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NOVEL SMART MATERIAL DEVELOPMENT WITH MXENE/BACTERIAL CELLULOSE NANOCOMPOSITE

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Abstract. The use of nanomaterials over the last few decades has resulted in technological advancements across various domains like wearable health monitoring devices, energy harvesters, smart materials and structures, etc. With increasing concerns about the development of new materials on the environment and the need for sustainable solutions for such endeavors, the current work focuses on the use of biodegradable and biocompatible properties of biomaterials in combination with the excellent physical properties of nanomaterials for the development of new smart materials.

Two-dimensional MXene nanomaterials, first reported in 2011, are finding applications in multiple research domains due to their properties of hydrophilicity, high conductivity, good mechanical properties, film-forming ability, biocompatibility, etc. Bacterial cellulose is a natural polymer produced by various species of bacteria, for example, Gluconacetobacter xylinus. The bacteria-derived material shows greater mechanical properties and chemical purity when compared to standard plant cellulose. This work investigates bacterial cellulose (BC) as a biomaterial combination with MXene nanomaterials for developing smart materials. Particular focus has been laid on the material characterization studies of the new materials. The electromechanical properties of the new MXene/BC smart material are studied to identify the possibility of applying these materials for measuring physical field variables like strain, force, etc. This work forms the basis for the development of new MXene/BC smart material for sensing, self-healing, smart packaging, and several other applications both for terrestrial and space sectors.

Keywords: Smart materials, 2D nanomaterials, Bacteria-derived materials, Cellulose, Self-aware

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1 INTRODUCTION

Novel material development in the twenty-first century is focused on multifunctional materials. The source of inspiration for such multifunctional materials comes from nature and is therefore referred to as bioinspired materials. Smart materials and nanostructured materials form a subset of such multifunctional materials [1]. Smart materials can change their properties as a response to external stimuli, such as temperature, pressure, electric or magnetic fields, mechanical stress, or structure damage. They have the ability to sense changes in their environment and respond adequately by altering their physical, chemical, or mechanical properties. There are various types of smart materials such as shape memory alloys (SMAs), piezoelectric materials, electroactive polymers (EAPs), or thermo-, photo- and pH-responsive materials. The discoveries and reporting of many nanomaterials [2] in recent times have led to the use of these nanostructured materials with unique physical and chemical properties as multifunctional materials with sensing and actuating capabilities. There is also a large field of bio-based smart materials based on resources obtained from plants, bacteria, or fungi such as shape memory biopolymers, smart hydrogels, and electroactive polymers that can be used for sensing, self-healing, or environment cleaning applications.

Within the framework of sustainable development goals formulated and accepted by the member states of the United Nations, there is a need for environmentally friendly, biodegradable, and biocompatible materials for terrestrial and future space applications. There is a need for the conceptualization and development of novel materials which can achieve these goals as well as provide functionality for engineering applications. With a motivation of developing such a novel smart material, the efforts at the Space Technology Centre AGH have been on trying to bring together the advantages offered by nanomaterials and biomaterials in the form of MXene nanomaterials and Bacteria-derived cellulose, respectively. This paper provides an initial overview of the material development and initial findings from such an endeavor.

1.1 MXENE NANOMATERIALS

MXenes are a family of two-dimensional (2D) nanomaterials which are inorganic compounds of metal carbides, metal nitrides or metal carbonitrides [3]. MXene compounds offer the possibility of altering their chemical structure and stoichiometry resulting (through pre-processing and water-based or acid-based or water-less processing approaches) in configurable electronic and chemical properties [4]. This is a major advantage and motivation to study MXenes over carbon-based nanomaterials which are restricted by their sp2 carbon structure.

The first MXene reported in 2011 was titanium carbide (Ti_3C_2-MXene) [5]. Ti_3C_2-MXene has over the last decade gained prominence among 2D nanomaterials because of its hydrophilicity, film-forming ability, high conductivity [6], good elastic behavior [7], dynamic response behavior [8], wireless communication possibilities [9], microwave absorption performance [10], etc. The single layer Ti_3C_2-MXene is reported to have low resistivity of

2.31 ± 0.57 μΩ.m [11], Young's modulus of 330 ± 30 GPa, and Yield's strength of 17 ± 1.6 GPa [7]. The pure Ti_3C_2-MXene films produced by the simple vacuum filtration process have Young's modulus of 3.52 GPa [12] and blade-coating processed Ti_3C_2-MXene resulting in the free-standing film has been reported to have a high tensile strength (about 570 MPa) and Young's modulus of 20.6 GPa [6]. The pure Ti_3C_2-MXene film has also shown an electrical conductivity of 4600 S/cm [13] and electromagnetic shielding behavior with multiple combinations of MXene nanocomposites.

Along with these properties, Ti_3C_2-MXene nanomaterials are found to be biocompatible based on several implementations reported [14]. The natural chemical degradation of MXenes which has been a major challenge for the last decade has been solved in the last few years by various means as well [4], [15]. These advantages offered by Ti_3C_2-MXene material make it a strong candidate for novel smart material development with biologically-derived materials like bacterial cellulose which is discussed in the next subsection.

1.2 Bacterial cellulose

Bacterial cellulose (BC) has been the subject of extensive research for a few years already, as it presents unique properties and potential applications in various fields [16]. BC is a unique form of cellulose that is produced by certain strains of aerobic bacteria of which the most commonly used is Gluconacetobacter xylinus (also Komagataeibacter sucrofermentans, Gluconacetobacter hansenii, Acetobacter pasteurianus) [17]. It has a complex fibrous structure of glucose molecules similar to plant-derived cellulose but with some differences in its physical and chemical properties. It shows high mechanical strength, high water-holding capacity, and high crystallinity [18]. Extensive research is being conducted to characterize BC in various areas such as production, structure modifications, and applications. One such area of research is production optimization which includes controlling nutrient composition, the temperature of fermentation, pH, and oxygen supply which focuses on enhancing the yield, quality, and reproducibility [19]. Another field in which research effort is being put is modifying the properties of synthesized cellulose material for different applications. BC has been used to produce a wide range of functional materials [20], such as films, membranes, hydrogels, aerogels, composites, coatings, food packaging, wound dressings, drug delivery systems, sensors, flexible electronics, etc. To achieve so many different potential applications, researchers incorporate into the material various nanoparticles (TiO_2, AgNWs, GO, CNTs), polymers (PEG, PVA, chitosan, PCL) or other additives (sodium alginate, gelatin, glycerol) [19]. Despite efforts to enhance BC with other materials, there is also a focus on enhancing its mechanical properties by ordering the arrangement of the fibers [21].

1.3 Concept of smart material with MXene/BC nanocomposite

In the initial efforts to develop novel smart material with a nano-biomaterial combination, we first propose the concept of nano-bio-smart material with MXene/Bacterial Cellulose (MXBC) nanocomposite. BCs have been used for several optoelectronic device developments [22] over the years and the electromechanical properties of this biomaterial/biopolymer derived from bacteria offer a biodegradable and biocompatible substrate or matrix for further composite development with nanomaterial. The fibers produced by bacteria in the Kombucha mixture form a complex network with fiber diameters as low as a few nanometers. Similar fibers are formed using an electrospinning process which still might have controllability and processing issues in producing such intricate fibers.

Nature-derived materials have their purpose. In this case, BC production by bacteria (in a symbiotic relationship with yeast present in the Kombucha mixture) is to avoid exposure to light and form a cover from exposure to air in the container used. Though they have very good morphological characteristics resulting in decent mechanical properties, they act as a dielectric for electrical applications. Though dielectric materials have their usage in developing electronic devices, our goal of developing a smart material requires the response to stimuli properties of the material in a wider electromechanical domain. This functionality of tunable electrical properties is provided by nanomaterials, in this case, Ti_3C_2-MXene. The hydrophilicity of BCs and Ti_3C_2-MXene complement each other in the development of this novel nano-bio-smart material. Thus, various experimental efforts are being made to have these MXBC nanocomposites made for applications in self-aware material. This is further elaborated in the next subsection.

1.4 Idea of self-aware Nano-Bio-smart materials

Self-aware materials are ones that mainly have the dual functionality of self-sensing and self-healing. The capability of self-sensing helps in the assessment of the state of the material under physical phenomena of changes of field variables (temperatures, strains, etc.), creep, damage initiation, and damage propagation. The capability of self-healing help in repair in case of damage initiation or propagation due to desirable or undesirable external stimuli (drop off the table, impact loads from the sand storm on civil infrastructure, the effect of shockwave load on substances in the realm of supersonic or hypersonic flows). Added to these two main functionalities are the requirement of an intelligent system for self-aware operation (maybe even called a central processing unit analogous to computers), energy self-sufficiency for sustained operation, and communication to be able to interface with humans.

There are several efforts to use living materials like fungi [23], [24], slime mold [25], etc., for such self-aware material development. In this paper, we look at the possibilities of using non-living BCs produced by bacteria in combination with MXene nanomaterials to achieve some of the functionalities mentioned in the preceding paragraph. The initial experimental implementation of developing MXBC is discussed in the following sections. Along with experimental studies of the structure and morphology of BCs and MXBC nanocomposite, the elastic properties and electrical resistance of BCs are reported as initial results. This forms the basis for further discussion on the possibilities of MXBCs for nano-bio-smart material development.

2 EXPERIMENTAL METHODS

2.1 MXene nanomaterial preparation

The water-based and acid-based preparation of Ti_3C_2-MXene has been discussed by the research team in previous works [8] and elaborated on the various aspects of chemical stability, uncertainty identification, etc [4]. The engineer-friendly less toxic approach of chemical etching with hydrochloric acid and lithium fluoride is used for removing the aluminum layers present in the precursor Ti_3AlC_2-MAX phase material. The proportions of materials used and the suppliers are provided in detail in the previous work of the authors. The mildly intensive layer delamination (MILD) process is employed for delaminating the multilayer Ti_3C_2-MXenes obtained after etching, and the material washing process is employed to achieve neutral pH colloidal solution along with centrifugation. The delaminated Ti_3C_2-MXene is then stored as a colloidal solution with high concentration to keep the MXenes stable from degradation in dark and refrigerated conditions. A portion of the delaminated Ti_3C_2-MXene colloidal solution is used with BCs for experimentation.

2.2 Bacterial cellulose production

BC material was obtained with the use of wild bacteria strains cultivated in the Space Technology Centre, AGH University of Science and Technology. 10 g of black tea was added into 1.5 liters of water and boiled for 15 min. After that time, the tea leaves were taken out of the liquid and 90 g of glucose was added and stirred until dissolved. The liquid was left to cool down to 25^{o}C and then 100 ml of pre-cultivated broth (mother culture) was added. As prepared broth was left for two weeks at room temperature ranging from 20^{o}C to 22^{o}C (controlled within laboratory room conditions). After the film grew thick (around 30-40 mm) it was taken out of the liquid and cleaned with water. This film is then dried to remove moisture for control experiments with BCs as well as MXBC nanocomposite preparation.

2.3 Structure and morphology analysis

The structure was examined by X-ray diffraction with a PANalytical Empyrean diffractometer (Malvern, UK) with CuKα radiation (λ=1.54056 Å). XRD patterns were recorded in the 2θ angle range of 5^{o} to 60^{o} with a resolution of 0.026^{o}. The morphology was examined by atomic force microscopy (AFM) with a Bruker Multimode 6 microscope (Billerica, USA). AFM measurements were carried out in PeakForce Tapping mode with ScanAsyst-Air probe.

2.4 Mechanical and electrical properties tests

Mechanical tests were carried out with the use of a universal testing machine inspect table of 5 kN by Hegewald & Peschke (Nossen, DE). The samples with dimensions equal to 50 mm x 5 mm were cut out of the BC material sheet parallel and perpendicular. The force sensor used was 100 N and the initial force of measurements was 1 N. The speed of measurements was 20 mm/min. A simple commercial direct current type multimeter (Brymen - BM907s) was used for the two-terminal measurement of electrical resistance (Open Circuit Voltage: 0.45 VDC) of the BC sample and screen-printed Ti_3C_2-MXene layer on BC.

3 RESULTS AND DISCUSSIONS

3.1 X-ray diffraction analysis

XRD patterns of the BC and MXBC are shown in Figure 1. The diffraction lines observed for the BC sample at 2θ = 14.5°, 16.8°, and 22.7° can be attributed to [002], I_α[110], I_β[110], respectively, which are planes of pure crystalline bacterial cellulose. BC diffraction lines reflecting I_α[110], I_β[110] planes decreased their intensities in the MXBC sample (seen at 16.8° and 22.7°), but the [002] plane line (at 14.5°) increased probably due to overlapping with the filler Ti_3C_2-MXene material. From the literature [8], Ti_3C_2-MXene prominently has [002], [004], [006], etc. planes after the delamination process. The diffraction lines in the MXBC sample observed at 2θ = 9.7°, 19.3°, 39.1° are signals coming from MXene material, however, there are many new diffraction lines which some could not be interpreted as plain MXene lines [8]. This might suggest that there is an interaction between the MXene and BC that changes the structure configuration. However, more investigations and analyses need to be carried out on MXBC in comparison with MXene and BC to prove this quantitatively.

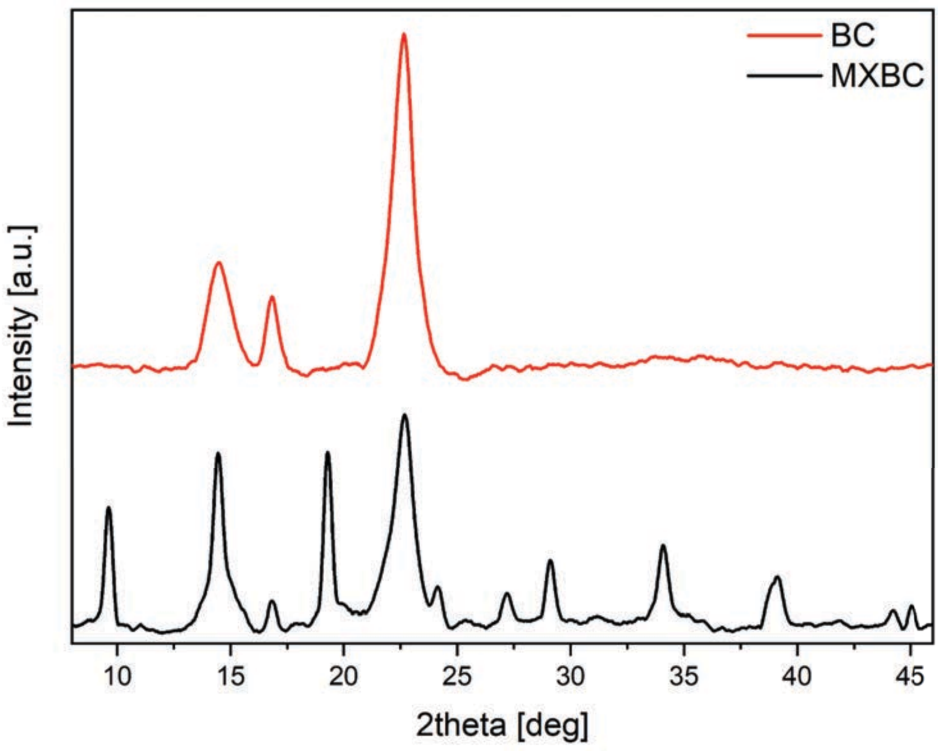

Figure 1: XRD patterns of BC and MXBC

3.2 Atomic forces microscopy

BC sample surface topography is shown in Figure 2. The surface features fibers of cellulose with different diameters mostly from 90 nm to 120 nm and depths going from 0 to 300 nm. They tend to stick slightly to each other to form bigger structures oriented in one direction, however, individual fibers appear to be not arranged in any particular direction. The fibrous structure shows high surface area thanks to spaces between bigger agglomerates of fibers which could possibly be increased by separating individual fibers from each other, creating spaces for particle intercalation.

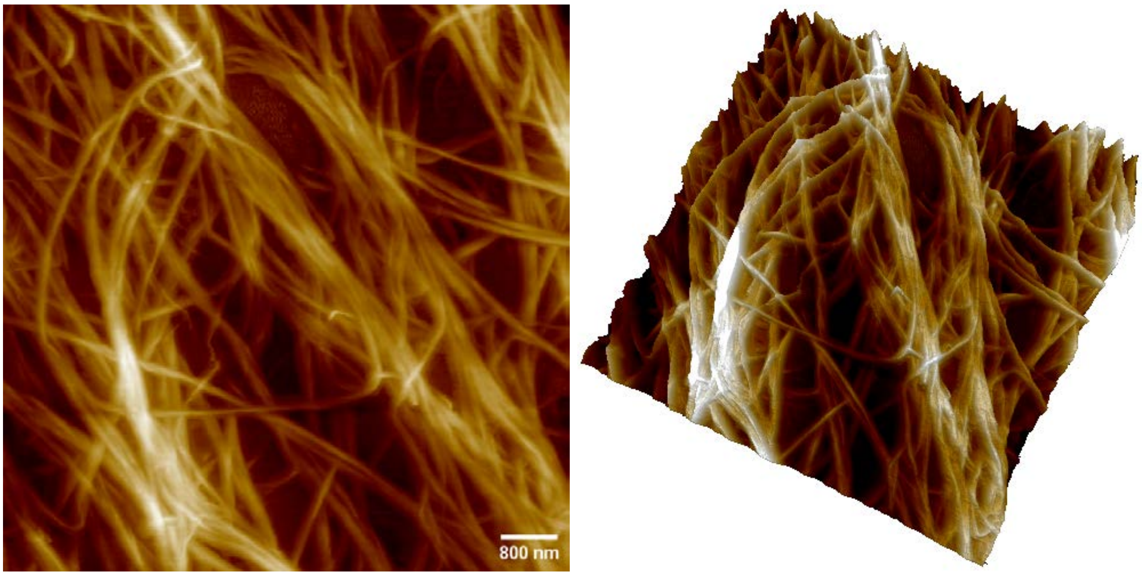

Figure 2: AFM image of BC nanofibers

3.3 Mechanical tests

The elastic properties of BCs are determined by the universal testing machine. The samples prepared by cutting the BC film in parallel and perpendicular (in comparison with the longer edge of the BC film) direction yield Young’s modulus, tensile strength, elongation at maximum force, and total elongation of the BC samples. A total of 14 samples for samples cut in a parallel configuration and 10 samples for samples cut in a perpendicular configuration are tested to obtain the mean values of elastic properties along with their standard deviations. The tensile strength and elongation at maximum force of the BC material are in the same range of 28 ± 4 MPa and 10.5 ± 0.5 percent, respectively, indicating the random orientation of the nanofibers of BC provides close to isotropic behavior in the plane of the BC sample. Young's modulus determined from the experiments with both parallel and perpendicular configurations does not exhibit a large difference as both mean quantities are in the same order of magnitude (251 ± 23 MPa and 293 ± 37). Further morphological studies might provide more information on the reasons for variations in the total elongation between the two sets of samples. These initial results (reported in Table 1) provide information on the possibility of using BCs as a matrix material for the addition of Ti_3C_2-MXene fillers. The modification of the elastic properties due to the addition of Ti_3C_2-MXene is beyond the scope of this paper and is reserved for future reporting.

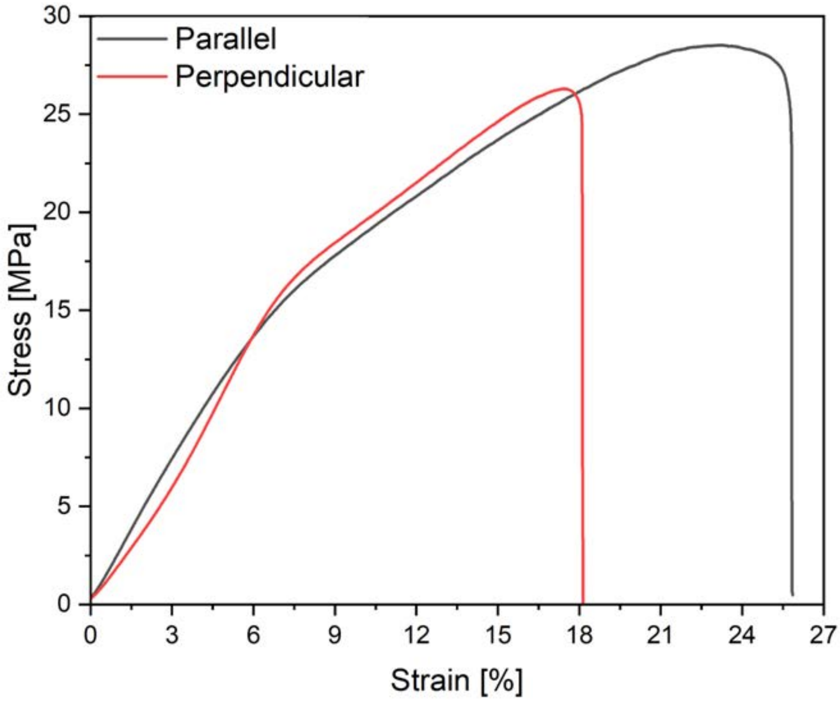

Figure 3: Stress-strain curves of tested BC samples

Table 1: Results of BC mechanical tests

Configuration	Parallel	Perpendicular
E [MPa]	251±23	293±37
σM [MPa]	28±4	28±3
εM [%]	11±1	10±1
εt [%]	24±3	19±3

3.4 Direct current electrical measurements

The direct current electrical measurement using commercial multimeter results in electrical resistance of the freshly prepared BC samples in the range of a few mega Ohms and dried samples exceeding the measuring range of the multimeter. Simple screen-printing of Ti_3C_2-MXene on the BC substrate yields Ti_3C_2-MXene material with electrical resistance in the range of a few Ohms. These initial studies indicate possibilities of using Ti_3C_2-MXene material for electrically conductive printing ink on BC substrates as well as the possibility of modifying the BC electrical properties by using Ti_3C_2-MXene as filler material.

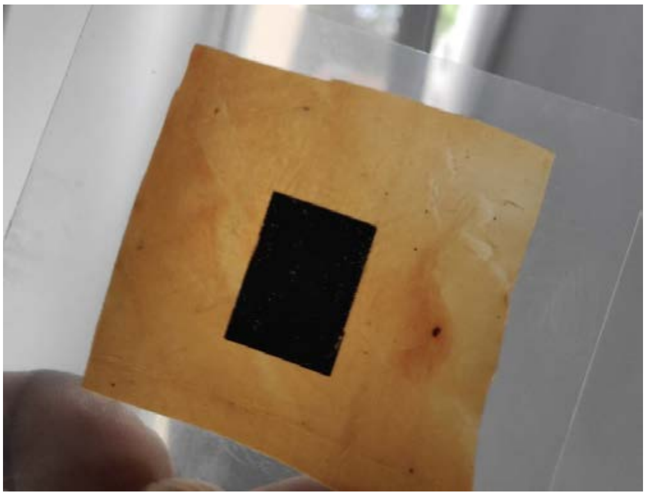

Figure 4: BC with screen-printed MXenes for initial investigation of electrical resistance

4 CONCLUSIONS

The possibility of developing a novel smart material that could be biodegradable, biocompatible, and environmentally friendly is explored in this paper. The advantages offered by BC in the form of being a biodegradable and biocompatible material are fused with the excellent physical properties offered by Ti_3C_2-MXene nanomaterial to form a new nano-bio-smart material. The paper discusses the concept of these new nano-bio-smart materials formed by MXBC nanocomposites. The idea of self-aware material development with MXBCs is discussed to propose the possible applications of these new materials.

The structure of BC and MXBC is reported by the XRD analysis. This study indicates possible interaction between BC and Ti_3C_2-MXene leading to modification in the material structure for MXBC compared to BC and Ti_3C_2-MXene. The morphology study of the BC is conducted with AFM and the diameters of the fibers are found to be in the range of a few tens or hundreds of nanometers, thus classifying this bacteria-derived material as nanofibers at the sub-micron scale. The macroscopic structure of the wet BC is more of a hydrogel and the dried BC is a biopolymer film. The elastic properties of the BC are measured for several samples in two different configurations of sample preparation. The average Young's modulus of the BC material is about 272 MPa with a tensile strength of about 28 MPa. Finally, the BC material is found to be dielectric as also reported in the literature, and further modification of BC with Ti_3C_2-MXene results in modification of the electrical properties. These initial investigations provide a fundamental idea of how the new nano-bio-smart material can be further developed for various applications in the terrestrial and space sectors. Further studies into the modification of BC material and various incorporation methods of MXene into BC matrix are envisaged for the development of self-aware materials.

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