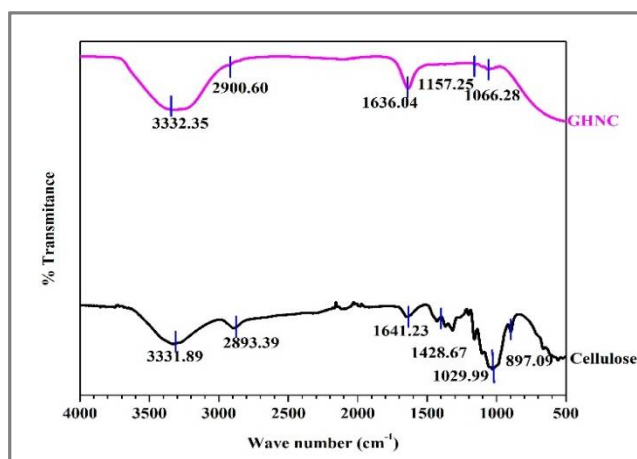


Figure 2. FTIR of Cellulose, IL-RSNC, IL-MWNC, and IL-GHNC

XRD Analysis: The XRD study reveals that nanocellulose is crystalline. Figure 3 illustrates the X-ray diffraction patterns of IL-GHNC treated with ionic liquid and cellulose. These XRD patterns depict semicrystalline materials with an amorphous wide hump and crystalline peaks. IL-GHNC exhibits the

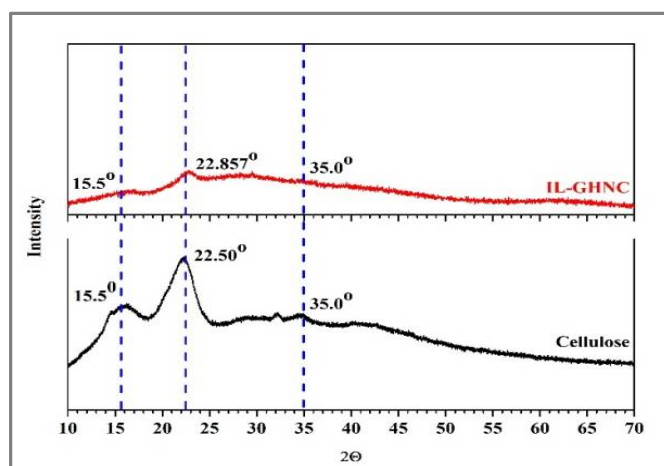


Figure 3. XRD pattern of IL-GHNC, and Cellulose

peaks in the range between 15.50° , 22.857° , and 35.0° , and Cellulose shows the peak at 15.50° , 85° and 35.0° . Due to the disruption of intermolecular and intramolecular hydrogen bonds of cellulose by the ionic liquid and due to sonication, the crystallinity index of the synthesized nanocellulose is reported to be slightly lower than that of cellulose.

SEM Analysis: High-resolution surface imaging is possible using a scanning electron microscope. Using an electron beam, the SEM produces images of the surface. Prepared nanocellulose was found to be fibrous with considerable aggregation, as seen by SEM images. The surface morphology and average size are depicted in the SEM images. They have nanoscale dimensions, including uneven cross-sections, lumens, and a vast number of microscopic microfibrils, as well as various forms and non-uniform surfaces. **Figure 4** shows the morphology of IL-GHNC corresponds to cluster-like nanofibers with smooth surfaces. Here, individualized nanofibers can be obtained with ultrasonic treatment followed by centrifuge. The average size of synthesized nanocellulose is found to be in the range of 200-300 nm.

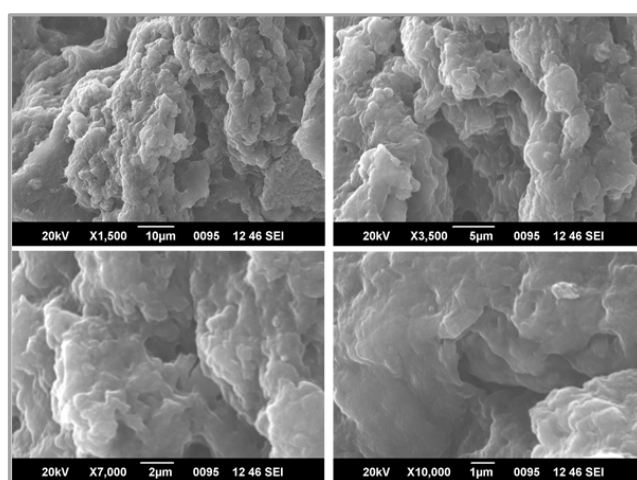


Figure 4. SEM monographs of IL-GHNC

Analysis: The TEM images of IL-GHNC were obtained to examine the internal morphology and structure of the synthesized nano cellulose, as shown in **figure 5**. The TEM image indicates that the nanocellulose that was made is in the form of nanofibers. Nanofibers are well-separated and readily visible. Even the tendency to agglomerate can be seen in all of the images. The IL-GHNC exhibits the formation of nanofibers with an average size between 11.72 and 31.16 nm.

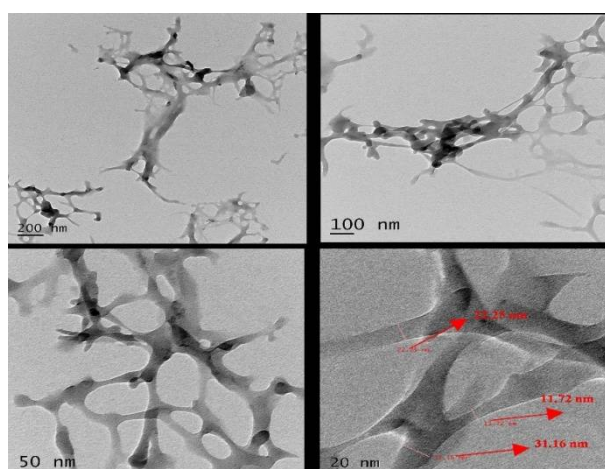


Figure 5. TEM Images of IL-GHNC

This is inferred from the above observation that the synthesized nanocellulose well exhibits nanoscale dimensions. IL-GHNC reveals nanofibers with a size distribution that is less scattered. The preceding investigation demonstrates that the ionic liquid rapidly dissolves cellulose, which influences the nanocellulose structure, shape, and size distribution.

TGA/DTA Analysis: The thermal stability of the IL-GHNC was evaluated by using TGA, and the DTA curves are shown in figure 6 and 7 respectively. The water evaporation in the synthesized nanocellulose is observed between 100°C and 145.9°C. The degradation temperature of IL-GHNC is 327.4°C and 467.0°C respectively. The major weight loss between 250.0°C and 329.5°C regions indicates the depolymerization of hemicellulose and glycosidic linkages and they have broken. The onset degradation temperature is found to be 327.4°C. Here the weight loss occurred in three stages, the first stage was associated with evaporation of water the second and third stages of weight loss occurred in the range 327.4° to 467.0°C. The results depict that the significance of IL-treated is that the synthesized nanocellulose shows superior thermal stability up to 467.0°C.

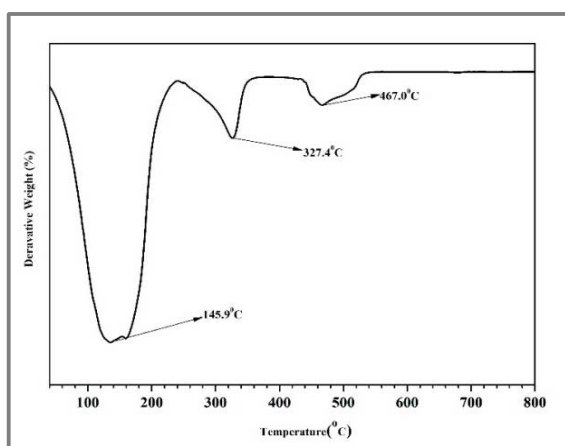


Figure 6. TGA of IL-GHNC.

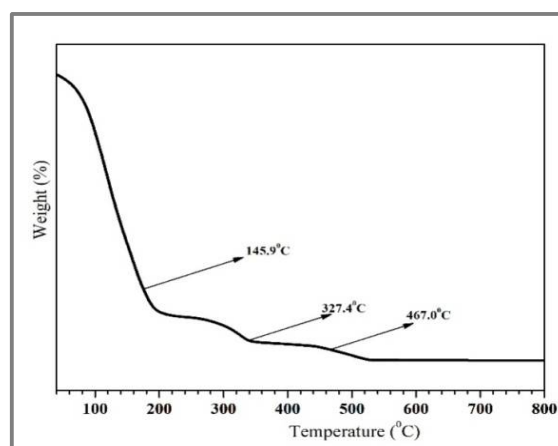


Figure 7. DTG of IL-GHNC.

APPLICATION

This method gives a new insight that upon ultrasonication followed by centrifuge there is an appreciable reduction in the size of nanoparticles which is less than 10 nm. So, that this kind of nanocellulose can be used for photo catalytic dye degradation studies and has not been done before. The Ionic liquid does not offer any chemical wastage

CONCLUSION

In the present study, Groundnut husk lignocellulosic agricultural biomass was utilized to produce nanocellulose from an ionic liquid. The FTIR spectra suggest that IL-GHNC exhibits the same distinctive cellulose peaks. The finding reveals that there was no derivational reaction during the cellulose dissolution process. According to X-ray crystallographic studies, manufactured nanocellulose is composed of semicrystalline materials with amorphous and crystalline peaks. It means that the cellulose's hydrogen bonds were broken, resulting in the disintegration of its crystal structure. Different shapes with a non-uniform surface, regular cross-sections, and a great number of microscopic microfibrils can be seen in SEM images. TEM images show that the cellulose synthesized is in the nanoscale dimension. The cellulose is dissolved by the ionic liquid, a promising green solvent, which impacts the morphology and size distribution of synthesized nanocellulose in nanometer size. According to TGA/DTA analysis, samples exhibit a minor weight loss of roughly 100°C, which is attributed to the evaporation of water bound in the cellulose samples. The strong troughs in the DTA curve reveal that the onset of the breakdown of ionic liquid treated cellulose is

327.4°C. TGA revealed that IL-GHNC treated with ionic liquid has a higher degradation temperature (467.0°C), indicating greater thermal stability. As a result of high thermal stability, this can be used as high temperature resist oven material. Furthermore, this research seeks to demonstrate the remarkable practical application of synthesized nano cellulose from agricultural biomass, which will undoubtedly meet the country's need for solid waste management and reduce chemical waste through a more realistic approach to nanocellulose manufacturing.

REFERENCES

- [1]. L. Brinchi, F. Cotana, E. Fortunati, J. M. Kenny, Production of nanocrystalline cellulose from lignocellulosic biomass: technology and applications, *Carbohydrate polymers*, **2013**, 94(1), 154-169.
- [2]. M. T. Islam, M. M. Alam, A. Patrucco, A. Montarsolo, M. Zoccola, Preparation of nanocellulose, A review, *AATCC Journal of Research*, **2014**, 1(5), 17-23.
- [3]. N. Lin, A. Dufresne, Nanocellulose in biomedicine: Current status and future prospect, *European Polymer Journal*, **2014**, 59, 302-325.
- [4]. S. Das, B. Ghosh, K. Sarkar, Nanocellulose as sustainable biomaterials for drug delivery, *Sensors International*, **2022**, 3, 100135.
- [5]. D. Klemm, F. Kramer, S. Moritz, T. Lindström, M. Ankerfors, D. Gray, A. Dorris, Nanocelluloses: a new family of nature-based materials, *Angewandte Chemie International Edition*, **2011**, 50(24), 5438-5466.
- [6]. S. Baker, S. Satish, Endophytes: toward a vision in synthesis of nanoparticle for future therapeutic agents, *Int. J. Bio-Inorg. Hybd. Nanomat*, **2012**, 1(2), 67-77.
- [7]. K. S. Kavitha, S. Baker, D. Rakshith, H. U. Kavitha, H. C. Yashwantha Rao, B. P. Harini, S. Satish, Plants as green source towards synthesis of nanoparticles, *Int Res J Biol Sci.*, **2013**, 2(6), 66-76.
- [8]. T. H. Wegner, P. E. Jones, Advancing cellulose-based nanotechnology, *Cellulose*, **2006**, 13(2), 115-118.
- [9]. C. Matano, T. M. Meiswinkel, V. F. Wendisch, Amino acid production from rice straw hydrolyzates, In *Wheat and rice in disease prevention and health*, *Academic Press.*, **2014**, 493-505.
- [10]. G. Lamichhane, S. Khadka, A. Acharya, N. Parajuli, Pretreatment of finger millet straw (*Eleusine coracana*) for enzymatic hydrolysis towards bioethanol production, *Biomass Conversion and Biorefinery*, **2021**, 1-15.
- [11]. C. A. D. C. Mendes, F. A. D. O. Adnet, M. C. A. M. Leite, C. G. Furtado, A. M. F. D. Sousa Cellulose, *Chemistry and Technology*, **2015**, 49(9-10) 727-735.
- [12]. I. Kvien, B. S. Tanem, K. Oksman, Characterization of cellulose whiskers and their nanocomposites by atomic force and electron microscopy, *Biomacromolecules*, **2005**, 6(6), 3160-3165.
- [13]. Y. Chen, Q. Wu, B. Huang, M. Huang, X. Ai, Isolation and characteristics of cellulose and nanocellulose from lotus leaf stalk agro-wastes, *BioResources*, **2015**, 10(1), 684-696.
- [14]. H. Tadesse, R. Luque, Advances on biomass pretreatment using ionic liquids: an overview, *Energy and Environmental Science*, **2011**, 4(10), 3913-3929.
- [15]. A. Dufresne, Nanocellulose: a new ageless bionanomaterial, *Materials today*, **2013**, 16(6), 220-227.
- [16]. Z. Lu, L. Fan, H. Zheng, Q. Lu, Y. Liao, B. Huang, Preparation, characterization and optimization of nanocellulose whiskers by simultaneously ultrasonic wave and microwave assisted, *Bioresour. Technol.*, **2013**, 146, 82-88.
- [17]. P. Phanthong, P. Reubroycharoen, X. Hao, G. Xu, A. Abudula, G. Guan Nanocellulose: Extraction and application, *Carbon Resour. Convers.*, **2018**, 11, 32-43.
- [18]. E. Abraham, B. Deepa, L. A. Pothan, M. Jacob, S. Thomas, U. Cvelbar, R. Anandjiwala, Extraction of nanocellulose fibrils from lignocellulosic fibres: A novel approach, *Carbohydrate Polymers*, **2011**, 86(4), 1468-1475.

- [19]. X. Y. Tan, A. H. Sharifah Bee, C. W. Lai. Preparation of high crystallinity cellulose nanocrystals (CNCs) by ionic liquid solvolysis, *Biomass Bioenergy*, **2015**, 81, 584-591.
- [20]. A. Sharma, M. Thakur, M. Bhattacharya, T. Mandal, S. Goswami, Commercial application of cellulose nano-composites—A review, *Biotechnology Reports*, **2019**, 21, e00316.
- [21]. A. H. Bhat, I. Khan, M. A. Usmani, R. Umaphathi, S. M. Al-Kindy, Cellulose an ageless renewable green nanomaterial for medical applications: An overview of ionic liquids in extraction, separation and dissolution of cellulose, *International journal of biological macromolecules*, **2019**, 129, 750-777.
- [22]. R. Curvello, V. S. Raghuvanshi, G. Garnier, Engineering nanocellulose hydrogels for biomedical applications, *Advances in colloid and interface science*, **2019**, 267, 47-61.
- [23]. H. Du, W. Liu, M. Zhang, C. Si, X. Zhang, B. Li, Cellulose nanocrystals and cellulose nanofibrils based hydrogels for biomedical applications, *Carbohydrate polymers*, **2019**, 209, 130-144.
- [24]. Y. Habibi, L. A. Lucia, O. J. Rojas, Cellulose nanocrystals: chemistry, self-assembly, and applications, *Chemical reviews*, **2010**, 110(6), 3479-3500.
- [25]. H. S. Jang, S. K. Jeon, S. H. Nahm, The manufacture of a transparent film heater by spinning multi-walled carbon nanotubes, *Carbon*, **2011**, 49(1), 111-116.
- [26]. S. Cui, S. Zhang, S. Ge, L. Xiong, Q. Sun, Green preparation and characterization of size-controlled nanocrystalline cellulose via ultrasonic-assisted enzymatic hydrolysis. *Industrial Crops and Products*, **2016**, 83, 346-352.
- [27]. S. Mishra, P. S. Kharkar, A. M. Pethe, Biomass and waste materials as potential sources of nanocrystalline cellulose: Comparative review of preparation methods (2016–Till date), *Carbohydrate polymers*, **2019**, 207, 418-427.
- [28]. N. V. Plechkova, K. R. Seddon, Applications of ionic liquids in the chemical industry, *Chemical Society Reviews*, **2008**, 37(1), 123-150.
- [29]. A. Samanta, Solvation dynamics in ionic liquids: what we have learned from the dynamic fluorescence stokes shift studies, *The Journal of Physical Chemistry Letters*, **2010**, 1(10), 1557-1562.
- [30]. S. Saha, H. O. Hamaguchi, Effect of water on the molecular structure and arrangement of nitrile-functionalized ionic liquids, *The Journal of Physical Chemistry B*, **2006**, 110(6), 2777-2781.
- [31]. S. Saha, S. Hayashi, A. Kobayashi, H. O. Hamaguchi, Crystal structure of 1-butyl-3-methylimidazolium chloride. A clue to the elucidation of the ionic liquid structure, *Chemistry letters*, **2003**, 32(8), 740-741.
- [32]. M. Babicka, M. Woźniak, K. Szentner, M. Bartkowiak, B. Peplińska, K. Dwiecki, I. Ratajczak, Nanocellulose production using ionic liquids with enzymatic pretreatment, *Materials*, **2021**, 14(12), 3264.
- [33]. P. Phanthong, S. Karnjanakom, P. Reubroycharoen, X. Hao, A. Abudula, G. A. Guan, facile one-step way for extraction of nanocellulose with high yield by ball milling with ionic liquid, *Cellulose*, **2017**, 24(5), 2083-2093.
- [34]. G. A. S. Haron, H. Mahmood, M. H. Noh, M. Z. Alam, M. Moniruzzaman, Ionic liquids as a sustainable platform for nanocellulose processing from bioresources: Overview and current status. *ACS Sustainable Chemistry & Engineering*, 2021, 9(3), 1008-1034.
- [35]. M. B. Turner, S. K. Spear, J. D. Holbrey, R. D. Rogers, Production of bioactive cellulose films reconstituted from ionic liquids, *Biomacromolecules*, **2004**, 5(4), 1379-1384.
- [36]. K. P. Tiwari, Anand Pandey, Electrical and Optical Properties of Semiconducting Nano Composites, *J. Applicable Chem.*, **2018**, 7(1), 219-223.
- [37]. P. Babji, I. Nageswara Rao, Synthesis and Characterization of Copper and Tin Codoped TiO₂ Nanoparticles, *J. Applicable Chem.*, **2015**, 4 (3), 857-864.
- [38]. H. C Charan Kumar, R. Shilpa, V. Ravi Shankar Rai, Sannaiah Ananda. Synthesis and Characterization of NiO Nanoparticles by Electrochemical Method: Photodegradation Kinetics of Indigo Carmine Dye and Study of Antibacterial Activities of NiO Nanoparticles, *J. Applicable Chem.*, **2019**, 8(2), 622-633.

- [39]. K. R. Raksha, S. Ananda, An investigation on: kinetics of photo catalysis, electrical property and biological activity of electrochemically synthesized ZnS and Ru: ZnS nano photocatalysis. *J. Applicable Chem.*, **2014**, 3, 397-412.
- [40]. L. Feng, Z. L. Chen, Research progress on dissolution and functional modification of cellulose in ionic liquids, *Journal of Molecular Liquids*, **2008**, 142(1-3), 1-5.