Surface Engineering of Biomaterials, Devices and Regenerative Materials: Health, Food, and Agriculture Applications
Room Town & Country B - Session MD2-1-TuA

Coatings and Sensors for Health, Food and Agriculture: Antibacterial, Bioactive, and Flexible Interfaces I

Moderators: Diego Mantovani, Université Laval, Canada, **Phaedra Silva-Bermudez**, Instituto Nacional de Rehabilitación Luis Guillermo Ibarra Ibarra, Mexico

1:40pm **MD2-1-TuA-1 Deposition and Surface Characterization of Low-Pressure Plasma Ultra-Thin Coatings Designed for Biomedical Applications**, ***Laurent Houssiau [laurent.houssiau@unamur.be]***, University of Namur, Belgium **INVITED**

Our research group combines expertise in surface characterization techniques, namely X-ray Photoelectron Spectroscopy (XPS) and Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS), with low-pressure plasma deposition of thin films and nanoparticles. This approach enables projects across diverse fields, including biomaterials, biosensing, tissue imaging, hybrid materials, adhesion, and cultural heritage. In this presentation, we will focus on the deposition and characterization of ultra-thin (<100 nm) films that impart new functionalities to substrate materials.

We will first present our work on gradient coatings for dental implants, designed to promote osseointegration while providing antibacterial properties. These coatings are deposited on Ti-6Al-4V (Ti64) alloys using Plasma-Enhanced Chemical Vapor Deposition (PECVD) from a titanium-containing organometallic precursor (titanium isopropoxide) injected in gas phase with argon and oxygen. By gradually decreasing the oxygen flow during the deposition, a compositional gradient is created, from an inorganic TiO_2-like layer at the implant interface to a more organic TiO_xC_y layer near the bone interface, ensuring a smooth transition between the metallic implant and bone tissue. A final magnetron sputtering step, within the same plasma chamber, introduces ZnO nanoparticles into the top TiO_xC_y layer, enhancing antibacterial activity. Depth profiling by XPS and ToF-SIMS confirms the compositional gradient and nanoparticle deposition.

We will then highlight our collaboration with Prof. Mantovani's group at Université Laval on plasma-deposited diamond-like carbon (DLC) and fluorocarbon coatings. Here, XPS and ToF-SIMS have been instrumental in elucidating coating quality, composition, and behavior under various conditions.

Finally, we will present recent research on graphene-based biosensors—an application that also integrates plasma modification and surface analysis for biomedical use. As a proof of concept, the biotin–streptavidin interaction was employed. Graphene layers were amine-functionalized by plasma polymerization, followed by biotin grafting, with XPS monitoring each surface modification step. Electrical I–V measurements revealed a Dirac point shift correlated with streptavidin concentration, demonstrating detection capabilities down to 0.1 nM.

2:20pm **MD2-1-TuA-3 Silver-Copper Nanocoating (Sakcu®) Deposited on Stainless Steel Brackets to Reduce Biofilm Formation of *Streptococcus Mutans* and Potentially Prevent Early Dental Caries**, ***Alejandra Cervantes-Ramírez [aleebathory1@gmail.com]***, *Lorena Reyes-Carmona, David Eduardo Martínez-Lara, Andrea Quiroz-Cervantes, Gina Prado-Prone, Sandra E. Rodil, Argelia Almaguer-Flores,* UNAM, Mexico

Introduction: Dental brackets facilitate the accumulation of bacteria during orthodontic treatments, favoring the biofilm formation of bacteria that produce organic acids, such as *Streptococcus mutans* (*S. mutans*). This bacterium can lead to enamel demineralization and cavity development. Therefore, creating antibiofilm devices can significantly contribute to preventing early-stage cavities during orthodontic treatments.

Objective: To deposit the Ag-Cu nanocoating (SakCu®) on conventional metallic brackets and evaluate its capacity to reduce the biofilm formation of the cariogenic strain *S. mutans.*

Methods: The deposition of the Ag-Cu nanocoating on stainless steel brackets (American Orthodontics®) was carried out using the magnetron sputtering technique. The micro-morphology and chemical composition of the coated and uncoated bracket surfaces were evaluated using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), respectively. The *in vitro* antibiofilm effect of surfaces was assessed by Alamar blue kit and the counting of colony-forming units (CFU) assay using *S. mutans* (ATCC 25175). The evaluation was conducted after 1, 3, and 7 days of incubation under anaerobic conditions. Qualitative observation of bacterial adhesion on the surfaces was performed using SEM.

Results: The stainless-steel surface of the orthodontic brackets was coated with a uniform nanolayer of silver and copper without significantly changing their surface morphology. The microbiological results showed that the Ag-Cu nanocoating reduced the biofilm formation of *S. mutans*, especially at the initial incubation times (55% on day 1 and 85% on day 3), compared to the biofilm formed on the surface of brackets without the nanocoating.

Conclusion: The SakCu® nanocoating on orthodontic brackets reduced biofilm formation of the cariogenic *S. mutans* strain, especially during early contact periods. This suggests that the Ag-Cu nanocoating can potentially prevent biofilm formation on orthodontic devices and the development of initial caries, thereby improving treatment and rehabilitation in the mid-term.

Acknowledgments: Financial support of UNAM-PAPIIT # IT207824, # TA10424and SECTEI #159, projects.

2:40pm **MD2-1-TuA-4 ZnO Nanowires: A Platform for Biosensing Applications**, *Rafael Salinas, Shirlley Martínez, Guillermo Santana Rodriguez, Carlos Ramos,* ***Ateet Dutt [adutt@iim.unam.mx]***, UNAM, Mexico **INVITED**

Our research focuses on the design and characterization of advanced nanomaterials, particularly hybrid gold–zinc oxide (Au–ZnO) nanowires, for chemical and biosensing applications. We investigate how variations in size, morphology, and composition influence their structural and functional performance. The synergistic combination of Au and ZnO imparts these nanowires with distinctive physicochemical properties, enabling the creation of highly sensitive and efficient sensing platforms.

In one study, we developed one-dimensional ZnO nanowire-based systems for the rapid detection of cancer biomarkers, demonstrating precise photoluminescent signal generation through the integration of nanoscale receptors. Furthermore, we achieved tunable photoluminescence responses across analyte concentrations ranging from 1×10^2 to 1×10^8 CFU mL^{-1}, allowing direct visualization of targeted bacterial cells on ZnO nanowire surfaces.

This contact-based nano-biosensing approach enables real-time detection while substantially reducing both processing and response times—an essential advantage for rapid pathogen identification in critical scenarios. By deepening our understanding and control of these hybrid nanostructures, we aim to advance their practical implementation in clinical diagnostics and broader biomedical technologies.

4:00pm **MD2-1-TuA-8 Photoresponsive Bilayer Coating Integrating Zinc and a Chitosan-Antibiotic Drug Delivery Film for on-Demand Antimicrobial Photodynamic Therapy in Biomedical Implants**, ***Samuel Santana Malheiros [samuelsmalheiros@gmail.com]***[1], *Maria Helena Rossy Borges,* University of Campinas (UNICAMP), Brazil; *João Gabriel Silva Souza,* UnG, Brazil; *Elidiane Cipriano Rangel,* UNESP, Brazil; *Carlos Fortulan,* University of São Paulo, Brazil; *Nilson Cristino da Cruz,* UNESP, Brazil; *Eduardo Buozi Moffa,* University of Saskatchewan, Canada; *Bruna Egumi Nagay, Valentim Adelino Ricardo Barão,* University of Campinas (UNICAMP), Brazil

Despite significant advances in surface treatments, failures of biomedical implants due to bacterial colonization, wear and insufficient bioactivity remain persistent clinical challenges. Here, we engineered a light-responsive antimicrobial bilayer coating for titanium implants consisting of: (i) an inner porous oxide layer doped with bioactive elements Ca, P, and Zn produced by plasma electrolytic oxidation (PEO) and covered by (ii) an outer biodegradable chitosan (CS) thin film for controlled delivery of the photosensitive antibiotic demeclocycline (DMC), enabling antimicrobial photodynamic therapy, a light-activated process where a photosensitizer produces reactive oxygen species (ROS) to eliminate microorganisms. After preparation, samples underwent morphological, physical, chemical, optical, crystallinity, and tribological characterization. Coating's photo-responsiveness was indirectly assessed via methylene-blue degradation under illuminated and dark conditions. Antimicrobial performance was tested under illuminated and dark conditions using a 96-hour polymicrobial biofilm model (human saliva as inoculum). Bioactivity was assessed by hydroxyapatite formation, proteomic analysis of the adsorbed proteins from human blood, and cytocompatibility with pre-osteoblastic cells.

[1] **Graduate Student Award Finalist**

Regarding results, PEO generated a moderately rough, porous oxide layer (Ra ≈ 1 µm) composed of calcium, phosphorus, and zinc oxide, partially covered by the CS film, which reduced roughness to Ra ≈ 0.6 µm while maintaining hydrophilic behavior (contact angle < 40°). CS and DMC incorporation was confirmed by EDS, FTIR, and XPS analyses and UV–Vis spectroscopy attested DMC's photoactive absorption within the visible light wavelength spectra (Soret band ~450 nm). XRD confirmed high crystallinity of the PEO inner oxide layer which imparted mechanical robustness under tribological loading, while the outer polymeric film provided a cushion effect evidenced by the lowest friction coefficient, minimal mass loss, and preservation of the inner layer morphology. Upon light irradiation, photoexcited DMC generated ROS, leading to > 3-log_{10} reductions in biofilm viability and > 50% decreases in metabolic activity, dry mass, and protein content, along with favorable shifts in microbial community composition. Beneficial protein adsorption profiles, enhanced hydroxyapatite formation, and cytocompatibility confirmed the coating's bioactive potential. Overall, the developed smart light-responsive coating unites ROS-mediated antimicrobial action on-demand, wear protection, and bioactivity in an industry-scalable platform, with potential to enhance biomedical implants longevity and reliability.

4:20pm **MD2-1-TuA-9 Electrospun Nanocomposite Membranes for the Development of Osteoinductive Microambients***,* ***Phaedra Silva-Bermudez [pssilva@inr.gob.mx]****, Julieta García-López,* Unidad de Ingeniería de Tejidos, Terapia Celular y Medicina Regenerativa; Instituto Nacional de Rehabilitación Luis Guillermo Ibarra Ibarra, Mexico*; Gina Prado-Prone,* Laboratorio de Biointerfases, DEPeI, Facultad de Odontología, Universidad Nacional Autónoma de México*; Monserrat Ramirez-Arellano, Gustavo E. Martínez-Murillo,* Unidad de Ingeniería de Tejidos, Terapia Celular y Medicina Regenerativa; Instituto Nacional de Rehabilitación Luis Guillermo Ibarra Ibarra, Mexico*; Lucia S. Flores-Hidalgo,* Posgrado en Ciencia e Ingeniería de Materiales, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México*; Sandra E. Rodil,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México

Displaced, particularly open fractures, represent a significant clinical challenge due to their complexity, variability andhigh complication rate, predominantly due to infections and delayed bone healing rates. In Mexico, approximately 50,000 cases occur annually, with infection rates reaching up to 10%, which is notably higher than those reported in developed countries. Given this concern, there is a growing interest for developing biomedical materials capable of promoting bone regeneration while minimizing infection risk.

Electrospinning is a versatile technique that enables the fabrication of polymeric nanofibrous membranes with high porosity, conformability, and suitable for controlled drug release and degradation. Recently, nanomaterials have attracted significant interest to develop composite materials with specific biofunctionalities. In particular, magnesium oxide nanoparticles (MgONPs) have demonstrated osteogenic properties by promoting cellular proliferation and differentiation towards the osteoblastic phenotype.

Thus, the aim of the present project is to develop composite (polycaprolactone, type B gelatin and MgONPs) membranes capable of thriving an appropriate microambient at the site of the bone fracture, aiming to contribute to increase osteogenesis and decrease bacterial infection. Membranes were fabricated via electrospinning based on polycaprolactone (PCL) and gelatin (Gel), and incorporated with MgO NPs at different concentrations (2, 5, and 7 wt%). Their micro-morphology, chemical composition, wettability, and mechanical properties were examined using SEM, EDS, FTIR, WCA, TGA, DSC and tension tests. The biocompatibility and osteoinductive capability of the membranes was assessed using human bone marrow-derived mesenchymal stem cells (BM-MSCs). Cell viability was assessed after 24 h exposure to membrane lixiviates (MTT assay), and after 24 and 72 h of cells directly cultured on the membranes surfaces (calcein/ethidium homodimer assay). The osteoinductive potential of the membranes was evaluated by assessing the osteogenic differentiation of BM-MSC in contact with membranes lixiviates, by using Alizarin Red staining and immunofluorescence assays against collagen Type I, Osteocalcin and Osteopontin.

The nanocomposite membranes exhibited a microfibrillar-porous structure, and appropriate wettability and mechanical properties for clinical use. The cytocompatibility and osteoinductive effects were dependent on the MgO NPs concentration, with higher NPs concentration increasing cell differentiation towards the osteoblastic phenotype.

4:40pm **MD2-1-TuA-10 Cationic Coatings for Titanium Implants: Integration of Tribocorrosion Resistance and Bioactive Performance***,* ***João Pedro dos Santos Silva [jpedrooss85@gmail.com]****,* Ecole des Mines de St-Etienne - Université de Lyon, France*; Maria Helena Rossy Borges, Thais Terumi Sadamitsu Takeda, Catia Sufia Alves Freire de Andrade, Valentim Adelino Ricardo Barão,* Universidade Estadual de Campinas, Brazil*; Jean Geringer,* Ecole des Mines de St-Etienne - Université de Lyon, France

Failures in implant therapies remain common, mainly due to biofilm formation and the exacerbated inflammatory response caused by coating degradation and release of metallic particles, which inhibit tissue healing and bone regeneration. To address these challenges, we developed a cationic coating (CC) combining antimicrobial properties, tribocorrosion protection, and enhanced biological response through electrostatic interactions, while also improving surface bioactivity and osseointegration potential, all while being drug-free. Titanium discs were treated by plasma electrolytic oxidation (PEO) to form –OH groups and then silanized individually with 3-aminopropyltriethoxysilane (APTES), tetraethyl orthosilicate (TEOS), or 3-glycidyloxypropyltrimethoxysilane (GPTMS), resulting in the CC. Five groups (Ti, PEO, APTES, GPTMS, and TEOS) were evaluated for surface characterization, tribocorrosion performance, microbiological behavior, and cytocompatibility. Micrographs revealed distinct morphologies among the groups, and 3D confocal images confirmed rough topography and increased surface area. After silanization, an increase in positive surface charge confirmed by zeta potential and higher hydrophobicity indicated effective chemical modification. Electrochemical tests under open circuit potential, cathodic, and anodic polarization showed that CCs exhibited low current densities, fast repassivation, and high stability under friction, unlike Ti and standalone PEO. The coefficient of friction and mass loss were reduced by up to 75% compared to titanium, demonstrating superior tribocorrosion resistance. Electrochemical impedance spectroscopy confirmed film integrity after wear, indicating resilient behavior and the formation of an effective barrier. The microbiological response revealed the best anti-adhesion performance for the APTES group, with reductions of 45% (Escherichia coli) and 65% (Staphylococcus aureus). Other experimental groups also showed significant reductions ($p < 0.0001$). Regarding biofilm formation, clear differences were observed between control and experimental groups, confirming that CC effectively controls microbial growth. Finally, human mesenchymal stem cell (hMSC) assays showed higher metabolic activity and proliferation on APTES and TEOS surfaces, with increases above 150% compared to the control and greater viability on days 1, 3, and 8. Thus, CCs demonstrated high electrochemical stability, tribocorrosion resistance, anti-adhesion effects, and the ability to modulate biofilm growth, along with superior biocompatibility, establishing them as a promising solution for more durable, stable, and biologically integrated implants.

5:00pm **MD2-1-TuA-11 PEO-Polymer-Bioglass Hybrid Coatings for Bioactivity and Tribocorrosion Improvement of Ti–6al–4v***,* ***Paulo Soares [pa.soares@pucpr.br]****,* Pontifícia Universidade Católica do Paraná, Brazil

Metallic implants play a crucial role in various medical applications. Achieving rapid bone regeneration, preventing corrosion and wear, controlling metal ion release, and preventing infections are key objectives in implant development. However, osseointegration and implant durability can be affected by several factors. This study focuses on the development of multifunctional coatings for metallic implants, particularly titanium alloys, to provide wear and corrosion protection, and bioactivity properties. Oxidation of titanium alloy (Ti6Al4V) was performed using the plasma electrolytic oxidation (PEO) technique. Subsequently, a bioabsorbable PCL polymer film containing bioglass particles (BGs) was deposited on the oxidized surfaces. The coatings were characterized using scanning electron microscopy, energy-dispersive spectroscopy, X-ray diffraction, and contact angle measurements. Tribocorrosion tests were conducted in artificial saliva, and in vitro assessments of bioactivity were made. The PEO technique successfully provided a suitable surface for subsequent polymer film adhesion. The presence of BG particles enhanced the bioactivity of the coatings. Tribocorrosion tests revealed improved wear and corrosion resistance and reduced friction coefficients for the hybrid coatings compared to uncoated surfaces. These findings pave the way for the development of implant coatings with improved clinical performance, addressing the challenges associated with metallic implants.

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