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Extraction, Preparation and Characterization: Nanocellullose

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Abstract: ***Nanocellulose the organic nanomaterial is an emerging and promising material with exceptional properties and a broad spectrum of potential applications in numerous fields, i.e. pharmaceutics, biomedical, food ingredients, cosmetic additives, packaging materials, hygiene products, film and absorbance media, electronics, barrier films, membranes, nanocomposites and supercapacitors etc. Due to its unique structural features and remarkable physicochemical properties such as biodegradability, biocompatibility, renewability, low density, optical transparency, adaptable surface chemistry and improved mechanical properties nanocellulose has drawn tremendous attention in academia as well as in industrial sector. The main aim of this article is to provide an overview of recent research in the field of nanocellulose production from different pre treatments and methods.***

Keywords: Nanocellulose, Renewable, Production, Processes

1. Introduction

Cellulosic materials (cellulose, hemicelluloses and lignin) due to their unique characteristics viz. biodegradability and renewability, low cost as well as low carbon dioxide release (Abdul Khalil et al., 2016) have been categorized as sustainable, green and environmental friendly materials (Maleki et al., 2017). Utilization of these cellulosic materials generated from different agricultural wastes to produce nanocellulose is a feasible approach (Elias et al., 2017).

The nanocellulose obtained from such biomaterials are equipped with superior characteristics which are high surface area, nanoscale dimension, high crystallinity, optical properties and stiffness alongwith biodegradability and renewability (Siro 2010; Abraham et al., 2011; Trache et al., 2017) and they also combine significant properties of cellulose such as crystalline structure, hydrophilicity and broad chemical modification (Mahfoudhi & Boufi 2016). Reallocation from cellulose to nanocellulose based materials is a productive method so far (Kalia et al., 2014; Khalil et al., 2014).

Cellulose is the most abundant polymer which is renewable and biodegradable (Jabbar et al., 2017). Cellulose is a linear homopolysaccharide composed of β-D-glucopyranose units linked together by β-1-4-linkages (Kumar et al., 2014). Each β-1-4 glucopyranose bears three hydroxyl groups where the primary OH group is located at the C-6 position and the secondary OH group is located at the C-2 and C-3 position. The secondary and the primary hydroxyl groups of the anhydro-glucose rings are the basis for extensive intra and intermolecular hydrogen bonding (Alemdar & Sain 2008) allowing the formation of highly ordered three-dimensional crystal structures; therefore making cellulose a semi-rigid polymer. As, Cellulose is linked by β (1→4) glycosidic linkage in a long chain therefore its properties are directly related to the chain length (Horn et al., 2012). One of the properties of cellulose is water insoluble, due to its glycosidic linkage between the monomers. Cellulose derived from plants has hemicellulose, lignin, pectin and other substances.

Hemicellulose is a branched amorphous polysaccharide that comprises about 20% of the biomass of most plants. In addition to glucose hemicellulose is derived from several sugars like mannose, galactose, rhamnose, arabinose and especially xylose (Moran et al., 2008). Lignin the most abundant plant-derived amorphous polymer is an organic substance having dendridic network of phenyl propane which bind the cells, fibres and vessels which constitute wood and the lignified elements of plants. After cellulose, it is the most abundant renewable carbon source on earth. Lignin is a complex molecular structure containing cross-linked polymers of phenolic monomers especially p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. Pectin is a heteropolysaccharide of 1-4 linked galacturonic acid with methyl esters of different sugar units (Klemm et al., 1998; Eichhorn et al., 2001).
In plant cell wall structure, crystal and amorphous cellulose structure form nano/micro fibers, which together produce cellulose fiber (Eichhorn et al., 2010). Therefore, cellulose contains highly ordered crystalline alternative with less ordered amorphous segments (Sjostrom E 1993). The amorphous segments can be removed to produce highly crystalline cellulose (Azizi et al., 2005). Cellulose occurs in four major polymeric forms: cellulose I, II, III and IV (Giri & Adhikari 2013). Among them cellulose I is natural cellulose with structures I_α and I_β. Cellulose I_α has a single chain triclinic structure and I_β has two chain monoclinic structure (Kukle et al., 2011). Cellulose in regenerated cellulose fibers is cellulose II that can be formed whenever the lattice of cellulose I is destroyed as on swelling with strong alkali or on dissolution of cellulose (Kamel 2007). The conversion of cellulose I to cellulose II is irreversible, suggesting that cellulose I is metastable and cellulose II is stable. With various chemical treatments it is possible to produce the structures of cellulose III on heating cellulose I and II with ammonia and cellulose IV on heating cellulose III (Park et al., 2010).

2. Overview of cellulosic materials

Cellulose microfibrils are the elementary components of cellulose formed during the biosynthesis (Chirayil et al., 2014). The term microfibril is generally used to describe the 2-10 nm thick fibrous cellulose structures with the length of several microns. Depending on the basis of dimensions, functions and preparation methods nanocellulose can be classified in two categories cellulose nanocrystals (CNC) and cellulose nanofibres (CNFs).

Cellulose nanofibres (Abe et al., 2003) and nanofibrils (Xie & Li 2017 ; Henriksson et al., 2008) are the terms used for microfibrillated cellulose (Siqueria et al., 2010).

Cellulose nanocrystals (Azeredo et al., 2016) are rod-like nanoparticles with limited flexibility as they do not contain amorphous regions (Brinchi et al., 2013). Other names for nanocrystals are nanowhiskers (John & Thomas 2008; Oksman 2006), nanorods (Dujardin et al., 2003) and rod-like nanocystals (Iwamoto et al., 2007). CNCs are the crystalline regions of the CNFs which under controlled conditions can lead to the formation of high-purity crystals (Azizi et al., 2005). Due to this its elastic modulus can be equivalent to the modulus of crystalline cellulose up to 140 GPa (Jonoobi et al., 2015).

Preparation of nanocellulose

Generally, preparation of nanocellulose is carried out in two steps. The first step involves the pretreatment of raw material (depending upon the source) to produce purified individual cellulosic fibres that can be further processed. The second step is the transformation of individual cellulosic fibres into microfibrils, nanofibres or nanocrystals (Garcia et al., 2015). Various methods have been suggested by different researchers to synthesize nanocellulose.

Enzymatic Hydrolysis

To understand the enzymatic hydrolysis of cellulose, most significant is (i) the structural behavior of cellulosic biomass and (ii) biological barriers for enzyme interactions. Enzymes introduced desired functional groups on the surface of nanocelluloses (Karim et al., 2015). Enzymes are employed to alter hemicelluloses and lignin contents while maintaining cellulose portion (Janardan & Sain 2006 ; Khalil et al., 2014). Enzymatic treatments facilitate the fabrication of cellulose nanocrystals with reduced energy consumption. The enzymes involved are cellobiohydrolases and endoglucanases. Cellobiohydrolases (the main enzymes) are able to attack highly crystalline cellulose and endoglucanases require some disorder in the structure to perform cellulose degradation. These enzymes show strong synergistic effects (Henriksson et al., 2007).

Oxidation

The TEMPO-mediated (2,2,6,6-tetramethyl-1-piperidine-1-oxyl) oxidation is the most widely used method to isolate nanofibres or nanocrystals. The carboxylate and aldehyde functional groups are put into the C_6 glucose unit of cellulose structure under moderate conditions (Iwamoto et al., 2011). A primary oxidizing agent (sodium hypochlorite) and an additional catalyst sodium bromide or an iodine are employed at pH 9-11(Saito et al., 2009).

Electrospinning

Electrospinning is simple, efficient and cost effective processes which formulate nanofibres from non-woven to the order of few nanometers with advanced mechanical properties, large surface area and ease of functionalisation. UV irradiation in concentrated crystalline liquid solutions containing isotropic solvents like acetone, ethanol, methanol, dimethylformamide or water are generally used (Christoforou 2010). Over the past few years, there has been a tremendous intensification of the research activities to explore electrospinning for nanofibers formation involving a large variety of materials. It operates on the principle that under the action of a high electric field a solution is extruded electrospun. Once the voltage is sufficiently high, a charged stream of matter is ejected following a rather complicated loop and 3D spiral deployment trajectory (Rebouillat & Pla 2013). During this, the solvent evaporates leaving behind randomly oriented nanofibers accumulating on the collector (Fang et al., 1997). For visualizing morphological features of the electrospun nanofibers different parameters such as electric field strength, tip-to-collector distance, solution feed rate and composition are used. To produce cellulose nanofibres by electrospinning technique have been used by (Frey & Joo., 2005) where cellulose fibers have been dissolved in solvents, such as ethylene diamine, with a salt selected from the group consisting of potassium thiocyanate, potassium iodide and mixtures thereof, the salt being present at their saturation points.

Mechanical Processes

Different mechanical processes have been used to produce microfibrillated cellulose and cellulose nanofibres. These include refining followed by high-pressure homogenization, cryocrushing, grinding, and high intensity ultrasonic treatments.

Refining and High pressure homogenization

Refining is a pretreatment before high pressure homogenization (Stenstad et al., 2008). For refining to be carried out, a disk refiner is generally used in which a diluted cellulosic suspension is forced through a gap between a rotor and stator. The disks surfaces are grooved and fitted with bars to subject the fibers to repeated cyclic frictional stresses. This mechanical treatment leads to irreversible alterations in the fibres, increasing their bonding potential by modification in their morphology and size (Herrick et al., 1983).

High pressure homogenization is a process in which a large pressure drop facilitated the micro fibrillation (Davoudpour et al., 2015; Khalil et al., 2014). The dilute cellulose slurry previously treated by refining are pumped at high pressure into a vessel through very small nozzle

and fed through a spring loaded valve. As this valve opens and closes at a fast rate, the fibers are exposed to a large pressure drop with shear and impact forces which alongwith high velocity reduce the size of fibers to nanoscale (Frone, et al., 2011). High pressure homogenization is among the proficient methods of refining cellulosic fibres because of its high efficiency, simplicity and without organic solvents requirement (Keeratiurai & Corredig, 2009).

Cryocrushing

Cryocrushing is an alternative method for producing cellulose nanofibers (Chakraborty et al., 2005). In the course of action water swollen cellulosic fibers are immerse in liquid nitrogen followed by crushing with mortar and pestle (Frone et al., 2011). Under high impact and shear forces the ice crystals exert pressure on the cell wall leading to rupture of cell wall of frozen cellulosic fibres and thus, liberating nanofibers (Siró & Plackett, 2010; Wang & Sain 2007a; Wang & Sain 2007b).

Grinding

Grinding is another approach to liberate cellulose nanofibers by rupturing cellulose. The cellulose suspension is passed between the static and the rotating grinding stones of the grinder set at ~1500 rpm. In this process, the cell wall structure and hydrogen bond is broken down by the shearing forces generating individual cellulose nanofibers (Abe et al., 2007; Iwamoto et al., 2005; Siró & Plackett, 2010).

High intensity ultrasonication

High intensity ultrasonication is a mechanical process which promotes the isolation of cellulose nanofibres with oscillating power and hydrodynamic forces of ultrasound (Cheng et al., 2009). In this process, cavitation leads to a powerful mechanical oscillating power and when molecules absorb ultrasonic energy (Chen et al., 2013) high intensive wave aids in formation and expansion of microscopic gas bubbles.

Microfluidization

Microfluidization is the process similar to high pressure homogenization which can be used to produce cellulose nanofibres. Microfluidizer includes intensifier pump to increase the pressure and interaction chamber to defibrillate the fibers using shear and impact forces against colliding streams and the channel walls (Ferrer et al., 2012). Aspect ratio of cellulose fibers increases when they pass through the microfludizer (Lee et al. (2009).

Steam explosion

Steam explosion is a thermo mechanical processe. Due to the high pressure, steam penetrates into the cellulose fibers and shear force is generated by sudden pressure release which hydrolyze the glycosidic and hydrogen bonds resulting in the formation of cellulose nanofibers (Giri & Adhikari, 2013: Cherian et al., 2010).

Chemical Treatment

Chemical treatment is the most efficient and easy method to isolate cellulose nanofibres. Alkali treatment, bleaching treatment and acid hydrolysis together disintegrate the cellulosic fibres to nanofibres. Different alkalis remove hemicelluloses and lignin thereby purifies the cellulose. Bleaching agents remove remaining hemicellulose and lignin (Kaur et al., 2017). Oxidative fragmentation of lignin takes place and some of the lignin form lignin chloride which is easily dissolvable. Highest percent of cellulose content is observed after bleaching. Acid hydrolysis induces individualization of microfibrils from the cell wall structure. With H_2SO_4, surface charged sulfates are formed, promoting the dispersion of nanocrystals (Revol et al., 1992). With H_2SO_4 aggregation of Cellulose nanofibres has been observed. Combination of H_2SO_4 and HCl during hydrolysis generates spherical nanoparticles due to less sulfate groups on their surface with improved thermal stability (Wang et al., 2007).

Characterization

Conventionally, microscopic methods have been used as advantageous tool to analyze the appearance, morphology, sizes and shapes of microfibres and cellulose nanofibres produced from various cellulosic raw materials (Kangas et al., 2014; Abe et al. 2007; Chakraborthy et al. 2005; Cheng et al. 2010; Jonoobi et al. 2009; Kaushik, Singh 2011; Pääkkö et al. 2007; Wang et al. 2012). The different techniques are (i) Scanning electron Microscopy (SEM), (ii) Field emission scanning electron Microscopy (FESEM), (iii)Transmission electron Microscopy (TEM), (iv) Atomic Force Microscopy (AFM), (v)X-ray diffraction (XRD), (vi) Fourier transform infrared (FTIR) spectroscopy, (vii)Particle size measurement (DLS) and (viii)Thermogravimetric analysis (TGA).

The surface morphology of cellulose nanofibres is generally analysed by scanning electron microscopy (SEM). In this technique samples are placed on the aluminium stub and incubated in the oven at 60^0C. Then the samples are coated with gold using a vaccum sputter coater. The accelerating voltage taken as 15kV. FE-SEM imaging can also be achieved by using low acceleration voltages without the conductive layer. The morphology of the regenerated cellulose nanofibres can also be carried out by transmission electron microscopy (TEM). A drop of a dilute aqueous suspension (0.1 wt%) is deposited on the surface of a copper grid coated with a thin carbon film. The accelerating voltage to be kept as 100-120 kV. Surface topography and morphology can also be determined by atomic force microscopy (AFM) using tip broadening effect where usually a drop of dilute suspension is placed on a mica surface and left to dry. The structural properties of the cellulose nanofibres like size and crystallinity index can be characterized using x-ray diffraction. The thermal stability of cellulose nanofibres can be determined by thermogravimetric analysis. Decomposition of fibres occurs at different temperatures. Fourier transform infrared spectroscopy measurements are carried out with finely ground sample is mixed with potassium bromide, KBr which is then compressed to

pellet form and analysis is carried out within the wave number range of 400-4000cm^{-1} to access the chemical changes occur during the process (Kangas et al., 2014; Abe et al. 2007; Henriksson et al. 2008; Johansson et al. 2011; Pääkkö et al. 2007).

3. Conclusion

Nanotechnology has the ability to manipulate and acquaintance at microscopic stratum. Bio-based nanocellulose has been widely considered as one of the most promising nanomaterials, which has phenomenal applications in industrial sector and in different fields. Nanocellulose is an environmental-friendly material that could serve as an indispensable renewable resource for progressive revitalization which has no harmful effects on human's health and on our environment. This review has provided an explanation of several aspects that are important for understanding of various nanomaterials.

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