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AN OVERVIEW ON RECENT ACCOMPLISHMENT OF NANOCELLULOSE AS SENSING MATERIAL IN VARIOUS APPLICATIONS

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ABSTRACT

Polymers made from plant and animal sources are attracting a lot of attention in the research area due to their cost-effectiveness, renewable, and sustainable characteristics. Their abundance in nature, nano-dimension size, large surface area, stability, and biocompatibility make nanocellulose (NC) one such polymer that has piqued researchers' attention. By 2025, the nanocellulose market is expected to be worth USD 783 million. Therefore, fast advances in nanotechnology and material sciences have aided nanocellulose research, culminating in their development as potential biomaterials. Nanocellulose has been studied extensively in numerous sectors such as healthcare, household product, electronic, renewable energy, and food agricultural. Nanocellulose applications in the sensing sector have gotten a lot of interest recently. The latest advancements in nanocellulose, with a particular emphasis on their performance as sensing material for various applications, are presented and explored in depth here.

1.0 INTRODUCTION

Cellulose, the most abundant polymer in the world, has excellent properties such as non-toxic, biocompatible, cost-effective, biodegradable, renewable and sustainable polymer. Nanocellulose (NC) is a cellulosic material with one dimension in a nanometer range possessing great properties including high surface area, high mechanical strength and stability [1]. Due to their amazing characteristics, nanocellulose become promising material for various applications including automotive application [2], sensor field [3], adsorbent [4-5], biomedical industry [6], food packaging [7], military textiles [8] and many more. Nanocellulose is often divided into two groups: nanostructured materials (cellulose microcrystals and cellulose microfibrils) and nanofibers (cellulose nanofibrils, cellulose nanocrystals, and bacterial cellulose), based on their isolation methods [9-10].

Cellulose nanocrystals (CNCs) are cylindrical, elongated, less flexible, and rod-like nanoparticles with a width of 4 – 70 nm, 100 – 6,000 nm long, and a crystallinity index of 54 – 88 %, which are often generated by acid hydrolysis. In comparison to CNCs, nano fibrillated cellulose (CNF), which is usually produced by mechanical treatment, has an entangled network structure with flexible, longer and wider nanofibers (20 –100 nm in width and >10,000 nm in length) and lesser crystallinity [11]. Chemical, mechanical, and enzymatic treatments, as well as a combination of these, have been used to produce CNF from lignocellulosic biomass, as recently detailed in detail elsewhere [9, 12-13]. Bacterial nanocellulose, also known as microbial nanocellulose, on the other hand, is regarded as a promising and cost-effective

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natural nanomaterial for biomedical applications. It is made up of pure, ultrafine, ribbon-shaped nanofibers with diameters of 20 – 100 nm and lengths of micrometers that entangle to form a three-dimensional network. This type of nanocellulose is generally synthesized from bacteria, however, its production is expensive due to the high cost of synthetic media [14].

Nanocellulose is hydrophilic in nature due to the abundance of hydroxyl groups on its surface, which allows for easy surface modification by chemical, physical, and biological means. Surface functionalization of nanocellulose can be done during the preparation stage or after it has been synthesized. These modifications result in the development of beneficial characteristics, which in turn improves their effectiveness for a specific application. Figure 1 shows the type of modification that can be conducted on nanocellulose.

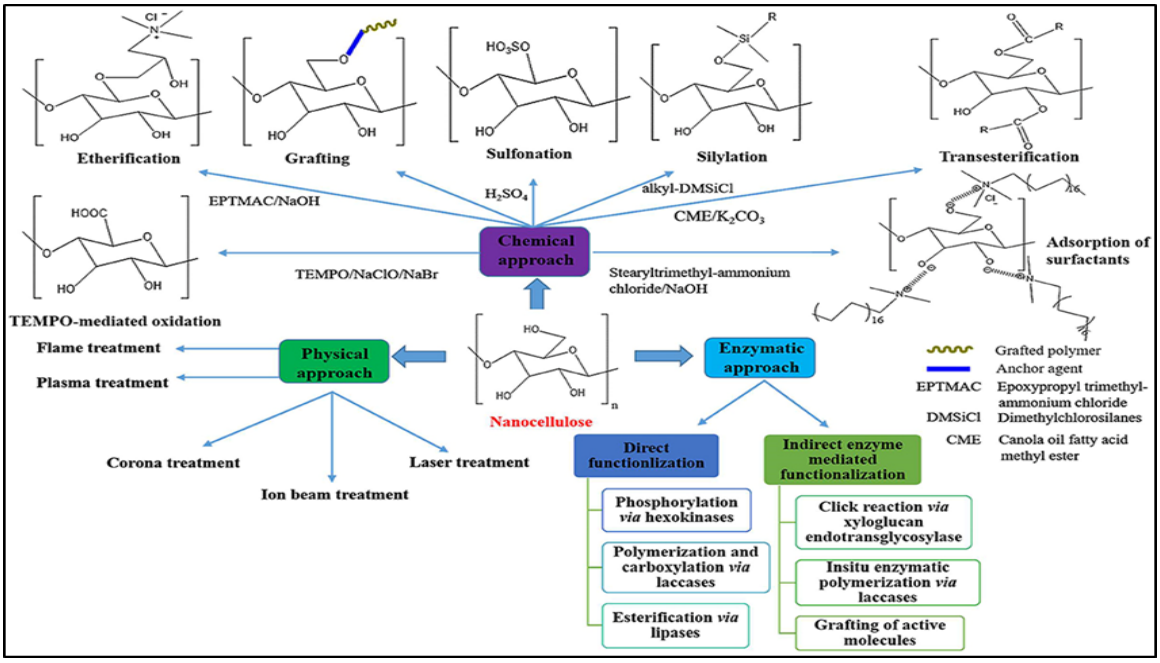

Figure 1. Surface modification of nanocellulose [11]

The hydroxyl surface groups allow huge possibilities for chemical reaction which are useful for improving sensitivity of sensor, make the nanocellulose suitable as sensing material. Moreover, functionalization of nanocellulose with metal oxides, nanoparticles (carbon nanotubes, carbon nanofibers or metal nanoparticles) and other polymer material is a key to enhance the capability of nanocellulose as sensing material. Several studies have shown that a nanocellulose-based sensor can detect at low concentrations with excellent response. Other nanomaterials, such as carbon nanotubes[15–18], carbon nanofibers [19-20], and metal nanoparticles [21-23], have been shown to detect various analytes in recent studies and hence, these current literatures reporting on the development of nanocellulose as a sensing material are highlighted in the next subchapter. Numerous approaches on nanocellulose-based composite for many types of sensors including biosensor, gas sensor, calorimetric sensor, strain sensor, electrochemical sensor as well as capacitive sensor were discussed.

2.0 CURRENT REVIEW ON NANOCELLULOSE AS SENSING MATERIAL

The development of nanocellulose as electrochemical sensor for detection of gas has been reported in various studies. Zhang et al. (2017) developed an efficient and green solid electrolyte using nanocellulose co-functionalized with graphene oxide (GO). In application of an alcohol fuel cell sensor, the electrolyte was tested for alcohol detection. It demonstrated a good response to different alcohol concentrations, as well as high linearity and sensitivity. It also showed a low detection limit (25 ppm), promoting strong candidates as solid electrolytes with ion conduction [24]. Sobhan et al. (2019) explores the influence of different compositions between cellulose nanofibers and activated carbon films in the context of smart food packaging. The mechanical, thermal, and electrochemical properties of prepared films were observed. According to the findings, nanocellulose-activated carbon films with a greater CNF concentration exhibited a dense and complex structure. Thermal stability tests also revealed that the

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films were stable up to 270 ◦ C. Despite the fact that the films had strong mechanical characteristics that made them acceptable for rough use, they had low electrical properties that made them unsuitable for biosensing films [25].

Shalauddin et al. (2019) fabricated electrochemical sensor for detection of diclofenac sodium (DCF), a non-steroidal anti-inflammatory drug (NSAID) and widely used as an electroactive painkiller in medical field using nanocellulose functionalized with carboxylated carbon nanotubes. The presence of hydroxyl group in nanocellulose provides more binding sites with analytes, and its integration with carbon nanotubes gives high surface area, great mechanical strength as well as good electrical conductivity. Based on data collected, the modified electrode showed a significant improvement in the anodic peak current (41.6 mA) for 50 mM DCF at 0.677 V peak potential. The newly manufactured electrode displayed two linear dynamic ranges from 0.05 to 1.00 mM and 2-250 mM DCF with low detection limit of 0.012 mM, indicating that it has good potential to determine DCF from commercial tablets, ampoules (pharmaceutical preparations), and clinical preparations. (human blood serum and urine sample) with good recoveries [26]. Ortolani et al. (2019) investigated electrochemical behaviour of nanocellulose (NC) and single walled carbon nanohorns (SWCNH) in detection of guanine and adenine. NC and SWCNH owing excellent characteristics as electrodes such as high surface area, good conductivity, large porosity, and great chemical stability. The electrochemical behaviour of NC and SWCNH were studied using cyclic and linear sweep voltammetry, which showed excellent result with limit of detection of 1.7×10^{-7} mol/L and 1.4×10^{-6} mol/L for guanine and adenine, respectively [27]. Wu et al. (2020) demonstrated hydrogel conductive as flexible and capacitive sensor using 2,2,6,6-tetramethylpiperidine-N-oxyl radical (TEMPO)-nanocellulose (TC) and carbon nanotubes into pigskin matrix (PS). The presence of TC in carbon nanotubes allows them to distribute uniformly in the PS matrix, resulting in a robust and well-conductive network. The reaction of a conductive hydrogel to cyclic tensile and pressing pressures was examined, and it demonstrated an excellent response with consistent and reproducible resistance change signals. Furthermore, the produced hydrogel has a specific capacitance of ~65 F/g and retains 60 % of its capacitance after 2000 charge-discharge cycles. This research encourages new ways to using animal skin in the fabrication of capacitance sensors that are both efficient and high-performing [28].

Wang et al. (2021) determined glucose concentration using nanocellulose/poly (vinyl alcohol)/carbon dot (NPC) multifunctional hydrogel. The hydrogel was fabricated via one-step *in-situ* hydrothermal method. At room temperature (25 ◦C), the hydrogel self-healed and showed double emission fluorescence as well as good mechanical strength (tensile strength of up to 2.98 MPa). Based on the data obtained, a NPC hydrogel-based capacitive sensor demonstrated exceptional linear capacitance responsiveness to glucose concentration, strain and pressure, as well as real-time synchronous quantitative pressure/glucose sensing with multiple linear correlations, which was a significantly important for biomechanical sensors [29]. Another biosensor utilized nanocellulose are developed by Neubauerova et al. (2020). For this purpose, microcrystalline cellulose (MCC) samples were treated with 2,2,6,6-tetramethylpiperidine-N-oxyl radical (TEMPO), sodium hypochlorite, and potassium bromide, to produce carboxylated NC. The carboxylated NC were characterized using Fourier Transform-Infrared Spectroscopy (FT-IR), Transmission Electron Microscopy (TEM) and conductometric titration to determine their carboxyl content. The sensing system was conducted by placed glucose oxidase (GO_X) on the carboxylated NC substrate. Upon reaction with glucose, the enzyme produced hydrogen peroxide, which turns to the blue-coloured product by reaction with horseradish peroxidase and 2,2′-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt. In the range of 1.5 to 30.0 mM, an excellent linear response to glucose was observed. The presence of carboxyl-NC enhanced the colour uniformity of the test-strip and the intrinsic linear response of the concentration range, offering carboxylated-NC a promising approach for glucose detection [30]. Ram et al. (2020) demonstrated calorimetric sensor for detection copper ion using spherical nanocellulose (SNC) modified with diethylenetriamine (DETA) and/or ethanolamine (EA) (SNC-DETA, SNC-EA, and SNC-DETA-EA). The results showed that SNC-DETA-EA and SNC-DETA exhibited calorimetric naked-eye and fluorescent activity with good selectivity at very low ion concentration, whereas SNC-EA showed poor response. SNC-DETA-EA had a detection limit of 1.03 ppm or 1.622×10^{-5} M and a strong Cu^{2+} ion absorption with a highest Langmuir capacity (q_m) of 212.76 mg/g [31].

Ling et al. (2019) fabricated calorimetric biosensor for detection of human neutrophil elastase (HNE) and a wide range of inflammatory diseases using cellulose nanocrystal as transducer surfaces. To produce a peptide-cellulose conjugate as a protease sensor of HNE, a deep eutectic solvent (DES) was used to facilitate the production of cotton cellulose nanocrystals (DCNCs). An equivalent derivative of TEMPO-

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oxidized wood cellulose nanofibrils (WCNFs) is compared to the tetrapeptide-cellulose analogue on DCNCs. Despite their lower surface area and pore diameters, DCNCs demonstrated more HNE tetrapeptide substitution and elastase sensitivity than WCNFs. The DCNCs-based colorimetric sensor has a sensitivity of less than 0.005 U/mL, making it a practical and sensitive sensor for enhanced colorimetric point-of-care protease biomarker detection [32]. Photoluminescence sensor using metal organic framework (MOF) on cellulose nanofibrils substrate was developed by Wang et al. (2020) for specifically detection of copper ions. Cellulose nanofibrils was oxidised with TEMPO and the abundance of hydroxyl groups facilitated large number of adsorption sites europium ion (Eu^{3+}), which beneficial for growth of Eu-MOF on TCNFs matrix. The functionalization was conducted via in-situ synthesis in hydroalcoholic medium. In the presence of other interfering metal ions, the produced film had a strong selectivity for copper ions. The film's fluorescence intensity declined gradually as copper ion concentration increased, and I_0/I_{-1} established a strong linear relationship with [Cu^{2+}], making the film a potential material for detecting Cu^{2+} in the human fluid [33]. Figure 2 showed schematic diagram of Eu-MOF@TEMPO-CNF film fabrication. Table 1 displayed other developments of nanocellulose as sensing material.

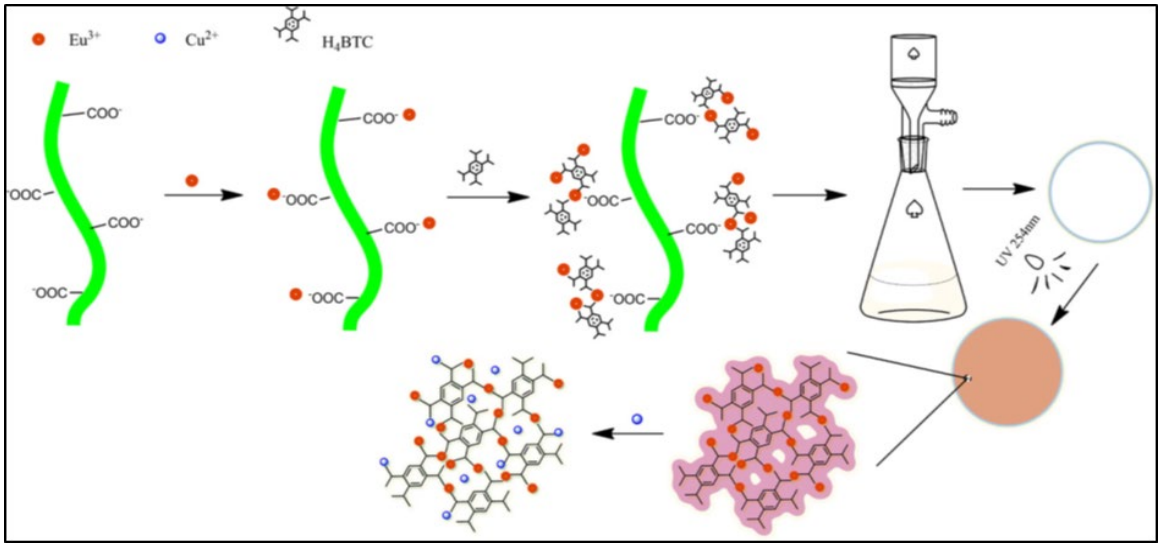

Figure 2. Schematic diagram of film fabrication Eu-MOF@TEMPO-CNFs [33]

Table 1. Developments of nanocellulose as sensing material for various applications

Nanocellulose	Type of sensor	Analytes	Limit of detection	Advantages	Ref
Microbial nanocellulose	Screen-printed carbon electrodes for electrochemical sensor	Uric acid 7β-estradiol Pb^{2+} Cd^{2+}	1.80 µM 0.58 µM 1.01 µM 0.43 µM	- oxygen species on surface improved wettability and hydrophilicity	[34]
Nanocellulose functionalized with silver nanoparticles	Surface enhanced Raman scattering (SERS)	Rhodamine 6G (R6G) Thiram Thiabendazole (TBZ) Malachite green (MG)	10^{-9} M 0.05 ppm 0.09 ppm 0.0014 ppm	- ppb level of detection - great for on-site and in-situ applications	[35]
Cellulose nanofibers coated with gold nanoparticles	Surface enhanced Raman scattering (SERS)	Thiram	10^{-8} M	- quick response (within 30 s) - less error in sampling	[36]
Gold nanorods coated with silver nanorods loaded on bacterial nanocellulose	Surface enhanced Raman scattering (SERS)	2, 4, 6-trinitrotoluene (TNT)	8×10^{-12} g/L	- less aggregation, high loading capacity - highly sensitivity	[37]

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Cellulose nanofibril (CNF) and a fluorescent carbon dot (CD)	Optical sensor	NO_x Glutaraldehyde	Not mentioned	- highly sensitivity and selectivity - improved mechanical strength - high fluorescence quantum yield	[38]
Nanocellulose from sugarcane bagasse	Calorimetric freshness indicator	CO_2	Not mentioned	- quick response	[39]
Bacterial nanocellulose	Ratiometric fluorescent sensor	Hg (II), Pb (II), Cd (II), Fe (III) and Cu (II)	60 μM	- great selectivity - good optical transparency - efficient and user friendly	[40]
Carboxylated nanocellulose functionalized with ruthenium	Photoluminescent sensor	Silver nanoparticles	1.11·10-5 mol L^{-1}	- strong sensing response - enhancement of photoluminescence - broad response range - excellent for antimicrobial activity	[41]
Nanocellulosic systems conjugated to the elastase substrate n-succinyl-Alanine-Alanine-Proline-Valine-7- amino-4-methylcoumarin and n-succinyl-Alanine-Proline-Alanine7-amido-4-methyl-coumarin	Biosensor	Protease	0.125 U/mL	- negatively charged nanocellulose promotes efficient uptake of positively charges proteases - high surface area provide extra binding sites for protease - great in prevent wound dehydration	[42]
Cationic nanocellulose	Strain sensor	Stabilize graphitic carbon nitride	Gauge factor≈5.6, 0–1.6 % strain	- ionic interaction highly provides stretchability of hydrogel - high strain sensitivity - good for self-powered devices with wide sensing ranges	[43]
Cellulose nanosheet (CNS)-multibond cross-linked poly(acrylic acid) (PAA)	Strain sensor	Human motion -face, throat, knee bending, finger knuckle	Gauge factor = 7.6 at 600–1100% strains	- stable conductivity and strain sensitivity - good mechanical and self-adhesive properties - highly stretchable hydrogel for wearable strain sensor application	[44]
Nanofibrilled cellulose incorporated with silver nanowires	Strain sensor	Human motion Voice monitoring and recognition Vehicle speed and loading	Gauge factor: 15.13 within the linear strain sensing range of 0 – 2%,	- strengthened mechanical stability - higher electrical conductivity - high sensitivity - accurate breathing detection for COVID-19 patient	[45]

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3.0 CONCLUSION

In conclusion, nanocellulose has a lot of potential as a sensing material. Basically, sensing process is hugely related to the surface reaction. Because of the large surface area to volume ratio provided by nanocellulose, it demonstrated superior responsiveness and selectivity. Furthermore, a high surface area increases the interaction between analyte and sensing material, which is one of the most important aspects of a good sensing material. The porous structure of nanocellulose with a high surface area appears to be the typical structure of nanocellulose sensor layers. Hence, nanocellulose creates a pathway for functionalization with a variety of species such as metal oxides, metal nanoparticles, and other functional groups. The agglomeration of nanocellulose upon preparation could be solved by functionalization with other substances. Thus, nanocellulose-based sensing materials are a new approach to creating biocompatible, environmentally friendly, cost-effective, simple, and conveniently disposable sensors for a variety of applications such as biomedical, engineering, and industrial purposes.

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