th BRAZILIAN
INANOCELLULOSE
SUMMIT 2025



# BOOK OF ABSTRACTS

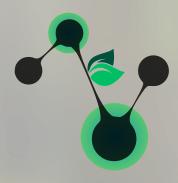
AUGUST 6<sup>TH</sup> - 8<sup>TH</sup>



LORENA - SÃO PAULO

Lorena School of Engineering (EEL), University of São Paulo

### **V BRAZILIAN NANOCELLULOSE SUMMIT**



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# Welcome Message

Dear Conference visitors,

It is with great pleasure that I welcome you to the V Brazilian Nanocellulose Summit, held from August 6 to 8, 2025, and organized by the Laboratory of Applied Bionanotechnology at the Lorena School of Engineering, University of São Paulo (EEL/USP). Since its first edition in 2015, the Summit has functioned as a platform for connecting researchers, students, and industry professionals involved in the development of nanocellulose science and technology in Brazil.

This year's program brings together a diverse range of experts from academia, research organizations, startups, and industry. The agenda includes plenary sessions, pitch talks, and poster presentations, enabling researchers at all stages of their careers to share their work and engage in productive dialogue.

I am grateful to all speakers, participants, sponsors, and supporters for making this edition possible. I also thank the Local Organizing Committee for their commitment and hard work throughout the preparation of the event.

With this collective effort, the 2025 Summit becomes an excellent opportunity to strengthen our community and advance the development and application of nanocellulose-based materials across sectors in Brazil and beyond.

#### **Prof. Valdeir Arantes**

Conference Chair

V Brazilian Nanocellulose Summit



#### **ABOUT**

### V Brazilian Nanocellulose Summit

The Brazilian Nanocellulose Summit is the leading national event dedicated exclusively to nanocellulose research, innovation, and applications. Since its first edition in 2015, the Summit has fostered collaboration across academia, industry, and government, supporting the growth of a robust and dynamic ecosystem for nanocellulose in Brazil. The 2025 edition, hosted at the Lorena School of Engineering, University of São Paulo (EEL/USP), consolidates its role as a key forum for scientific exchange and strategic discussion on sustainable bio-based materials. Over three days, participants will engage in discussion on cutting-edge research, emerging technologies, regulatory frameworks, and market-driven trends shapping the field.

The technical program spans the full spectrum of nanocellulose materials, including cellulose nanocrystals (CNC), nanofibrils (CNF), microfibrillated cellulose (CMF), and cellulose nanospheres (CNS), with topics ranging from production and characterization to applications and feasibility studies. New initiatives introduced in this edition include a Nanocellulose Characterization Workshop to strengthen analytical capabilities; a session on industry applications connecting research and commercialization; and a round-table discussion, titled "Challenges and Future Perspectives for Nanocellulose", bringing together key actors across the value chain. For the first time, the program also features a dedicated session on the Brazilian regulatory landscape, addressing the implications of standardization efforts for both research and industry.

By promoting interdisciplinary exchange and cross-sector collaboration, the Summit aims to accelerate the translation of scientific advances into innovative products, scalable technologies, and informed policy. It serves not only as a showcase of recent progress, but also as a strategic platform to align efforts, strengthen networks, and drive the bioeconomy through nanocellulose.

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# BACTERIAL CELLULOSE FROM HB BIOTEC: BRIDGING BIOTECHNOLOGY AND INDUSTRY ACROSS HEALTH, FOOD, CIRCULAR FASHION AND COSMETICS

Authors: FONTES, M.<sup>1</sup>; MEDALHA, V.M.<sup>1</sup>; IZIDORO, K.C.<sup>1</sup>; BARUD, H.S.<sup>1</sup>.

Institution: <sup>1</sup>HB Biotec Company, Araraguara, Brazil

My journey into science and entrepreneurship began during my undergraduate studies in Biomedicine, under the mentorship of Dr. Hernane Barud, who introduced me to scientific research and later became my partner in founding HB Biotec. Over the years, my academic experience in biocellulose research evolved into applied innovation. I completed my PhD developing monoclonal antibodies for prostate cancer biomarkers, and later coordinated technology-based projects with companies such as Ambev. Founded initially to offer research consultancy services, HB Biotec now focuses on scaling the production of bacterial cellulose through fermentation and developing cellulose-based bioproducts for diverse applications. Our portfolio includes medical wound dressings (smooth, porous, and hydrogel formats), pharmaceutical-grade dressings nanoparticles, cosmetic facial masks, kombucha, and even biosensors. The company has also directed its biocellulose toward applications in the circular fashion industry, as well as using it as a food thickening agent and in animal feed additives.In 2022, we executed a technology transfer agreement with World Veterinária Ltda, resulting in a patented PHMBbased hydrogel for veterinary use (BioCell BS Wet). We've also participated in public innovation programs such as Startup Araraguara and Catalisa ICT, and in strategic alliances with SUZANO and SENAI, enabling technical and financial growth.Today, I coordinate HB Biotec's R&D projects and innovation strategy, working to validate our products, secure funding, and expand market presence. Our approach combines scientific rigor, sustainability (using citrus industry byproducts), and cross-sector innovation. Finally, the HB Biotec case exemplifies how science and entrepreneurship can drive impactful solutions in wound care, cosmetics, biotechnology, and beyond.

# COFFEE AGRO-WASTE AS A SUBSTRATE FOR BACTERIAL CELLULOSE PRODUCTION BY K. INTERMEDIUS

Authors: ARIAS, L.V.A.<sup>1</sup>; SILVA, V.S.<sup>2</sup>; MIRANDA, J.F.D.<sup>3</sup>; SILVA, N.C.C.<sup>3</sup>; USBERTI, F.C.S.<sup>1</sup>

Institution: <sup>1</sup>School of Agricultural Engineering, University of Campinas, Campinas, Brazil

<sup>2</sup>Department of Materials Science and Engineering, Universitat Politècnica de Catalunya (UPC BarcelonaTech), Poly2 Group, ESEIAAT, Terrassa, Spain

<sup>3</sup>Department of Food Science and Nutrition, School of Food Engineering (FEA), University of Campinas (UNICAMP), Campinas, Brazil

Cellulose nanofibrils (CNFs) are promising biomaterials, but their pronounced hydrophilicity restricts their applicability, especially in interactions with apolar media or in products requiring water and vapor barriers. Surface modification of CNFs to reduce their water affinity is crucial for expanding their applications, enhancing dimensional stability, improving moisture resistance, and increasing compatibility with hydrophobic components. In this study, we developed and characterized thin CNF films with reduced hydrophilicity, focusing on three systems: (i) CNFs enzymatically esterified with butyric acid; (ii) CNFs containing lignin nanoparticles (LNPs); and (iii) CNFs both esterified and containing LNPs. Low-angle laser light scattering (LALLS) was employed to determine the nanofibril size distribution in suspension, thereby ensuring film homogeneity. X-ray diffraction (XRD) was used to determine the crystallinity index, while atomic force microscopy (AFM) and scanning electron microscopy (SEM) were employed to visualize the surface morphology and fibril entanglement. Fourier Transform Infrared Spectroscopy (FTIR) confirmed ester bond formation and lignin presence. Thermogravimetric analysis (TGA) assessed thermal stability, while contact angle measurements guantified hydrophilicity reduction. Zeta potential measurements indicated surface charge and colloidal stability, shedding light on intercomponent interactions. This comparative analysis elucidated the impact of enzymatic esterification and LNP incorporation on the structural, thermal, chemical, electrokinetic, and surface properties of CNF films. The findings support the potential use of these optimized films as platforms for the transdermal delivery of hydrophobic drugs, such as ibuprofen.

# DEVELOPMENT OF AN INNOVATIVE ANTIMICROBIAL AND PH-RESPONSIVE BACTERIAL NANOCELLULOSE/ALGINATE HYDROGEL FOR ADVANCED WOUND CARE

Authors: GONÇALVES, I.S.<sup>1</sup>; COLTURATO, V.M.M.<sup>1</sup>; BRAGUINI, L.F.<sup>1</sup>; FONTES, M.L.<sup>1</sup>; BARUD, H.S.<sup>1</sup>

Institution: <sup>1</sup>HB Biotech, São Carlos, Brazil

# ELECTROCHEMICAL SENSOR MODIFIED WITH BACTERIAL NANOCELLULOSE AND CARBON NANOTUBES FOR FUROSEMIDE ANALYSIS

Authors: SANTOS, G.T.V.<sup>1</sup>; BARRETO, F.C.<sup>1</sup>; LEÃO, A.L.<sup>1</sup>; CESARINO, I.<sup>1</sup>

Institution: <sup>1</sup>UNESP, Botucatu, Brasil

Chronic wounds represent a major global public health challenge, affecting millions of individuals and leading to substantial social, economic, and healthcare burdens. In this context, the development of multifunctional wound dressings capable of promoting tissue repair, preventing infections, and enabling real-time monitoring is a strategic priority. This project, developed within a biotechnology startup, aims to develop an advanced wound care product based on a hydrogel composed of bacterial nanocellulose and sodium alginate. Bacterial nanocellulose is a highly pure biopolymer with a threedimensional nanofiber network that supports the healing process and enables the incorporation of bioactive compounds. Based on these properties, our goal is to expand our current wound dressing product line by incorporating two key features: antimicrobial activity and pH-responsiveness. These additions are expected to enhance infection control while enabling the monitoring of wound status through pH changes in the exudate, an important biomarker of inflammation and infection. The hydrogel will undergo comprehensive validation through analysis, physicochemical and structural characterizations, antimicrobial testing, and preclinical evaluation in an animal wound healing model. Ultimately, this work is intended to support the transition to clinical trials and potential market introduction.

Nanocellulose in electrochemical sensors has the potential to increase the stability, sensitivity and selectivity of the modified electrode for detecting species of environmental and biological interest. Bacterial nanocellulose (BNC) can be used as a polymeric matrix for electrical conductors such as multi-walled carbon nanotubes (MWCNT) and silver nanoparticles (AgNPs). The use of organic waste in its culture medium is a sustainable alternative to reduce the production costs of BNC. The objective of this work is the dispersion in water of BNC, together with MWCNT and AgNPs, in the modification of a glassy carbon electrode (GCE) for the detection of the drug furosemide (FUR). BNC was produced in culture medium containing residual yeast from beer production. Syntheses were performed in ultrapure water with three different amounts of BNC (2, 4 and 6 mg) for each 20 mg of functionalized MWCNT. The morphological and electrochemical characterizations of the nanocomposites were performed. The electrochemical analyses were conducted by cyclic and differential pulse voltammetrhe voltammetric parameters were optimized. The developed GCE was evaluated in the determination of FUR in 0.2 mol L-1 PSB solution at pH 3.0. It was observed that the GCE modified with the composite containing 2.0 mg of NCB presented a better definition of the electrochemical processes and increased the sensor sensitivity. The linear correlation for the two irreversible oxidation peaks of furosemide was determined between 0.5 to 9.0 µmol L-1. The detection and quantification limits for the two oxidation peaks were 0.83  $\mu$ M, 2.76  $\mu$ M, and 2.76  $\mu$ M and 4.84  $\mu$ M, demonstrating that bacterial nanocellulose produced from waste can be used to obtain a high added value product, through a simple and lowcost synthesis with nanotubes and nanoparticles dispersed in water, increasing the sensitivity of the electrochemical sensor for the rapid analysis of furosemide.

# INTEGRATION OF BISMUTH COMPOUNDS AND BACTERIAL CELLULOSE AEROGELS WITH VISIBLE PHOTOCATALYTIC ACTIVITY

Authors: SILVA, V.A.<sup>1</sup>; RIBEIRO, S.J.L.<sup>1</sup>; NETO, E.P.F.<sup>1</sup>

Institution: <sup>1</sup>São Paulo State University (Unesp) - Instituto de Química, Araraquara, Brasil

# The increase in anthropogenic contamination of water resources and demand for drinking water worldwide results in the urgency of research for wastewater remediation. Among the technologies aimed at degradation and removal of contaminants, stands out the heterogeneous photocatalysis promoted by semiconductor nanomaterials, considered a lowcost process, which can make use of sunlight to occur. Many bismuth compounds show promising photocatalytic activity under visible light, mainly due to their low band gap energy, low toxicity and relative chemical stability. The typical synthesis of photocatalysts involves the production of their micro or nanometric size particles, desirable for the effectiveness in heterogeneous photocatalysis. but raises challenges for its

photocatalysts involves the production of their micro or nanometric size particles, desirable for the effectiveness in heterogeneous photocatalysis, but raises challenges for its extensive application. In this work we report the association of bismuth compounds and bacterial cellulose for heterogeneous photocatalysis in a flow system. The modification of the surface of the organic biopolymer was performed by crystallization of BiVO4, BiOI and BiOCI in solvothermal treatment in different syntheses, followed by supercritical drying in CO2 to become an aerogel. The meticulous characterization of the materials allowed us to relate their physical-chemical and structural properties with photocatalytic activity, resulting in up to 80% degradation of methylene blue dye in 120 minutes of simulated solar irradiation. Therefore, the work marks a significant

advance in membrane synthesis for continuous photocatalytic applications of flux under sunlight. This work plays a crucial role

in the development of new materials for the removal of organic

pollutants and water purification.

#### SUSTAINABLE VALORIZATION OF DAIRY BY-PRODUCTS AS SUBSTRATES FOR BACTERIAL NANOCELLULOSE PRODUCTION

Authors: FRANZ, L.M.<sup>1</sup>; LIMA, D.B.<sup>1</sup>; BRITO, A.G.S.<sup>1</sup>; MONTANHER, P.F.<sup>1</sup>; SOUZA, S.S.<sup>1</sup>

Institution: <sup>1</sup>Universidade Tecnológica Federal do Paraná, Dois Vizinhos, Brasil

The production of bacterial nanocellulose (BNC) depends significantly on the nutritional composition of the culture medium, especially the carbon and nitrogen sources. Although the standard Hestrin-Schramm (HS) medium is effective, its high cost-mainly due to peptone and yeast extract—limits large-scale applications. Dairy by-products such as cheese whey (CW) and stretching water (SW) are nutrient-rich and widely available in the dairy industry, which struggles with the disposal of these coproducts. This study aimed to evaluate BNC production using HS medium modified with CW and SW to reduce costs and promote waste valorization within a circular economy approach. Komagataeibacter xylinus was cultivated in six formulations for each by-product: (A) pure HS (control), (B) HS + 20% byproduct, (C) HS + 50% by-product, (D) HS without peptone (replaced by the by-product), (E) HS without yeast extract (replaced), and (F) HS without peptone and yeast extract (both replaced). Wet membrane masses were statistically analyzed, revealing significant effects of both by-product type and medium composition. For CW, significant differences occurred between groups A-C, A-F, C-D, and D-F; for SW, between A-B, A-C, A-F, C-D, D-F, and E-F. SEM analysis showed variations in microfibril arrangement, resulting in membranes with distinct porosity and density. FTIR spectra showed similar patterns across all treatments, with key functional groups present, indicating the by-products did not compromise BNC chemical structure. These findings demonstrate that K. xylinus successfully produced BNC using the proposed media. Both CW and SW proved functional in medium composition, supporting sustainable and costeffective BNC production, particularly for supplementing or replacing conventional carbon and nitrogen sources. This exploratory study supports future research into fully replacing standard nutrients with dairy byproducts to enable industrial BNC production with properties suitable for biofunctional applications.

# APPLIED NANOCELLULOSE RESEARCH AND INNOVATION: PERSPECTIVES FROM THE SENAI INSTITUTE OF TECHNOLOGY IN PULP AND PAPER

Authors: MAROTTI, B.1; ALMEIDA, M.1

Institution: ¹Cellulose and Paper SENAI Institute, Telêmaco Borba, Brasil

The SENAI Institute of Technology in Pulp and Paper (IST-CP), located in Telêmaco Borba, Brazil, is part of the national SENAI Network of Innovation and Technology, composed of more than 80 institutes across the country. This structure was inspired by international models such as the Fraunhofer Institutes in Germany, aiming to bridge applied research and industrial demands through mission-driven innovation. Telêmaco Borba holds a long-standing role in Latin America's pulp and paper sector, not only as the home of the first technical course in pulp and paper in the region, created in the 1960s, but also as part of a broader regional cluster of major industrial operations in this field. This combination of educational tradition and strong industrial presence led to the region being chosen as the headquarters of the only SENAI Institute in Brazil specialized exclusively in pulp and paper technologies. The Institute operates through three main pillars: consulting, metrology and technical services, and research, development, and innovation (RD&I). Among its areas of expertise, nanocellulose stands out as a strategic focus. IST-CP has dedicated infrastructure for fiber functionalization, characterization, and pilot-scale validation. Currently, the Institute manages over 30 RD&I projects in collaboration with industrial and academic partners, using both direct contracting and public funding. Key nanocellulose initiatives include: Surface-functionalized nanocellulose for polymer reinforcement Bio-based barrier coatings for packaging Integration into sustainable industrial products

# BIOMASS RECALCITRANCE IN ORGANOSOLV PRETREATMENT: FROM NANO TO MOLECULAR SCALE INSIGHTS

Authors: NASCIMENTO, M.D. $^1$ ; COLOMBARI, M.F. $^2$ ; FOCASSIO, B. $^1$ ; SCHLEDER, R.G. $^1$ ; CAPAZ, B.R. $^1$ ; DRIEMEIER, C. $^2$ ; BERNARDES, S.J. $^1$ 

Institution: <sup>1</sup> Brazilian Nanotechnology National Laboratory - LNNano, Campinas, Brasil <sup>2</sup>Biorenewables National Laboratory - LNBr, Campinas, Brasil

Lignocellulosic biomass is an abundant renewable resource, but its recalcitrance limits the fractionation of lignin and cellulose, hindering the production of fuels and materials. This study investigates the effect of organosolv pretreatment on the adhesion force between cellulose microfibrils (CMFs) and lignin. Force spectroscopy was used to quantify the adhesion between CMF and lignin in different organic solvent concentrations. Approximately two thousand force curves were collected for each solvent condition and analyzed using machine learning to classify the experimental data into types of celluloselignin interactions. The results show that the adhesion force increased by adding organic solvent, from ~135 pN in water to ~260 pN in pure organic solvent. Molecular dynamics simulations were employed to investigate the impact of solvent composition on lignin conformation. The simulation results indicate that the presence of organic solvent induces a more open lignin conformation. Radial distribution function analysis was used to examine the spatial arrangement of hydrophilic and hydrophobic groups of lignin relative to the cellulose crystal facets, revealing that these distances change depending on the solvent composition. Our results demonstrate that the combination of experimental force curves, data analysis, and molecular simulations provides a robust approach for studying the impact of organosolv solvent composition on celluloselignin interactions at the nanoscale and molecular levels.

# CAPTURE AND DETECTION OF MICROPLASTICS AND DYE IN WATER USING CELLULOSE MICROFIBERS VIA RAMAN SPECTROSCOPY

Authors: OLIVEIRA, S.M.J.  $^1$ ; CARVALHO, B.I.  $^1$ ; DOGNANI, G.  $^1$ ; CONSTANTINO, L.C.J.  $^1$ 

Institution: <sup>1</sup>FCT UNESP, Presidente Prudente - SP, Brasil

# COATING PAPER WITH MICROFIBRILLATED CELLULOSE, NATURAL RUBBER, AND PECTIN TO IMPROVE BARRIER PERFORMANCE

Authors: RAMASINI, B.1; SILVA, D.1; BERNARDES, J.1

Institution: 1 CNPEM, Campinas, Brasil

Cellulose-based materials have gained prominence in the development of new materials. The main reason for this interest lies in the fact that cellulose originates from renewable sources such as plants in general, wood, agricultural residues, and cereal straw, making it a strategic resource for sustainable applications [1]. Among these materials, cellulose has proven to be efficient in capturing contaminants in water, where pollutants such as microplastics (MPs) are concentrated on the surface of the fibers [2]. MPs are plastic particles ranging in size from 1  $\mu m$  to 5 mm and are considered emerging pollutants whose environmental impacts are still not well understood [3]. This study employed Raman spectroscopy to investigate the interaction between polyamide 6 (PA6) microplastic and nickel tetrasulfonated phthalocyanine dye (NiTsPc), as well as to evaluate the effectiveness of cellulose microfibers (CMFs) in capturing these pollutants from water. The experiments involved mixing PA6 (at different concentrations) with NiTsPc (3×10-6 mol/L), followed by the addition of CMFs (1.0 mg/mL). UV-Vis analyses showed that the intensity of NiTsPc characteristic bands decreased with increasing PA6 concentration, indicating dye adsorption onto the microplastic surface, facilitated by CMFs. The study of analyte interactions also revealed the characteristic bands of both NiTsPc and PA6, without spectral alterations, further indicating a physical interaction between NiTsPc and PA6. The results suggest that CMF+PA6 exhibits a synergic effect on NiTsPc removal by its adsorption onto the microfibers and microplastic (0.9 mg/g for PA6 and 1.9 mg/g for CMF+PA6). While Raman measurements confirm the physical interaction of the pollutants, they also demonstrate that CMFs assist in capturing MPs without causing damage during the detection process. This highlights CMFs as a sustainable alternative for capturing and detecting microplastics in water.

The use of paper in packaging is a sustainable alternative to traditional plastics due to its biodegradable and recyclable nature. Nevertheless, paper has limited barrier properties against oil, gases, and water vapor, which restricts its applications. Microfibrillated cellulose (MFC) has been recognized as an eco-friendly solution to enhance these barrier properties, providing strong resistance to oxygen, grease, and mineral oils, although it struggles with water and water vapor resistance. This study introduces an easy onepot method for combining MFC and natural rubber latex (NRL) with pectin to improve the barrier capabilities of paper. Initially, the mixture of MFC and NRL in water was found to be unstable, which adversely impacted its coating performance. However, the inclusion of pectin greatly improved both the dispersibility and colloidal stability of the blend, leading to a more consistent formulation. To assess the barrier properties of the coated paper, we measured the water vapor transmission rate (WVTR at 23°C and 50% relative humidity) and grease resistance. One coating layer effectively reduced the WVTR by over eight times, dropping from 732 to 88 g.m<sup>2</sup> day-1. Furthermore, the coated paper exhibited remarkable resistance to oil and grease, earning the highest rating on the barrier test kit (kit rating of 12). This research achieved the creation of a colloidally stable formulation that provides outstanding barrier properties and a sustainable packaging option.

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#### CROSSLINKING OF POROUS CRYOGELS FROM **OXIDIZED SUGARCANE BAGASSE MICROFIBRIL**

HYDROCHAR, **BIOCHAR** AND **CELLULOSE** NANOFIBRILS PREPARED FROM AGRO-WASTE FROM THE SEMI-ARID REGION OF MINAS GERAIS

**AEROGELS** 

Authors: FERREIRA, E.1; NASCIMENTO, D.1; BERNARDES, J.1; ALMEIDA, T.2

Authors: SETTER, C.1; LOPES MEDINA, M.1; LAGO, A.1; LAGO, R.1; SANADI, A.2; TONOLI, G.1

ΩF

DEVEL OPMENT

Institution: 1CNPEM/LNNANO, Campinas, Brasil

Institution: <sup>1</sup>Universidade Federal Lavras, Lavras, Brasil <sup>12</sup>University of Copenhagen, Copenhagen, Denmark

<sup>2</sup>UFABC/CNPEM, Campinas, Brasil

Global demand for sustainable products has increased, emphasizing materials and technologies that reduce environmental impact. Biomass presents a promising alternative due to its renewable nature and carbon neutrality. Cellulose, the most abundant biopolymer in lignocellulosic biomass, can be processed in its fibrillar form or through the regeneration of solubilized cellulose into various morphologies. In this study, cellulose microfibers (MFC) with residual lignin were directly isolated from sugarcane bagasse using a simplified oxidation process developed by our research team. Radical oxidation mediated by the catalyst 2,2,6,6- tetramethylpiperidine-1-oxyl (TEMPO) utilized 10 and 20 mmol/g of the oxidizing agent sodium hypochlorite. The MFC isolated with a lower degree of oxidation (MFC10) is yellowish due to a higher lignin concentration and contains larger particles. As the degree of oxidation increases (MFC20), the color becomes lighter, and the particle size decreases. Dispersions containing 3 and 6 % w/w of MFC10 and MFC20, with and without mechanical treatment, were used to manufacture porous materials, which possess potential applications in environmental decontamination. When dried, all samples have high porosity (above 90%), low density (ranging from 30 to 70 mg.cm-3) and physical integrity. However, they disintegrate easily in water, limiting their application. Therefore, these porous materials were chemically cross-linked with citric acid (CA) and 1,2,3,4-butane tetracarboxylic acid (BTCA), in the presence of sodium hypophosphite (SHP). Preliminary results show that the mechanical properties and water stability of MFC20 were significantly improved by crosslink. However, cryogels with high lignin (MFC 10) disintegrated in water, indicating that residual lignin negatively impacts the crosslinking with CA and BTCA.

To investigate the use of alternative materials for the production of environmentally friendly and sustainable adsorbents, this study proposed the production of cellulose nanofibril aerogels with biochar and hydrochar incorporated in different proportions (0 to 35% w/w). The aerogels were produced from cellulose nanofibrils from Eucalyptus sp. and carbonaceous materials from umbu residues. The samples were evaluated for their physical, mechanical, and morphological properties, as well as their oil absorption and retention capacity. The aerogels exhibited extremely light and porous structures, with densities ranging from 0.25 to 0.31 g/cm<sup>3</sup> and porosities ranging from 96.73 to 98.23%. The best mechanical properties were observed in the aerogels with 5% biochar and 15% hydrochar, which showed increases in compressive strength of 19% and 36%, respectively, compared to the control aerogel. Regarding oil absorption, the high performance of aerogels containing 0%, 5%, and 15% biochar was favored by the high porosity of the structure. Similarly, despite having lower porosity, the aerogel containing 35% hydrochar also demonstrated good oil absorption capacity, attributed to the presence of interconnected macropores. The results demonstrate that the aerogels produced have great potential for application as adsorbent and insulating materials, especially due to their high porosity and low density.

# INFLUENCE OF COMPOSITION AND DRYING METHOD ON BISPHENOL A REMOVAL BY MICROFIBRILLATED CELLULOSE/BENTONITE

Authors: NUNES, A.B.S.M.1; PETRI, F.S.D.1; ROJAS, J.O.2

Institution: ¹University of São Paulo, São Paulo, Brazil ² University of British Columbia, Vancouver, Canada

#### KINETIC AND COMPARATIVE STUDY OF ANIONIC DYE REMOVAL ON FUNCTIONALIZED CELLULOSE MICROFIBERS

Authors: CARVALHO, B.I.  $^1$  ; OLIVERIA, S.M.J.  $^1$  ; DOGNANI, G.  $^1$  ; CONSTANTINO, L.C.J.  $^1$ 

Institution: 1 FCT Unesp, Presidente Prudente, Brazil

This study explores the modification of microfibrillated cellulose (MFC) with bentonite intercalated with cetyltrimethylammonium bromide (B@C) for removing Bisphenol A (BPA), an endocrinedisrupting chemical, from water. Two composite materials were developed with different MFC/B@C ratios: MFC/2B@C (70.91wt% MFC) and MFC/8B@C (37.90wt% MFC). These composites were processed into two forms - cryogels (freeze-dried) and xerogels (oven-dried). The results showed that both composition and drying method significantly affected adsorption capacity. The MFC/8B@C cryogel demonstrated superior performance with a maximum adsorption capacity (gmax) of 113.70 mg/g according to the Langmuir model, substantially higher than MFC/2B@C (40 mg/g). This enhancement is attributed to the greater proportion of B@C available for BPA interaction. The freeze-drying method consistently produced materials with better adsorptive properties than oven drying. In the MFC/8B@C system, gmax decreased from 113.70 mg/g (cryogel) to 43.94 mg/g (xerogel), while in MFC/2B@C, the reduction was less pronounced (40 mg/g to 34.23 mg/g). The Redlich-Peterson model best described the adsorption mechanism across all systems, reflecting the heterogeneous nature of the MFC/B@C adsorbents. The observed  $\beta$  values (0.60-0.78) indicate adsorption on an energetically heterogeneous surface involving multiple simultaneous interaction mechanisms, including ion-dipole interactions, hydrophobic interactions, and hydrogen bonding. In conclusion, the combination of high B@C content and freezedrying creates the most effective material for BPA removal from aqueous media, offering a promising approach to address this environmental contaminant.

Synthetic dyes are widely used in the food industry to enhance appearance and boost consumption. However, their use and improper disposal are linked to environmental and health risks (carcinogenesis, fetal malformations, and fertility issues) [1,2]. To address this, a substrate of cellulose microfibers (CMF: 1.0x10<sup>-3</sup>g) and CMF + copper nanoparticles (CMF/CuNPs: 9.15x10-4g) is being investigated for the removal of dyes from aqueous solutions. This study focuses on the adsorption kinetics of two anionic dyes: nickel tetrasulfonated phthalocyanine (NiTsPc) and Allura Red (AR), from 2 min to 24 h of adsorption time. For NiTsPc  $([NiTsPc]_0=5.0x10^{-5}mol/L),$ CMF/CuNPs demonstrated superior removal efficiency compared to CMF, likely due to their higher positive surface charge (CMF=6.6±0.4mV; CMF/CuNP=11.5±0.2mV) and greater surface area due to the nanoparticles. CMF showed a stable adsorption plateau varying slightly over time considering SD (13.8±0.4% at 2min vs. 21.7±5.7% at 24h). In contrast, CMF/CuNPs showed overall higher removal rates, with stable adsorption over time considering SD, and an increase observed at 24h (40.7±2.9% 2min vs. 70.0±4.1% at 24h). For CMF+AR ([AR]<sub>0</sub>=1.0x10<sup>-5</sup>mol/L), adsorption ranged from 19.9±5.3% to 26.7±0.8% in the first 120 minutes, with a significant increase after 24h. However, for CMF/CuNPs ([AR]<sub>0</sub>=5.0x10<sup>-5</sup>mol/L), the 24h removal did not improve significantly (50.9±4.2% at 90min and 57.6±7.1% at 24h). All systems reached adsorption plateaus over time. The pseudo-second-order (PSO) kinetic model best fit the data (R2: 0.98-0.99). In contrast, pseudofirst-order (PFO) showed poorer fits (R2: 0.11-0.84). The prevalence of PSO model indicates chemisorption (stronger and irreversible bonds). However, other analyses suggest a physical adsorption mechanism, supported by unchanged Raman and UV-Vis spectra, and a shift in zeta potential values, where the microfiber surface becomes negatively charged after adsorption of the anionic dyes. The occurrence of PSO may be related to the rapid adsorption equilibrium and strong electrostatic interaction.

# MICROFIBRILLATED CELLULOSE: A MULTIFUNCTIONAL SUSTAINABLE PLATFORM

Authors: SANTOS, W.1; MUGUET, M.C.S.1; SOMMER, S.M.1

Institution: <sup>1</sup>Klabin, Telêmaco Borba-PR, Brazil

# RHEOLOGICAL PROPERTIES OF CELLULOSE NANOFIBRILS MODIFIED BY ESTERIFICATION

Authors: MEDINA, M.L.<sup>1</sup>; LAGO, A.S.<sup>1</sup>; LAGO, R.C.<sup>1</sup>; GUERRERO, A.<sup>2</sup>; TONOLI, G.<sup>1</sup>

Institution: ¹Universidade Federal de Lavras, Lavras, Brazil ²Universidad de Seville, Seville, Spain

In recent years, sustainability has become a central focus in discussions across various markets due to increased awareness, environmental legislation, and technological advancements. The use of renewable ingredients is gaining attention and has become an essential factor, making microfibrillated cellulose (MFC) a potential material for several markets. MFC is a biopolymer derived from cellulosic pulp that undergoes an intensive fibrillation process. This process increases the surface area, exposing more hydroxyl groups, which enhances its reinforcing, and stabilizing properties formulations. The objective of this work is to present three successful case studies across different sectors, demonstrating the technical feasibility, versatility, and multifunctionality of MFC. Case 1: MFC in Cosmetics - MFC acts as a substitute for structuring agents, thickeners, and stabilizers of fossil origin. In cleansing formulations, MFC was compatible with anionic, nonionic, and cationic surfactant systems. In emulsions, MFC stabilized and modified the sensorial, also helped stabilize mineral in sunscreens. Case 2: MFC in Architectural Paints -Adding MFC to architectural paints enables optimization of thickener and resin systems, bringing more sustainability and reducing the use of fossil-derived raw materials. It also improves performance regarding scrub resistance, mud cracking, and sagging. Case 3: MFC in Printing Paste - Partial replacement of fossil-based thickeners was achieved in textile printing formulations. Improvements were observed in the color fastness during abrasion and wash cycle tests. Case 4: MFC in Papers - MFC can be applied as a reinforcement agent enabling a gain in the mechanical and physical properties which leads to an increased value for the product or savings in the pulp consumption. These case studies demonstrate that MFC, besides being renewable, offers versatile and high-performance applications, contributing to sustainability in multiple markets.

Cellulose nanofibrils (CNFs) possess outstanding properties such as high mechanical strength, biodegradability, and a large specific surface area, which have attracted growing interest for various industrial applications. However, their inherent hydrophilicity significantly limits their performance in moisture-sensitive environments. To overcome this limitation, chemical modification has emerged as a promising strategy to enhance the surface hydrophobicity of CNFs. Additionally, understanding the rheological behavior of CNFbased systems is crucial not only for proper material characterization but also for optimizing processing conditions -especially when aiming for uniform dispersion within polymeric matrices. This study aimed to investigate the effects of surface modification of CNFs via esterification using babassu oil, focusing on changes in the rheological behavior of the resulting suspensions. Aqueous suspensions (1% w/w) of unmodified and babassu oilmodified CNFs were prepared and analyzed using an Anton Paar MCR102e rotational rheometer (Austria). Viscosity flow curves and thixotropy tests were performed in triplicate at 25°C. The viscosity results revealed a marked decrease in apparent viscosity at a shear rate of 100 s<sup>-1</sup>, dropping from 203.76 mPa·s for pure CNFs to 75.019 mPa·s for the modified sample. This reduction is likelydue to the introduction of hydrophobic moieties on the CNF surface, which diminish intermolecular hydrogen bonding and weaken the three-dimensional fibrillar network typically formed in aqueous media. Thixotropy tests showed values exceeding 100% for both samples (121.6% for unmodified and 122.1% for modified CNFs), indicating pronounced network rebuilding after shear. Although such behavior is considered atypical, it has been reported in highly fibrillated nanocellulose systems and may be associated with the formation of secondary interactions or microaggregates during resting periods—evidence of a strong self-organizing tendency within the suspension. Overall these findings demonstrate that functionalization through esterification is a viable approach to tuning their rheological properties, potentially broadening their application in advanced colloidal and composite systems.

# SUSTAINABLE PACKAGING DESIGN STRATEGIES WITH CELLULOSE NANOMATERIALS

Authors: LAVOINE, N.1

Institution: <sup>1</sup>NC State University, Raleigh, USA

Global population increases are directly associated with increases in waste from society. This, in combination with societies' desire for single-use products (or disposable packaging) and the unintended littering of plastic products, is negatively affecting our environment. Although packaging materials are integrally important to the food chain (food protection, shipping, and preservation), the contribution of packaging material to the entire environmental footprint in the food value chain is estimated to be up to 45% depending on the type of food and packaging materials. The overconsumption of petroleum resources and increasingly stringent environmental legislation for waste management demand the development of more sustainable and environmentally compliant packaging solutions. Over the past two decades, the valorization and conversion of low-cost lignocellulosic biomass into high valueadded applications has gained strong attention. The synthesis of nanomaterials from renewable resources is particularly creating a revolution in biobased materials, as they combine the advantages of synthetic nanomaterials with renewability, biodegradability, and biocompatibility. This creates the potential for 'game-changing' advances in the design of sustainable and functional food-packaging substrates. In this presentation, I will share our latest strategies towards designing sustainable packaging substrates from cellulose nanomaterials, spanning from process-barrier properties studies, multilayer vs. nanocomposites to plasma surface modification and tailored interfacial design for biodegradability. This presentation aims to discuss challenges and opportunities in the field, with strong emphasis on developing high water vapor barrier from nanocellulosic materials.

# 3D-PRINTED PHOTONIC STRUCTURES BASED ON GELLAN GUM AND NANOCELLULOSE WITH ANISOTROPIC PLASMONIC PARTICLES

Authors: TORRES, F.R.1; SIQUEIRA, G.2; BARUD, H.S.3; CAIUT, J.M.A.3

Institution: <sup>1</sup>Universidade de São Paulo (USP), Ribeirão Preto, Brazil <sup>2</sup>Swiss Federal Laboratories for Materials Science and Technology (Empa), Dübendorf, Switzerland

<sup>3</sup>Universidade de Araraguara (UNIARA), Araraguara, Brazil

# AN INTEGRATED FRAMEWORK FOR LIFE CYCLE ASSESSMENT OF NANOCELLULOSE: FROM DEVELOPMENT TO APPLICATION

Authors: CRUZ, T.T.D.1; ARANTES, V.1

Institution: ¹Lorena School of Engineering, University of São Paulo, Lorena, São Paulo

The production of photonic devices from bio-based functional composites can take advantage of the gelation properties of gellan gum (GG), a natural polysaccharide, and the anisotropy generated by cellulose nanocrystals (CNC) to achieve hydrogels that can be processed to suit different applications. Here, GG and CNC are the basis for the formulation of a 3D printing ink designed to fabricate three-dimensional multilayer porous structures with interesting optical responses. The ink was combined with а network-forming molecule, glycidyloxypropyltrimethoxysilane (GPTMS), for the study of photopolymerization of the structure after printing . Hydrogels exposed to UV light demonstrated this polymerization effect, as confirmed by rheological studies showing an increase in the material's viscoelastic moduli. The CNC ratio was optimized to achieve ideal printing conditions, acting as a rheological modifier and resulting in a printable ink capable of maintaining the shape of a three-dimensional well-defined structure. The extrusion process produces shear forces that align anisotropic structures along the printing direction, therefore, AFM and SEM analyses revealed the preferred orientation of the CNC within the printed filament. This alignment was quantified through calculations based on 2D wide-angle X-ray scattering (WAXS) diffractograms, revealing a degree of orientation of 64% for two printing needle diameters used. Following these results, the oriented environment enables the incorporation of anisotropic gold nanostructures that can induce an arrangement with spectroscopic responses based on the surface plasmon resonance effect, allowing the development of efficient sensors as an active medium for surface-enhanced Raman scattering (SERS), an analytical technique with promising applications in chemical sensing. Taking advantage of these properties, luminescent nanoparticles (Nd:YAG and Nd:YAB) were inserted into the ink for ongoing studies of circularly polarized laser emission from the printed structure.

Cellulosic nanomaterials (CNs), also known as nanocelluloses, are one of the most prominent green materials of modern times due to its versatility of use and a unique combination of physical, chemical and biological properties. The application of these renewable materials has become increasingly important for producing composite materials, paper, adsorbent products, food and beverages, paints and coatings, adhesives, packaging, electronics, and medical and cosmetic products. The objective of this work is to evaluate the sustainability of CNs. To achieve this, the life cycle assessment methodology will be used through the application of a framework previously created by the authors and applied to cellulose nanofibrils (CNF) and cellulose nanocrystals (CNC) production processes on a laboratory and industrial scale. The attributional study covers the cradle-togate boundary of 1 kg CNs suspension obtained from bleached eucalyptus Kraft pulp (BEKP) enzymatically processed. Inventory data were obtained from laboratory experiments, published literature and databases and then used for scale-up calculations. The Recipe Midpoint impact assessment method is applied to estimate the effects of the principal process parameters on the environmental impacts of the obtained CNs. The hotspots will be highlighted, as well as the environmental performance of both types of NC obtained will be compared to conventional production routes, aiming to elucidate the potential of the enzymatic route. Obtaining reliable results will guide the eco-design of these materials and contribute to strengthening the bioeconomy and promoting sustainable development.

# CELLULOSE NANOCRYSTALS AND NANOFIBRILS GRAFTED WITH POLY(2-ALKYL-2-OXAZOLINE)

Authors: GUMIERO, J.P.1; REZENDE, A.C.1

Institution: 1UNICAMP, Campinas, Brazil

# CELLULOSE NANOCRYSTALS CHOLESTERIC FILMS: INFLUENCE ON LANTHANIDE SPECTROSCOPY AND POTENTIAL CIRCULARLY POLARIZED EMISSION

Authors: SANCHES, P.H.L.  $^1$ ; PUGINA, R.S.  $^2$ ; SANTOS, M.V.  $^2$ ; BARUD, H.S.  $^3$ ; RIBEIRO, S.J.L.  $^3$ ; CAIUT, J.M.A.  $^3$ 

Institution:  $^1$ Universidade de São Paulo (USP), Ribeirão Preto, Brazil

<sup>2</sup>Universidade Estadual Paulista (UNESP), Araraquara, Brazil <sup>3</sup>Universidade de Araraquara (UNIARA), Araraquara, Brazil

This study presents the development of thermoresponsive grafted nanocelluloses hvdrogels based on thermoresponsive poly(2-alkyl-2-oxazoline)(POx) for biomedical applications. Cellulose nanocrystals (CNC) were obtained via acid hydrolysis, while cellulose nanofibrils (CNF) were produced through mechanical fibrillation using a Masuko Ultragrinder. Both nanocellulose types were derived from sugarcane bagasse, a renewable and abundant agricultural residue in Brazil, and were oxidized to introduce carboxyl groups onto their surfaces. The synthesis of POx was performed to achieve low polydispersity and tunable lower critical solution temperatures (LCSTs) by varying polymer molar mass and side chains. Polymers with molar masses ranging from 5 to 20 kDa were synthesized from monomers such as 2-methyl-, 2-ethyl-, 2npropyl-, and 2-isopropyl-2-oxazoline. A grafting-to strategy was used to functionalize oxidized CNC and CNF with amineterminated POx, which was achieved through an EDC/NHS coupling reaction, enabling covalent bonding via amide formation. The resulting nanocomposites formed stable dispersions with a modulated phase transition, depending on the hydrophobicity of the POx chain, as well as the concentration and molar mass, achieving values ranging from 26°C to 45°C. Hydrogels containing only CNC-POx, CNF-POx, or mixtures of CNC-POx and CNF were also prepared through physical crosslinking with CaCl2. Future research will focus on optimizing the rheological and mechanical properties of the hydrogel and the biocompatible profile of these materials will be assessed for possible biomedical applications.

The physicochemical properties of cellulose nanocrystals (CNC), combined with the luminescent behavior of lanthanide ions (Ln3+), have been proving their potential role in the development of new functional materials. In previous studies conducted by our group, Eu3+ and Tb3+-CNC doped films resulted in iridescent materials with interesting characteristics and luminescent properties. Photoluminescence studies have shown that the emission band intensity depends on the angle of incidence of the light beam used, an effect attributed to the broad reflection band of the films, which overlaps with the excitation band of the matrix/Ln3+. The excitation wavelengths were selectively influenced by the film preparation method and its cholesteric pitch, emphasizing the material's utility in optical systems. In this way, the main point of interest lies in the ability to control the cholesteric pitch and reflectance band of the iridescent CNC-doped films, which directly affects the selection of the excitation wavelength and, consequently, the material's emission, as already observed for the Tb3+ ion. Through this approach, a new perspective is being explored on the circularly polarized luminescence (CPL) of this type of material, presenting an innovative potential application in polarized luminescence of CNC-based optical devices. Overall, studies are being carried out to control the cholesteric structure pitch to better understand how this structure influences the spectroscopy of others Ln3+ and their CPL. Along Ln3+ ions, which are fundamental for CPL due to their potentially high asymmetry factors, the incorporation of plasmonic nanorods (such as gold or silver) into this system may further expand the applications. The development of CNC-based cholesteric materials could enable efficient chiral induction of the circularly polarized emission of Ln3+ ions by guiding plasmonic nanoparticles through the self-organization of CNC and intensifying the near-field electric effect on the doped ions.

# CELLULOSE NANOMATERIALS AS RHEOLOGICAL ADDITIVES FOR COSMETIC LAMELLAR FORMULATIONS

Authors: SABINO, C.M.S.1; GENTILE, L.2; FERREIRA, G.A.3; LOH, W.1

Institution: <sup>1</sup>University of Campinas, Campinas, Brazil <sup>2</sup>University of Bari, Bari, Italy <sup>3</sup>Federal University of Bahia, Salvador, Brazil

Petroleum-based polymers are commonly used as additives to tailor specific properties or functionalities in formulations. However, bio-based and biodegradable polymers such as polysaccharides, including nanocelluloses, have been proposed as more sustainable alternatives. In this study, we investigated the impact of incorporating cellulose nanocrystals (CNC) and cellulose nanofibrils (CNF) into aqueous surfactant phases widely used in cosmetic applications, particularly the lyotropic lamellar phase. While CNC and CNF carry negatively charged surface groups, the lamellar phase is formed by a cationic surfactant, double-chain dioctadecyldimethylammonium chloride (DODAC). Rheological changes were correlated with the system's internal organization using a multiscale approach involving scattering, calorimetry, and microscopy techniques. For samples containing CNC, the yield value increased with CNC concentration up to a maximum, beyond which it decreased. In contrast, samples with CNF and CMC at the same surfactant and additive concentrations showed a continuous decrease in yield value with increasing additive content. Small-Angle X-ray Scattering (SAXS) revealed similar trends: in CNC-containing systems, the bilayer repeat distance (d-spacing) decreased with CNC concentration until reaching a minimum, then increased thereafter. Conversely, in CNF and CMC samples, the d-spacing consistently decreased with additive concentration. Small-Angle Neutron Scattering (SANS) with contrast matching suggested that cellulose can be confined within the aqueous regions between bilayers. Cryogenic Transmission Electron Microscopy (Cryo-TEM) and Fourier-Transform Infrared Microscopy (FTIR) confirmed the integration of nanocelluloses into the lamellar matrix, revealing both micro- and nano-scale aggregates. Furthermore, tribological measurements demonstrated enhanced lubrication properties in formulations containing CNC and CMC. These findings suggest that both the rigidity and structural characteristics of the lamellar phase are influenced by the surfactant/additive mass ratio and the type of cellulose additive—CNC, CNF, or CMC. Overall, this work highlights the potential of cellulose materials to effectively tailor the rheological behavior of cosmetic formulations, providing a sustainable alternative to petroleum-derived polymers.

# CHARACTERIZATION OF ENZYMES FOR CELLULOSE NANOCRYSTAL PRODUCTION

Authors: BASSO,  $M.^1$ ; DIAS,  $I.^2$ ; VELASCO,  $J.^3$ ; OLIVA,  $B.^2$ ; MERCADO,  $M.^2$ ; SEGATO,  $F.^2$ ; ARANTES,  $V.^2$ 

Institution: <sup>1</sup>Department of Biotechnology, Lorena School of Engineering, University of São Paulo (USP); Center for Natural and Human Sciences, Federal University of ABC, Santo André, SP, Brazil

<sup>2</sup>Department of Biotechnology, Lorena School of Engineering, University of São Paulo (USP)., Lorena, SP, Brazil <sup>3</sup>Department of Biotechnology, Lorena School of Engineering, University of São Paulo (USP); Department of Biological Sciences, Universidad de los Andes., Bogotá, Colombia

The enzymatic production of cellulose nanocrystals (CNCs) offers several advantages over the acid hydrolysis route, including milder reaction conditions, absence of chemical waste, and the possibility of recovering soluble sugars. However, selecting appropriate enzymes and optimizing hydrolysis conditions remain challenging due to the unique properties of each enzyme, which are primarily determined by their amino acid sequence and three-dimensional structure. To assess cellulase performance, several experimental methods have been developed, typically based on model substrates. These methods are generally effective for evaluating enzymatic saccharification, as they correlate well with sugar release from cellulosic biomass. Nonetheless, their relevance for CNC production is questionable. For example, activity on carboxymethyl cellulose (CMC), a standard assay for amorphous cellulose hydrolysis, is often used to estimate enzyme loading for CNC isolation. This approach assumes that removing non-crystalline regions is sufficient to yield CNCs. However, our results, based on studies involving enzymes with distinct structural and biochemical properties, demonstrate that CMC activity is a poor predictor of enzyme efficiency in CNC production. This discrepancy likely arises from the fact that CMC does not replicate the insoluble and recalcitrant structure of native cellulose, which significantly restricts enzyme accessibility during hydrolysis. To overcome this limitation, we propose a novel screening method specifically tailored to identify enzymes better suited CNC production.

# ENERGY-EFFICIENT ENZYMATIC PLATFORM FOR THE TUNABLE PRODUCTION OF CELLULOSE NANOSPHERES AND OTHER NANOCELLULOSE STRUCTURES

Authors: YUPANQUI-MENDOZA, S.L.1; ARANTES, V.1

Institution: <sup>1</sup>Universidade de São Paulo, Lorena, Brasil

Cellulose nanospheres (CNS) represent a new category of nanocellulose, distinct from cellulose nanocrystals (CNC) and cellulose nanofibrils (CNF), with unique structural and functional properties [1]. In this study, we present an enzymatic hydrolysisbased approach to efficiently produce CNS and other types of nanocellulose by simply adjusting the hydrolysis conditions. The optimized process for CNS production employed endoglucanase and xylanase on cellulose fibers pretreated through mechanical ultra-refining and hydrodynamic cavitation. Optimization, conducted through a statistical experimental design, enabled a high CNS yield of 82.7% with precise control over its diameter (20-100 nm). Critical factors included specific surface area (SSA), enzyme loading, reaction time, and solids content, with SSA being the most influential, as no CNS was obtained below 350 m<sup>2</sup>/Kg. Furthermore, by modifying the hydrolysis conditions, it was possible to achieve controlled production of CNC, CNF, and hybrid CNC/CNF suspensions with tunable properties. The optimal conditions (hydrolysis time = 38 h, enzyme loading = 300 U/g, enzymatic ratio EG:EX = 7:1, SSA = 500 m<sup>2</sup>/kg at 1 wt%) allowed for the energy-efficient production of CNS, with an energy consumption of just 39.36 kWh/Kg—significantly lower than conventional nanocellulose production methods [2]. This flexible, low-energy platform not only facilitates scalable, tailormade nanocellulose production but also reduces operational and investment costs, offering a sustainable and efficient alternative for various industrial applications.

# EXPLORING THE POTENTIAL OF CELLULOSE AUTOFLUORESCENCE FOR OPTICAL DETECTION OF TANNIN IN RED WINES

Authors: TEODORO, K.B.R. $^1$ ; SILVA, M.J. $^2$ ; ANDRE, R.S. $^3$ ; SCHNEIDER, R. $^4$ ; MARTINS, M.A. $^1$ ; MATTOSO, L.H.C. $^1$ ; CORREA, D.S. $^1$ 

Institution: ¹Nanotechnology National Laboratory for Agriculture, Embrapa Instrumentation, Sao Carlos - SP, Brazil

<sup>2</sup>PPGQ, Federal University of São Carlos (UFSCar), Sao Carlos - SP, Brazil

<sup>3</sup>Scientific and Technological Institute of Brazil University, Sao Paulo - SP Brazil

<sup>4</sup>ICGM - Institut Charles Gerhardt Montpellier - Institut de Chimie Mo

The growing demand for optoelectronic devices has oppened up opportunities for using cellulosic (nano)materials in sensors and biosensors. Cellulose is typically employed as a support or substrate for sensing materials, while its autofluorescence remains largely unexplored. Although its molecular structure lacks aromatic groups or  $\pi$ -conjugation, typically prerequisite required for photoluminescence (PL), the observed autofluorescence is hypothesized from clustering-triggered emission phenomenon. This theory proposes that electron-rich moieties, interact spatially to reduce energy gaps and enhance electron delocalization, leading to emission, which is favored in aggregated states in carbohydrates, like cellulose, due to their abundance of oxygen groups and the strong intermolecular hydrogen bonding between them. Herein, we present a novel approach that leverages the PL of pristine cellulose to detect tannins in red wine. For this, cellulose nanocrystals (CNC), extracted from wood through enzymatic hydrolysis were firtly characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction and termogravimetry (TGA), in order to identify the absence of compounds which could influence the cellulose autofluorescence, like lignin. By adjusting the experimental setup. PL emission bands were analyzed across three spectral regions: 300 - 400 nm, 400 - 500 nm, and 550 - 700 nm. The presence of tannins affected emissions in all three regions, with significant quenching specifically over 300 - 400 nm range. The proposed mechanism suggests the occurance of dynamic quenching, without ground-state complex formation. According clusteroluminescence theory, tannins disrupt interparticle interactions between CNC, causing reduction in PL intensity. The sensing platform responded selectively to tannins in wines in a dynamic detection range of 10 to 1060 µg mL-1. It provided a limit of detection (LoD) of 6.1 µg mL-1 and achieved recovery rates of 99.7% and 95.3% for Cabernet Sauvignon and Tannat wines, respectively, highlighting its potential for monitoring tannic acids in beverages and food products.

# FROM AGRICULTURAL WASTE TO ADVANCED MATERIALS: CNC AND CNF PRODUCTION FROM PINEAPPLE LEAF FIBERS

Authors: COSTA, A.T.F.1; DUFRESNE, A.2; SONG, T.3; PARRA, F.D.1

Institution: <sup>1</sup>Nuclear and Energy Research Institute, IPEN-CNEN/SP, São Paulo, Brazil

<sup>2</sup>LGP2, Université Grenoble Alpes (UGA), Gières, France <sup>3</sup>State Key Laboratory of Pulp and Paper Engineering, South China University of Technology, Guangzhou, China

Pineapple leaf fibers (PALF), a lignocellulosic byproduct of pineapple cultivation, are often discarded or burned after harvest, contributing to environmental waste. However, PALF is rich in α-cellulose, making it an ideal candidate for producing cellulose nanofibrils (CNF) and cellulose nanocrystals (CNC), which are valuable reinforcements in polymer composites. This study explores the extraction of CNF and CNC from PALF using an integrated approach involving alkalization, bleaching, sulfuric acid hydrolysis for CNC and ball milling for CNF production. The influence of processing parameters on the properties of the resulting nanomaterials was systematically investigated to optimize their production. Chemical analysis revealed a high  $\alpha$ cellulose content (78.14%), confirming PALF's suitability for nanocellulose extraction. Morphological and structural characterization through XRD, AFM, FTIR, and TGA demonstrated successful isolation of CNC and CNF with desirable properties. CNC exhibited a crystallinity index of 78.5% and nanoscale dimensions (647-1105 nm), while CNF displayed lengths of approximately 256 nm. Thermal analysis indicated that CNC had lower thermal stability due to reduced molecular weight and sulfate group introduction. Optimized conditions of acid hydrolysis (55% H<sub>2</sub>SO<sub>4</sub>, 45°C, 30 min) yielded CNC with superior characteristics, highlighting PALF's potential as a sustainable resource for advanced applications such as biocomposites, drug delivery systems, and tissue engineering.

# FROM NATURE TO MATERIALS: CELLULOSE NANOFIBRILS AND NANOCRYSTALS APPLIED TO THE PRODUCTION OF FILMS, GELS, AND LIGHTWEIGHT MATERIALS

Authors: REZENDE, C.A.1

Institution: ¹LaQuiMoBio/ Institute of Chemistry - Unicamp, Campinas, Brazil

In this lecture, I will present some of the work currently being developed at LaQuiMoBio, the Laboratory of Chemistry and Morphology of Biomass, which I have coordinated at the Institute of Chemistry at Unicamp since 2012. Currently, the group main focus is on the extraction of cellulose and lignin from plant biomasses, aiming at the production of cellulose fibers, nanocrystals, nanofibrils, and nanolignin, as well as the study of their applications in polymer composites and nanocomposites, coating films, hydrogels, and lightweight materials. Starting from different types of plant biomasses (e.g., sugarcane bagasse, eucalyptus pulp, or elephant grass), we use various pretreatment and isolation methods to obtain nanostructures with morphological, chemical, and structural characteristics better suited for different applications. The results presented will focus mainly on films, gels, and sorbents prepared from nanocellulose.

# HYDROGELS AND ELECTROSPUN POLYMERIC NANOFIBERS CONTAINING NANOWHISKERS AND THEIR POTENTIAL TECHNOLOGICAL APPLICATIONS

# IONICALLY PLASTICIZED CELLULOSE-BASED POLYELECTROLYTE COMPLEX FILMS

Authors: MUNIZ, E.C.1

Authors: SILVA, V.G.H.1; LOH, W.1; OTONI, C.1

Institution: <sup>1</sup>Chemistry Dept., State University of Maringá (UEM), Maringá, Brazil

Institution: 1Unicamp, Campinas, Brazil

This presentation will discuss some results obtained by our research groups in the development of polymeric materials containing nanowhiskers (cellulose, chitin, etc.), with potential applications in the medical, pharmaceutical, and tissue engineering fields. Two classes of polymeric materials will be emphasized: hydrogels and electrospun nanofibers. These materials have potential applications mainly for: i) controlled drug release [1,2]; ii) bactericidal action [3,4]; iii) dye adsorption [5]. Various methodologies for obtaining matrices with different geometries (cylindrical, spherical, irregular particles, thin films, nanofibers, etc.) at different scales (macro, micro, and nanometric) will be demonstrated and discussed. Relevant information about INCT-Polysaccharides will also be presented, along with a brief report on research related to nanowhiskers developed by INCT-Polysaccharides members.

Saloplastics are materials derived from polyelectrolyte complexes (PECs) with reversible ionic cross-links, whose plasticity can be triggered by exposure to concentrated saline solutions. Salt ions compete with interpolymer ionic interactions, replacing intrinsic cross-links with extrinsic ones, which increases chain mobility and allows the material to be reshaped. The use of polysaccharides such as chitosan, carboxymethylcellulose (CMC), and cellulose nanocrystals (CNC) enables the fabrication of biodegradable saloplastics that are responsive to both ionic strength and pH, due to the tunability of electrostatic interactions among the building blocks. In this study, saloplastic films were prepared via solvent casting of polyelectrolyte complexes (PECs) composed of high-molecular-weight chitosan and CMC and/or sulfated CNC. The Mechanical properties were tuned by exposure to saline solutions of varying concentrations, humidity control, and the addition of polyethylene glycol (PEG). The viscoelastic behavior was evaluated using dynamic mechanical analysis (DMA) as a function of temperature. Tensile tests were performed to determine Young's modulus and ultimate tensile strength. The results showed an increase in the viscous moduli (E") following salt-induced softening, confirming effective film plasticization. Furthermore, the films demonstrated reprocessability by redispersion in water and re-drying, with retention of their mechanical properties.

**CELLULOSE** 

ON

# LIGNOCELLULOSE-BASED NANOSTRUCTURED MULTILAYERS FOR SUSTAINABLE PACKAGING

NANOCRYSTALS FOR 3D PRINTING

INKS

LUMINESCENT

Authors: ACOSTA, T.L.N.<sup>1</sup>; APARíCIO, R.R.<sup>1</sup>; SILVA, W.D.<sup>1</sup>; DIAS, I.A.<sup>1</sup>; GRETTER, C.P.<sup>1</sup>; MUNIZ, G.I.B.<sup>1</sup>; NISGOSKI, S.<sup>1</sup>; CADERMATORI, P.H.G.<sup>1</sup>

Authors: GOMES, R.C.<sup>1</sup>; CAIUT, A.J.M.<sup>1</sup>; SANTOS, V.M.<sup>1</sup>; TORRES, R.F.<sup>1</sup>; BARUD, S.H.<sup>2</sup>

RASED

Institution: <sup>1</sup>UFPR, Sao Jose dos Pinhais, Brazil

Institution: <sup>1</sup>Universidade de São Paulo, Ribeirão Preto, Brasil <sup>2</sup>Universidade de Araraquara, Araraquara, Brasil

Due to environmental regulations and sustainability demands, biobased alternatives replace petroleum-based plastics in packaging. Lignocellulosic resources like Kraft lignin provide a sustainable strategy with antioxidant activity, UV protection, and water repellency for an eco-friendly market. This study aimed to develop nanostructured multilayer films composed of cellulose nanofibrils (CNF), cellulose nanocrystals (CNC), and Kraft lignin (KL), targeting applications in active and sustainable packaging. Additionally, it investigated the use of cold plasma treatment to enhance the interaction between the layers. The initial films (CNF-CNC) were prepared using a layer-by-layer casting method. Two additional strategies were employed: (i) the incorporation of KL via dip coating (CNF-CNC+KL), and (ii) surface modification using cold plasma in a diffuse coplanar surface barrier discharge (DCSBD) system, followed by KL dip coating (CNF-CNC+P+KL). Rheological characterization of the CNF-CNC solution revealed gel-like behavior, shear-thinning properties, and high water retention, indicating strong film-forming ability. Oscillatory tests showed the storage modulus (G') exceeded the loss modulus (G") across frequencies, confirming viscoelastic formation network. The scanning electron microscopy analysis of film crosssections confirmed a uniform structure without delamination, showing interlayer adhesion. It also revealed lignin coverage on the surface after deposition. Plasma-treated samples had smoother, compact surfaces with fewer cracks, indicating better compatibility and interfacial interactions. The water contact angle (WCA) of uncoated CNF-CNC films was very hydrophilic. After adding KL, hydrophobicity increased, especially in plasma-treated films (CNF-CNC+P+KL), which exhibited WCA values ranging from 69.9° to 73.6°. Plasmainduced surface activation improved lignin anchoring and decreased hydrophilic exposure groups. The stable WCA in plasma-treated films indicates uniform lignin distribution on the surface, improving their water barrier effectiveness. These findings highlight the potential of cold plasma treatment as an effective strategy to enhance the surface hydrophobicity and functional performance of biobased films for active packaging applications.

Bioprinting is a layer-by-layer fabrication technique used to produce three-dimensional structures from functional components, including biological and biochemical materials. This technology has gained prominence, particularly in medicine and tissue engineering. However, commercially available bioprinters are typically expensive, and effective printing relies on hydrogels or inks with optimal chemical and physical properties. As a cost-effective alternative, conventional 3D printers can be adapted for bioprinting applications. In this study, a low-cost homemade bioprinter based on microextrusion technology was developed by modifying a Graber i3 fused deposition modeling (FDM) 3D printer. Additionally, cellulose nanocrystal (CNC) suspensions combined with GPTMS were developed as functional inks, employing in situ photopolymerization to fabricate solid nanostructured objects exhibiting chiral nematic ordering, birefringence. . Preliminary results showed birefringence in CNC suspensions between crossed polarizers, confirming their liquid crystalline behavior. CNC/GPTMS composite films analyzed by UV-Vis, FTIR, TGA, and SEM demonstrated helical ordering with periodic layering and structural coloration. Obiects printed using CNC/GPTMS inks retained birefringence, confirming structural integrity postprinting. These findings highlight the potential of CNC-based inks for developing advanced optical materials applicable in sensors, photonics, and bioengineering. Future research includes doping these materials with lanthanide ion nanoparticles to explore their luminescent properties.

**SORBENT** 

**BASED-CELLULOSE** 

FOR

THE

# NANOCELLULOSE AND GELATIN-BASED HYDROGELS FOR THE SURFACE CLEANING OF ARTWORKS

PRECONCENTRATION OF PAHS IN WATER SAMPLES

NANOCRYSTALS AS

**NANOCOMPOSITE** 

Authors: SILVA, A.I. $^{\rm 1}$  ; CAMARGOS, H.M.C. $^{\rm 2}$  ; SCOPEL, E. $^{\rm 3}$  ; REZENDE, A.C. $^{\rm 1}$ 

Authors: REIS, L.  $^{1}$  ; DIAS, I.  $^{2}$  ; ARANTES, V.  $^{2}$  ; LUCAS, E.  $^{1}$  ; SOARES, B.  $^{1}$ 

Α

Institution: <sup>1</sup> UNICAMP, Campinas, Brasil <sup>2</sup>UFMG, Belo Horizonte, Brazil <sup>3</sup>UBC, Vancouver, Canada

Institution: <sup>1</sup>Universidade Federal do Rio de Janeiro, Rio de Janeiro, Brazil

<sup>2</sup>Universidade de São Paulo, Lorena, Brazil

Cleaning procedures are essential for preserving cultural heritage objects, but conventional methods using aqueous or organic solvents can damage original materials and expose restorers to toxic vapors. Gels offer a more controlled cleaning approach due to their high solvent retention, but most are based on non-biodegradable synthetic polymers. This research converts cellulose fibers obtained from sugarcane bagasse into cellulose nanofibrils (CNF) and chemically crosslinking them with gelatin using N-(3-dimethylaminopropyl)-N'-ethylcarbodiimide (EDC) and N-hydroxysuccinimide (NHS). To investigate the effects of CNF and gelatin on hydrogel properties and determine the most suitable formulation for cleaning, gels with different gelatin:CNF weight ratios were prepared: 50:50 (G50-EDC and G50-EDCNHS), 25:75 (G25-EDC), and 75:25 (G75- EDC). All the formulations were prepared with 2% wt. of total solid content. Among the G50 formulations, G50-EDCNHS showed a lower gel fraction and higher swelling, suggesting reduced crosslinking. G75-EDC showed mass loss due to gelatin leaching in water, whereas G25-EDC demonstrated the highest gel fraction and swelling capacity, attributed to strong CNF-water interactions and intrinsic physical entanglement. In the compression test, G50-EDC was the stiffest, indicating a higher crosslinking degree. G25-EDC and G75-EDC exhibited lower resistance, indicating that very high proportions of either gelatin or CNF can compromise mechanical strength. The most promising formulations for cleaning are G50-EDC and G50- EDCNHS. The hydrogels were applied in cleaning tests for the removal of artificial soil and varnish (Paraloid B72), loaded with differents cleaning fluids (water, triammonium citrate, and commercial microemulsions). They maintained mechanical integrity during application. Ongoing studies are evaluating morphology and cleaning efficiency. This study may contribute to the preservation of tangible cultural heritage and promotes positive economic and environmental impacts by using agricultural waste to produce high-value-added materials.

Nowadays, water contamination is an extremely important issue and its decontamination and care are everyone's job. Companies, governments, communities and individuals are concerned about access to quality water and are looking for ways to mitigate the spread of contamination of water bodies used as drinking water supplies. Therefore, this work aimed to offer the scientific community a new sorbent basedcellulose nanocrystals (CNCs) for the preconcentration of three target polycyclic aromatic hydrocarbons (PAHs), naphthalene, fluorene and pyrene, organic pollutants classified as high priority chemicals (EPA, 2019; WHO 2019) and found in a several types of water samples. For this proposal a study was carried out about the synthesis of a magnetic nanocomposite based on CNCs as an sorbent for the preconcentration technique magnetic solid phase extraction (MSPE) prior to determination by gas chromatograph coupled to a flame ionization detector (GC-FID). The synthesis method via electrostatic attraction between Fe2O3 nanoparticles and CNCs was optimized means of the classic method and the MSPE technique was optimized means of a Plackett-Burman design. The nanocomposite based-CNCs, synthesized under optimized experimental conditions, was characterized by scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDX) and Fourier transform infrared spectroscopy (FTIR). The nanocomposite that showed the highest adsorption capacity for the studied PAHs was the CNCs/Fe2O3 3/1. The optimized extraction conditions were: amount of CNCs/Fe2O3, 20 mg; extraction time, 10 min; type of eluent, hexane; volume of eluent, 0.5 mL; desorption time, 5 min; volume of sample, 10 mL; pH of sample, 5; and no NaCl addition. The preconcentration factors obtained for naphthalene, fluorene and pyrene were 2, 4 and 7, respectively.

# OPTIMIZATION OF CELLULOSE NANOCRYSTAL PRODUCTION PROCESS FOR RHEOLOGICAL APPLICATIONS IN TISSUE ENGINEERING BIOINKS

Authors: SILVA, L.L.C.1; ARANTES, V.1

Institution: <sup>1</sup>University of São Paulo, Lorena, Brazil

# PILOT SCALE CO-PRODUCTION OF SHORT-CHAIN XYLOOLIGOSACCHARIDES AND NANOCELLULOSE WITHIN BIOREFINERY CONCEPT

Authors: PEREIRA, B.  $^{1}$  ; MARQUES, L.B.M.  $^{1}$  ; MARCONDES, W.F.  $^{1}$  ; ARANTES, V.  $^{1}$ 

Institution: <sup>1</sup>Escola de Engenharia de Lorena -USP, Lorena, Brazil

Cellulose nanocrystals (CNCs) are high-value nanomaterials with expanding applications in fields such as packaging, composites, and tissue engineering. Traditionally obtained via sulfuric acid hydrolysis, their production still presents technical, environmental, and economic challenges. As an alternative, enzymatic hydrolysis, preceded by mechanical pretreatment of cellulose pulp, emerges as a promising route. Moreover, the enzymatic pathway enables greater control over the physicochemical properties of CNCs, expanding their potential for biomedical applications. This study aimed to develop and apply an optimized enzymatic route for CNC production, with a focus on technical feasibility and use in tissue biofabrication. Initially, a statistical experimental design was employed to optimize enzymatic hydrolysis, considering variables such as particle size, solid concentration, enzyme loading and hydrolysis time. The results demonstrated high yields (up to 65.5%), along with reduced energy consumption and enzyme demand. In the subsequent stage, the functional application of CNCs in tissue biofabrication via 3D printing was investigated. One of the main challenges in extrusion-based 3D bioprinting is the development of bioinks with rheological properties compatible with high cell viability. In this context, CNCs acted as rheological modifiers in polysaccharide-based hydrogels, providing shear-thinning behavior and enhancing the mechanical stability of the printed structures. Therefore, this work integrates an optimized CNC production process with its strategic application in advanced systems for three-dimensional bioprinting.

The transition towards a sustainable circular bioeconomy relies heavily on the comprehensive valorization of lignocellulosic biomass through integrated biorefinery approaches. These strategies aim to convert all major biomass components into a portfolio of value-added products, enhancing economic viability and environmental sustainability. Building on previous research that established a process for obtaining cellulose nanocrystal (CNC), cellulose nanofibers (CNF), nanolignin, and xylooligosaccharides (XOS) from lignocellulosic sources where only XOS production was validated at pilot scale—this study details significant advancements. While prior work demonstrated multi-product potential, nanocellulose and nanolignin fractions were obtained only at laboratory scale. This investigation focuses on advancing this biorefinery model through pilot-scale co-production of high-value shortchain xylooligosaccharides (SC-XOS) and nanocellulose (NC), alongside a lignin-rich stream. A key emphasis is the practical application of biorefinery products, with hemicellulosic hydrolysate rich in SC-XOS currently under evaluation as poultry feed additive. The integrated process began with steam explosion of biomass at pilot scale for selective hemicellulose extraction, yielding SC-XOS-rich hydrolysate. After SC-XOS recovery, the lignocellulosic material underwent delignification, producing a lignin-rich black liquor (precursor for nanolignin) and purified cellulosic pulp. This pulp was subjected to enzymatic hydrolysis at pilot scale to produce NC. Pilot-scale operations successfully yielded SC-XOS suitable for chicken feed evaluation, highlighting our group's commitment to bridging bioproduct development with applications. Concurrently, enzymatic hydrolysis of the delignified pulp generated high-quality nanocellulose. The delignification process also yielded a lignin-rich stream, available for further valorization into nanolignin or other lignin-based chemicals. This study demonstrates major progress in the implementation of integrated lignocellulosic biorefineries. The successful co-production of SC-XOS for feed, nanocellulose, and a lignin co-product underscores the technical feasibility and economic potential of holistic valorization strategies. This application-driven approach accelerates scalable biomaterials and biochemicals toward industrial relevance, contributing to a circular bioeconomy.

# RHEOLOGICAL CHARACTERIZATION OF 3D-PRINTABLE CNC-BIOPOLYMER HYDROGELS FOR SMART WOUND HEALING APPLICATION

Authors: ESPER, L.B.1; SANTOS, M.V.2; FONTES, M.L.3; BARUD, H.S.1

Institution: <sup>1</sup>University of Araraquara (UNIARA), Araraquara, Brasil <sup>2</sup>State University of Campinas (UNICAMP), Campinas, Brasil <sup>3</sup>Federal University of São Carlos (UFSCar), São Carlos, Brasil

Current wound care therapies incur significant costs, especially for chronic wounds linked to conditions like diabetes. In this context, this study presents the development of a stimuliresponsive 3D-printed hydrogel based on cellulose nanocrystals (CNC), hyaluronic acid (HA), and silk fibroin (SF), designed to promote wound healing and enable real-time detection of pH and glucose levels. The formulations were prepared by mixing different proportions of CNC, HA, and SF at room temperature using a SpeedMixer homogenizer to ensure uniformity. Rheological analyses showed that all formulations exhibited shearthinning (pseudoplastic) behavior, essential for extrusionbased 3D printing. Formulations with 20% CNC displayed high initial viscosities (6366 Pa·s at 0.1 s-1) and elevated storage (G' 21872 Pa) and loss (G" 1798 Pa) moduli, which decreased with increasing shear rates (0.1 to 1000 s<sup>-1</sup>). The addition of HA and SF further increased the initial viscosity by 42%, but these formulations exhibited a more pronounced viscosity reduction under shear, likely due to polymer chain disruption. Amplitude sweep tests indicated that HA and SF enhanced the linear viscoelastic region (LVR), improving material stability under deformation. Additionally, incorporating HA and SF increased both storage (G' 49799 Pa) and loss (G" 7547 Pa) moduli at low strain, demonstrating superior elastic and viscous properties. Frequency sweep tests revealed that these formulations maintained a stable G' across the 0.1 to 100 Hz range, with G' consistently higher than G", confirming a solid-like, viscoelastic gel structure. The hydrogel scaffolds were successfully printed at 25 °C using a 3D Educational Starter™ printer. The material showed excellent printability, forming continuous, uniform filaments with good shape fidelity and structural integrity. Future perspectives include the functionalization of the hydrogel with pH-sensitive indicators and glucosemonitoring enzymes, followed by comprehensive in vitro evaluation of cytocompatibility and detailed physicochemical characterization, aiming to create an intelligent dressing for chronic wound management.

# A DECADE OF APPLICATION DEVELOPMENT IN NANOFIBRILLATED CELLULOSE: PERFORMANCE BIOFILAMENTS' JOURNEY INTO CONCRETE AND MINING APPLICATIONS

ALL-CELLULOSE COATINGS FOR LOW-GRAMMAGE PAPER

Authors: GOURLAY, K.1

Institution: <sup>1</sup>Performance BioFilaments; Vancouver, Canadá

Institution: <sup>1</sup>Universidade de São Paulo, São Carlos, Brasil

Authors: FLORES, M.F.1; CURVELO, A.A.S.1

Since 2014 Performance BioFilaments Inc (PBI) has been producing nanofibrillated cellulose through the mechanical refining of Northern Bleached Softwood Kraft pulp (NBSK). This presentation will cover the story of PBI's application development journey, from the early days working on a wide variety of low-Technology Readiness Level (TRL) applications, to its current position as a lean and focused company targeting a small number of high-value high-TRL applications. A review of some of the key pieces of data that steered PBI towards its current application areas will be presented, along with highlighting some milestone moments along the way.

The development of sustainable materials for surface treatment has gained growing attention in recent years, especially in the context of replacing petroleum-based products. In the paper industry, coating processes are essential to improve surface properties by filling the spaces between cellulosic fibers with functional agents such as pigments, binders, and optical brighteners. In this context, nanocellulose - particularly cellulose nanocrystals (CNCs) and cellulose nanofibers (CNFs) - has emerged as a promising alternative due to its biodegradability, abundance, and ability to form robust hydrogen-bonded networks. Furthermore, the concept of all-cellulose composites (ACCs), which utilize both regenerated and native cellulose phases, opens new pathways for the design of high-performance, fully bio-based coating systems. In this work, from a CNF dispersion and from a cellulose xanthate solution, all-cellulose materials were produced by immersing low-grammage cellulose sheets (10 g/m2) into these systems, allowing the spaces among fibers to be filled either by nanofibers or by a regenerated cellulose matrix. The resulting material from CNF impregnation was opaque, whereas the regenerated cellulosebased material was translucent to nearly transparent. Scanning electron microscopy revealed superior interfacial adhesion in the regenerated cellulose samples, with a continuous matrix phase homogeneously permeating the fiber network of the paper. Mechanical tensile tests are planned in the short term to further investigate the influence of the different coating approaches on the elastic modulus and elongation behavior of the resulting materials.

#### ARGININE-MEDIATED REDISPERSION OF TEMPO-OXIDIZED CELLULOSE NANOFIBRILS FOR SCALABLE COLLOIDAL GLASS APPLICATIONS

Authors: NASCIMENTO, M.D. $^1$ ; PORTUGAL, V.R. $^1$ ; BERNARDES, S.J. $^1$ ; LIMA, R. $^1$ 

Institution: 1LNNano, Campinas, Brazil

The high-water content (~99%) of TEMPO-oxidized cellulose nanofibril (TOCNF) suspensions presents a significant barrier to transportation and large-scale processing, due to increased logistical costs and challenges in handling. However, drying TOCNFs often leads to hornification, which hinders their redispersion and reuse. In this study, we investigated the interaction between TOCNFs and arginine (ARG), a known protein aggregation inhibitor, as a strategy to enhance redispersion postdrying. At neutral pH, ARG adsorption onto TOCNF surfaces led to mild charge screening, as evidenced by zeta potential measurements and atomic force spectroscopy (AFM-FS). Despite the presence of excess ARG, nanofibril aggregation was prevented, and repulsive inter-fibrillar interactions were maintained ( $\zeta$ -potential > -25 mV), enabling the formation of a colloidal glass at 0.5 wt% TOCNF with enhanced viscosity and elastic modulus. Upon redispersion in ARG-rich medium, steric repulsion and reduced TOCNF adhesion facilitated nanofibril recovery. The redispersed system retained its original rheological properties, and cryogenic transmission electron microscopy (cryo-TEM) confirmed the return of nanofibrils to the nanoscale. This work presents a promising approach to scale up TOCNF applications by preserving their nanostructure and colloidal behavior after redispersion.

# CASE STUDY: INFLUENCE OF NANOCELLULOSE ON MASS VARIABILITY IN 3D-PRINTED PLA/NC COMPOSITES

Authors: OLIVEIRA, V.  $^1$ ; HORIUCHI, L.  $^1$ ; SOUZA, M.  $^1$ ; GONÇALVES, A.P.  $^1$ ; POLKOWSKI, R.  $^1$ 

Institution: <sup>1</sup>TRL9 TECH Testing and Technical Analysis, Salvador, Brazil

3D printing is a method of producing customizable and highperformance parts, production of test specimens for evaluating new materials, among other applications. The most common printing process is fused filament fabrication (FFF), which deposits molten material in predefined paths for the production of the part [1]. The rheology of the polymer plays an important factor for the quality of the final material, a property also known as printability. Poly (lactic acid), being of plant origin, serves as a sustainable alternative to the use of other non-renewable polymers. However, it does not have comparable mechanical strength [2]. Nanocellulose is obtained from the processing of plant sources and is capable of increasing the final strength of PLA [3]. However, printability can be expressively altered when adding a nanofiller, and the literature still lacks quality criteria for evaluating printed parts. This article aims to study the variability of the mass of 3Dprinted composite specimens as a quality criterion for printability. For this purpose, tensile and impact test specimens were produced following the ASTM D638 and ASTM D256 standards, respectively, and the Tukey test was used to assess whether there was a statistically significant difference between the compositions. Five specimens were 3D-printed with the pure material and for each nanocomposite's composition. Formulations with pure PLA (F1) and with 0.5 wt. (F2), 1.0 wt. (F3), and 2.0 wt.% of nanocellulose (F4) were tested. When evaluating the nanocomposite impact samples, the average masses of all compositions had no statistical significance when compared to the pure polymer. Regarding the tensile test specimens, a statistical difference was observed in the average masses of F2 and F4 when compared to the reference polymer. This difference requires careful consideration to distinguish problems arising from the 3D printing process itself versus the addition of the nanofiller.

### CHEMICAL FUNCTIONALIZATION OF CELLULOSE NANOFIBRILS WITH ORGANIC HALIDE

Authors: BARROS, J.M.F.  $^1$ ; PEREIRA, V.R.  $^2$ ; MENEZES, A.J.  $^2$ ; DUFRESNE, A.  $^3$ 

Institution: <sup>1</sup>UFSCar-So, Sorocaba, São Paulo <sup>2</sup>UFRJ, Macaé, Brazil <sup>3</sup>Grenoble INP – Pagora, UGA, Grenoble, France

Given the development of materials derived from biomass, cellulose has stood out as an abundant, low-cost, biodegradable material with excellent mechanical, thermal properties, and a high specific surface area. Its polymeric chain is linear, unbranched, and rich in hydroxyl functional groups, enabling various chemical modifications. Following the methodology proposed by Jakubovic (1960), the present study aims to functionalize cellulose nanofibrils (CNFs) with an organic halide etherifying agent, with the goal of developing filtration membranes for the removal of dyes and heavy metals from aqueous media. The chemical characterization was conducted using Fourier-transform infrared spectroscopy (FTIR), where the appearance of new bands associated with chemical modification was observed when compared to the FTIR spectra of unmodified CNFs. Elemental analysis (EA) confirmed the presence of nitrogen, indicative of amine groups incorporated into the CNFs. X-ray diffraction (XRD) analysis revealed a decrease in crystallinity following the etherification reaction. To support these initial findings, future work will include further chemical characterizations and pollutant removal tests.

# CONTRIBUTIONS OF NANOCELLULOSE ON THE CRYSTALLINITY AND DIMENSIONAL STABILITY OF 3D-PRINTED PLA PARTS

Authors: OLIVEIRA, V. $^1$  ; HORIUCHI, L. $^1$  ; SOUZA, M. $^1$  ; GONÇALVES, A.P.  $^1$  ; POLKOWSKI, R.  $^1$ 

Institution: <sup>1</sup>TRL9 LAB Testing and Technical Analysis, Salvador, Brazil

The study of material performance is of extremely importance to establish their suitability for practical applications. Polylactic acid (PLA) presents interesting characteristics such as biodegradability and biocompatibility, however, its inherently low thermal stability and limited crystallinity restrict its application. The improvement of crystallinity in the PLA is therefore considered a prerequisite for increasing its resistance to thermal deformation. In this sense, several studies have investigated different strategies to improve this in the PLA matrix [1]. Cellulose nanoparticles have received great attention in the areas of materials science and engineering because they possess characteristics such as biodegradability and high mechanical strength [2]. The present study consisted of the elaboration of 3D printed PLA and fibrillated nanocellulose composites, subsequently subjected to thermal annealing under conditions of 75 and 90°C for 2 hours. Based on the crystallinity results obtained through differential scanning calorimetry (DSC) analysis, values of 12.8, 37.6 and 30.4% were obtained, respectively, for the pure PLA samples without treatment; pure PLA after annealing at 75°C for 2 hours and pure PLA after annealing at 90°C for 2 hours. The increase in crystallinity among the samples composed of pure PLA was significant, demonstrating the contribution of annealing. Regarding nanocomposites, the addition of nanocellulose led to a significant increase in crystallinity in the untreated sample when compared with to the control piece. This increase in crystallinity was also observed for the nanocomposites that were subjected to annealing, regardless of the temperature used. This effect may be associated with the ability of cellulose nanostructures to act as a nucleating agent for crystallization in a polymer matrix. From the crystallinity results and the observation of dimensional behavior,

### CONTROLLING NANOCELLULOSE (DIS)ASSEMBLY VIA THE SUPRAMOLECULAR TOOLBOX

Authors: OTONI, C.1

Institution: <sup>1</sup>Institute of Chemistry, University of Campinas, Campinas, Brazil

Nanocellulose has emerged as a versatile building block for sustainable material technologies, offering high aspect ratio, tunable surface chemistry, and mechanical robustness. However, harnessing its full potential requires precise control over its interactions and organization across scales. This talk explores how supramolecular chemistry (non-covalent forces such as electrostatic complexation, hydrophobic interaction, and metal-phenolic coordination) enables the controlled assembly and disassembly of nanocellulose-based materials. The regioselective surface esterification of never-dried cellulose nanofibers (CNF) via water-assisted acylimidazole chemistry will be demonstrated to yield colloids with adjustable surface energy and interfacial behavior without compromising morphology. Also, metal-phenolic networks (MPNs) will be shown as a route to assemble tannin/CNF hybrids into porous foams with tunable mechanical strength, shrinkage, and hierarchical structure, showcasing how coordination chemistry can direct bottom-up material construction. The co-grinding of wood fibers and polyphenols will also be highlighted as a means of interfacial design to achieve tailored antioxidant activity, wettability, and optical properties in packaging films. Finally, electrostatic complexation will be discussed as a green, reversible route to engineer nanocellulose interfaces, enabling dynamic assemblies responsive to environmental stimuli (e.g., pH, ionic strength) in what is called saloplastics. A special angle will be used not only in the assembly but also in the disassembly of nanocellulosebased materials, which is equally important from a circularity standpoint.

# EFFECT OF FUNCTIONALIZATION ON CELLULOSE MICROFIBERS ON THE REMOVAL AND DETECTION VIA SERS OF THE PESTICIDE THIABENDAZOLE IN WATER

Authors: DOGNANI, G.¹; GOMES, S.A.¹; TOLOSA, R.G.¹; SANTOS, M.¹; CARVALHO, B.I.¹; OLIVEIRA, S.M.J.¹; CONSTANTINO, L.C.J.¹

Institution: <sup>1</sup>UNESP - Pres. Prudente, Presidente Prudente, Brasil

Cellulose is an abundant natural polysaccharide composed of glucose units. Its structure provides properties such as high mechanical strength, biocompatibility, and biodegradability, making it a sustainable and versatile material [1]. Widely used in the paper industries, cellulose also has advanced applications in bioplastics, membranes, sensors, and even flexible devices. The possibility of functionalizing its structure makes it even more attractive, allowing to produce different types of modifications on the fibers, which can contribute for various applications, including in the removal of contaminants from water. Thiabendazole (TBZ) is a fungicideclass pesticide used for preharvest and postharvest treatment of fruits and vegetables, reducing pests, rot, and deterioration during storage/transport [2]. However, excessive use of this pesticide can cause significant harm to human health and the environment [3]. Herein, three different cellulose microfibers were evaluated to remove and detect TBZ from water. Cellulose microfibers were oxidized to form dialdehyde cellulose (DAC), cationic dialdehyde cellulose (cDAC) and dicarboxyl cellulose (DCC). Zeta potential measurements showed that DAC, cDAC, and DCC exhibited surface charges of -24.1, +0.47, and -30.6, respectively. The microfibers were added to 10<sup>-3</sup> mol/L TBZ solution for 24 h at room temperature. After separating the supernatant and the microfibers, further analyses were performed. Surfaceenhanced Raman spectroscopy (SERS) using silver nanoparticles (AgNPs) demonstrated that cDAC exhibited a certain repulsion of TBZ, hindering the adsorption process. However, DCC, which had the lowest surface charge due to the carboxyl groups, showed a higher affinity for the evaluated pesticide. TBZ contains a benzimidazole ring, which can carry a positive charge due to molecule protonation. This allows interactions between the pesticide and the negative microfibers, which contain aldehyde and carboxyl groups in varying amounts (DAC and DCC). Thus, different types of cellulose microfibers interact differently with TBZ molecules, enabling removal and subsequent detection of the pesticide via SERS.

ENGINEERING HYDROPHOBIC CELLULOSE NANOFIBRILS VIA LIPASE-CATALYZED ESTERIFICATION: MECHANISTIC AND FUNCTIONAL ANALYSIS

Authors: COSTA, G.R.1; THIELEMANS, W.2; ARANTES, V.1

Institution: <sup>1</sup>Universidade de São Paulo, Lorena, Brazil <sup>2</sup>KU Leuven, Kortrijk, Belgium

Cellulose nanofibrils (CNFs) are renowned for their exceptional mechanical strength, tunable rheology, and biodegradability, making them central to the development of sustainable nanomaterials. However, their inherent hydrophilicity confers limitations for advanced applications, particularly in hydrophobic systems. To overcome this, we modified CNFs via enzymatic esterification, introducing hydrophobic butanoate groups through lipase-catalyzed attachment of butanoic acid, producing modified CNFs (CNF-BA). These materials were evaluated for their performance in hydrophobic thin film formation and as stabilizers in Pickering emulsions. Contact angle measurements revealed enhanced hydrophobicity: CNF-BA films maintained a 72.7° water contact angle after 200s, whereas unmodified CNFs absorbed water instantly. In oil-in-water Pickering emulsions, CNF-BA provided superior stabilization of cyclohexane-inwater systems, resisting coalescence. After 28 days, the creaming index remained close to 100%, with performance largely dependent on CNF concentration. To gain insight into the enzymatic process, isothermal titration calorimetry (ITC) was used to assess kinetic and thermodynamic parameters. The acyl-enzyme complex binding to CNF followed Michaelis-Menten kinetics, with a Km of  $5.3 \times 10^{-4} \pm 1.02 \times 10^{-4}$  mol·L<sup>-1</sup> and a Vmax of  $6.07 \times 10^{-5} \pm 1.6 \times 10^{-5}$  mol·L<sup>-1</sup>·s<sup>-1</sup>. The reaction enthalpy was -10.83 ± 0.7 kJ·mol<sup>-1</sup>, suggesting a moderately exothermic process. Km value indicates a comparable substrate affinity when compared with smallchain alcohols. The Vmax values where within a similar magnitude depending on substrate diffusivity. However, the heterogeneous nature of the CNF substrate may limit enzyme accessibility, partially explaining the observed values. The enthalpy change falls within the lower end of reported values for lipase-catalyzed reactions, which typically range from -10 to -40 kJ·mol-1, indicating moderate binding energy and reaction spontaneity. This study presents, to our knowledge, the first kinetic and thermodynamic characterization of a lipase acting on a nanocellulosic substrate. The successful enzymatic esterification of CNFs with butanoic acid improves their hydrophobicity and stability in emulsified systems, broadening their application scope.

#### ENHANCING PAPER BARRIER PROPERTIES THROUGH MULTILAYER COATINGS OF CATIONIC CELLULOSE NANOFIBRILS AND EXFOLIATED NANOCLAYS

Authors: BERTOLIM, F.B.1; POLEZI, G.1; BERNARDES, S.J.1

Institution: <sup>1</sup>Brazilian Nanotechnology National Laboratory (LNNano), Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brazil

Paper has emerged as a promising sustainable alternative to fossil-derived plastic materials. Nevertheless, its inherent high porosity and hydrophilic character result in limited barrier performance against the permeation of water, oils, and gases. To address these limitations, papers are commonly coated with waxes or synthetic polymers, which can compromise the packaging's biodegradability, recyclability, and repulpability. This work investigates the incorporation of cationic cellulose nanofibrils (cCNF) and anionic exfoliated nanoclays, montmorillonite (MMT) and laponite (LAP), to improve paper barrier properties, offering a more sustainable alternative to conventional coatings. The exfoliation degree of the anionic minerals' suspensions was evaluated by UV-Vis spectroscopy treatments by magnetic stirring, homogenization, probe sonication, and microfluidization. Higher transmittance values, particularly achieved microfluidization, indicated a greater extent of exfoliation, with the number of cycles also influencing particle size reduction. Paperboard substrates were coated with multilayers of cCNF and MMT or LAP suspensions. Permeability tests showed that the cCNF/MMT/cCNF three-layer coating reduced the water vapor transmission rate by up to 6.4% compared to the control group, while the cCNF+MMT mixture coating achieved a 4.6% reduction. Additionally, the cCNF/LAP/cCNF formulation showed a decrease of up to 3.7% compared with mixture formulations (cCNF+LAP), demonstrating that layering oppositely charged components enhances water barrier properties. About the oil barrier, the cCNF/LAP/cCNF and cCNF+MMT coatings proved efficient, withstanding up to kit #12. cCNF+MMT formulations showed significant reductions in oxygen transmission rate, with improvements of at least 96% compared to the control group. Aiming at the application of coated papers in food packaging, future tests will evaluate other coatings' formulations regarding oxygen and oil/grease resistance. In addition, the interfacial interaction between cCNF and nanoclays will be studied to understand its impact on the coating structure and improve the substrates' barrier properties.

## INFLUENCE OF NANOCELLULOSE CHARGE DENSITY AND SIZE ON KAOLIN FLOCCULATION PERFORMANCE

Authors: SILVA, D.1; BERNARDES, J.1

Institution: <sup>1</sup>Brazilian Nanotechnology National Laboratory, Brazilian Center for Research in Energy and Materials, Campinas, Brazil

Developing more efficient and sustainable water treatment solutions is critical in addressing global water quality challenges. Water treatment is mainly based on coagulation and flocculation processes since they are highly efficient and cost-effective. Although this process is extensively applied, the commonly used coagulants and flocculants are inorganic salts and synthetic polymers that can be associated with environmental concerns. In this context, nanocellulose-based compounds present a greener alternative to replace these compounds due to their renewability, biodegradability, and significant potential for chemical modification. Although several studies have addressed using nanocellulose in adsorbents and filtration membranes, only a few works have studied nanocellulose suspension as a flocculant. In this study, we used cationic (positively charged) cellulose nanofibrils (CCNFs) and explored the influence of their charge density and particle size on the flocculation performance of kaolin suspensions (contaminated water model). The flocculation efficiency was determined through visual inspection and transmittance measurements using UVvisible spectroscopy at 750 nm. The results showed that all cationic celluloses interact with the negatively charged surface of kaolin, promoting its decantation. However, the colloidal stability and phase separation of the systems is shown to depend on the concentration, size, and charge density of the fibers. In general, smaller fibers with higher charge densities demonstrated greater flocculation efficiency. The highly charged cellulose with the smallest size achieved efficient flocculation (%T > 85%) with just 0.5 mg L-1. These findings highlight the potential of customizing fiber properties to develop effective flocculants that facilitate direct flocculation with high efficiency, thus enhancing the environmental sustainability of wastewater treatment processes.

## INVESTIGATING OIL ABSORPTION KINETICS IN NANOCELLULOSE CRYOGELS VIA SYNCHROTRON 4D X-RAY TOMOGRAPHY

Authors: SILVA, J.M. $^1$ ; CLARO, P.I. $^2$ ; SALVADOR, A.J. $^2$ ; LOREVICE, M.V. $^2$ ; GOUVEIA, R.F. $^1$ 

Institution: <sup>1</sup>Brazilian Center for Research in Energy and Materials (CNPEM) | Federal University of ABC (UFABC), Campinas, Brasil <sup>2</sup>Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brasil

Oil spills and untreated industrial waste continue to pose severe threats to aquatic ecosystems, creating an urgent demand for effective environmental remediation strategies. In this context, cryogels fabricated from cellulose nanofibrils (CNF) and natural rubber latex (NRL) have emerged as particularly promising materials, synergistically combining the structural resilience of CNF-based porous materials with the intrinsic hydrophobicity of NRL for efficient oil capture. In this context, this study evaluated absorption kinetics and mechanical-morphological properties of CNF@NRL cryogels (80/20 wt.%) for oil capture inspired in our previous work. The cryogel maintained structural integrity after oil uptalking and absorption capacity of 46 g g<sup>-1</sup>. The kinetics of oil absorption and adsorption, as well as the interfacial interactions governing oil uptake, were investigated using synchrotron-based time-resolved microtomography (4D μCT). Furthermore, the material resilience under mechanical load was assessed by µCT focusing in understand the centrifugation effects to the material aiming reuse applicability without organic solvents. These findings revealed cryogels absorption efficiency and mechanical robustness. This cryogel system represents a significant technological advancement in oil remediation, offering mechanical resilience and synchrotron 4D μCT interface analysis. These features present a novel approach for designing eco-friendly materials, where multiscale experimental validation enables the development of environmental solutions.

# LIGNOCELLULOSIC NANOFIBRILS FILLING BIODEGRADABLE THERMOPLASTIC NANOCOMPOSITES: THE ROLE OF RESIDUAL LIGNIN IN THE COMPATIBILITY BETWEEN HYDROPHILIC AND HYDROPHOBIC PHASES

Authors: NISHIMOTO, P.H.K.<sup>1</sup>; MACHADO, G.S.<sup>1</sup>; LOREVICE, M.V.<sup>1</sup>

Institution: <sup>1</sup>CNPEM, Campinas, Brazil

Biodegradable thermoplastic nanocomposites are promising alternatives to conventional polymers due to their lower environmental impact. This study developed thermoplastic reinforced nanocomposites with lignin-containing nanofibrillated cellulose (LCNF), focusing on the effects of LCNF content and residual lignin on composite properties and filler-matrix compatibility. Sugarcane bagasse (SCB) was pretreated with NaOH solutions at different temperatures followed by mechanical fibrillation. The pretreatment parameters tuned LCNF average diameter (12-24nm) and residual lignin content (7-15%), evidenced by Atomic Force Microscopy (AFM) and chemical composition characterization. The lignin distribution along the LCNF surface was analyzed using infrared nanospectroscopy (AFM-IR), revealing its presence on the surface of the nanofibrils. The LCNF was solvent-washed, mixed with PBAT pellets, and cast into thin films (~30 µm thick). Mechanical testing demonstrated that films with 3wt.% LCNF containing 15wt.% residual lignin exhibited 40% and 55% increases in elastic modulus compared to films reinforced with bleached CNF (containing 3wt.% residual lignin) and neat PBAT, respectively. Scanning Electron Microscopy (SEM) revealed interfacial debonding in LCNF containing 7wt.% residual lignin reinforced films, indicative of poor fiber-matrix adhesion. In contrast, no debonding was observed in films reinforced with LCNF containing 15 wt.% residual lignin, suggesting enhanced interfacial compatibility. These findings indicate that lignin present on the LCNF surface enhances its interaction with the PBAT matrix. Improved UV-blocking performance was observed in the nanocomposite films, increasing proportionally with the residual lignin content. Additionally, the films maintained high flexibility and transparency, even at elevated LCNF:PBAT ratios, suggesting homogeneous dispersion of the nanofibers. Water contact measurements showed increased hydrophobicity with higher lignin content. Overall, the results highlight the potential of LCNF as an effective reinforcing agent for biodegradable polymer matrices, enhancing mechanical and functional properties while preserving key active features.

### ONE-STEP PREPARATION OF MULTIPLE PICKERING EMULSIONS STABILIZED BY CELLULOSE NANOFIBRILS

Authors: OLIVEIRA, S.M.C. $^1$ ; SILVA, E.P.C. $^1$ ; FERREIRA, S.E. $^1$ ; ALMEIDA, M.J. $^2$ ; BERNARDES, S.J. $^1$ 

Institution: <sup>1</sup>Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brazil
<sup>2</sup>Illum School of Science, Campinas, Brazil

Pickering emulsions consist of dispersions of immiscible liquids stabilized by solid particles, offering high resistance to coalescence. Nanoparticles can be employed to create simple or multiple emulsions, with the latter being especially advantageous for encapsulating both hydrophobic and hydrophilic substances, enabling controlled release and protection of active agents. Traditional methods for preparing multiple emulsions generally involve a two-step emulsification process and the use of two types of emulsifiers, typically lipophilic and hydrophilic surfactants or inorganic particles. To develop more eco-friendly alternatives for emulsion stabilization, cellulose nanofibrils (CNFs) are promising biobased particles due to their renewability and biodegradability. Nonetheless, their application has mostly been limited to the stabilization of simple oil-inwater (O/W) emulsions. This study investigates a one-step method to produce multiple Pickering emulsions using a single type of surface-functionalized CNF. Cationic or anionic CNFs were used to stabilize almond oil-based emulsions, with their colloidal stability and morphology monitored over time through optical and confocal microscopy. Experimental results and classical molecular dynamics simulations indicated that both CNFs effectively stabilized oil-inwater droplets, while the water-in-oil droplets stabilization was attributed to the presence of oleic acid within the oil phase. Notably, anionic CNFs proved to be more effective in stabilizing water-in-oil-in-water (W/O/W) emulsions, preserving the multiple morphology for over two months under ambient conditions. These results highlight the potential of functionalized CNFs as sustainable stabilizers for developing multiple emulsions suitable for food, pharmaceutical, and cosmetic formulations.

#### OPTIMIZED NITRO-OXIDATION OF SUGARCANE PULP FOR THE PRODUCTION OF CARBOXYLATED NANOCELLULOSE HYDROGELS FOR HEAVY METAL ADSORPTION

Authors: GOMES, A.S. $^{1.2}$ ; WENG, J. $^2$ ; PAN, L. $^2$ ; POTOFF, R. $^2$ ; DOGNANI, G. $^1$ ; ABDEL AZIZ, Y. $^2$ ; JOB, A.E. $^1$ ; CONSTANTINO, C.J.L. $^1$ ; HSIAO, B.S. $^2$ ;

Institution: <sup>1</sup>São Paulo State University – UNESP, Presidente Prudente, Brasil <sup>2</sup>Stony Brook University, Stony Brook, United States

Conventional cellulose isolation methods typically involve several steps, often resulting in considerable waste and byproduct generation [1]. Alternatively, the nitro-oxidation process (NOP) enables the one-step production of carboxylated nanocellulose fibers by simultaneously removing lignin and hemicellulose while oxidizing the cellulosic components [1]. In this study, NOP conditions were optimized for extracting and oxidizing cellulose nanofibers (CNFs) from sugarcane pulp (SCP). A large batch of carboxylated CNFs was obtained by treating 60 g of SCP with 50% (v/v) nitric acid at 50 °C for 5 hours. After washing to a pH>3, the suspension was homogenized under high pressure in the yield of 1% (w/w) CNFs. Hydrogel beads were then formed by crosslinking CNFs with 150 mM iron(III) nitrate solution and left for 3 hours for complete iron diffusion [2]. To evaluate the potential application of the hydrogel beads in wastewater treatment, adsorption experiments were performed using 3 g of hydrogel beads in 50 mL of a solution containing 50 mg/L of Cr(VI), under varying pH conditions (1-11) and different contact times (5-360 min). FTIR analysis verified the successful oxidation of CNFs, evidenced by the strong C=O stretching peak at 1672 cm<sup>-1</sup>, with a degree of oxidation of 1.29 mmol/g. For the adsorption tests, the removal efficiency exceeded 65% under all tested pH conditions. Therefore, pH 7, which achieved a removal efficiency of 73.5%, was selected for subsequent experiments. These findings highlight the effectiveness of the NOP approach in valorizing agricultural residues into sustainable hydrogel adsorbents with promising potential for environmental remediation applications.

### PREPARATION OF CELLULOSE NANOFIBRILS VIA OXYPROPYLATION OF CELLULOSE FIBERS

Authors: ALVES, R.L.  $^1$ ; CARRIELLO, M.G.  $^1$ ; PEGORARO, M.G.  $^1$ ; PEREIRA, B.  $^2$ ; ARANTES, V.  $^2$ ; REZENDE, L.M.  $^3$ ; MENEZES, A.J.  $^1$ 

Institution: <sup>1</sup>Federal University of São Carlos, Sorocaba, Brazil <sup>2</sup>School of Lorena, University of São Paulo, Lorena, Brazil <sup>3</sup>José Crespo Gonzales Faculty of Technology, Sorocaba, Brazil

Chemical modifications, such as oxypropylation, aim to graft poly(propylene oxide) chains onto lignocellulosic substrates and agro-industrial residues rich in hydroxyl groups (-OH), enabling the production of composites, polyurethanes (PU), and partially modified materials, depending on the reaction conditions, thus adding value to these materials. In this study, cellulose nanofibrils (CNFs) were prepared from bleached eucalyptus fibers modified via oxypropylation. Initially, 5 g of cellulose were pre-activated in an ethanolic solution containing KOH and LiOH (50/50, v/v) for 1 hour under vacuum in a desiccator. The oxypropylation reaction was conducted in an autoclave reactor equipped with a manometer and a heating mantle, using a molar ratio [OP]/[OHcellulose] = 1, at temperatures of 130 and 165 °C, maintained for 1 hour. The modified fibers underwent Soxhlet extraction with n-hexane to remove the homopolymer formed. Subsequently, suspensions containing 1.5% modified fiber were prepared, dispersed using an Ultra-Turrax, and defibrillated with a Masuko refiner connected to a digital energy meter. Defibrillation was performed in cycles, and aliquots were collected at each stage for particle size analysis. The obtained samples were characterized by Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS), atomic force microscopy (AFM), thermogravimetric (TG), and dynamic light scattering (DLS). FTIR spectra evidenced the appearance of new bands attributed to CH<sub>3</sub> groups, confirming the success of the chemical modification. The crystalline structure showed a reduction in the crystallinity index of up to 64%, becoming more amorphous, while the thermal stability of the samples modified with LiOH was higher than that of the fibers treated with KOH. Additionally, the CNFs exhibited a significant reduction in energy consumption during defibrillation, ranging from 67% to 93% compared to the unmodified fiber, highlighting the effectiveness oxypropylation in the process of obtaining cellulose nanofibrils.

# PRODUCTION OF ANTIMICROBIAL BIOAEROGELS FROM LIGNIN-CONTAINING CELLULOSIC MICROPARTICLES AS A COPRODUCT OF A 2G SUGARCANE BIOREFINERY

Authors: FERREIRA, G.P.  $^{1}$  ; SOARES, M.C.  $^{1}$  ; PEREIRA, P.H.  $^{1}$  ; ARANTES, V.  $^{1}$ 

Institution: ¹Lorena School of Engineering, University of São Paulo (EEL/USP), Lorena, Brazil

This study explores the valorization of lignin-containing cellulosic solid residue (LCSR), generated after the enzymatic hydrolysis of sugarcane bagasse pulp processed under the second-generation (2G) biorefinery concept at pilot scale, for the production of bioaerogels with antimicrobial activity. Initially, the LCSR was characterized for its physicochemical properties, including chemical composition, particle size and distribution, surface area, Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA). Subsequently, LCSR microparticles were used to produce aerogels via freeze-drying, following two approaches: with and without crosslinking. For the cross-linked samples, citric acid was used at concentrations ranging from 0.5% to 5% (w/v), followed by stirring for 30 minutes. The suspensions were then molded into silicone molds (5 to 10 mL per cavity) and frozen at -20°C for 24 h prior to freeze-drying. The resulting bioaerogels were subjected to antimicrobial activity tests against two model bacteria: Staphylococcus aureus (Gram-positive) and Escherichia coli (Gramnegative). The results showed that the aerogels produced from LCSR exhibited superior antimicrobial performance compared to those made with nanofibrillated cellulose (NFC) derived from bleached eucalyptus kraft pulp, highlighting the functional potential of LCSR due to its residual lignin content. This study reinforces the importance of fully utilizing lignocellulosic biomass within biorefinery platforms, contributing to the development of high value-added coproducts with applications in areas such as biomaterials and sustainable packaging.

#### SB-SPUN CELLULOSE ACETATE NANOFIBERS AND 3D-PRINTED MICRONEEDLE PATCH AS TRANSDERMAL ANTIMICROBIAL SYSTEM

Authors: TEODORO, K.B.R.¹; ALVARENGA, A.D.²; OLIVEIRA, L.F.R.³; PEREIRA, T.S.⁴; MERCANTE, L.A.⁵; GONÇALVES, R.M.⁵; CORREA, D.S.¹

Institution: ¹Nanotechnology National Laboratory for Agriculture, Embrapa Instrumentation, São Carlos - SP, Brazil

<sup>2</sup>GPEA, Institute of Chemistry, University of São Paulo (USP), Sao Carlos - SP, Brazil

<sup>3</sup>PPGBiotec, Federal University of São Carlos, Sao Carlos - SP, Brazil <sup>4</sup>PPGQ, Federal University of São Carlos (UFSCar), Sao Carlos - SP, Brazil

<sup>5</sup>PPGQ, Institute of Chemistry, Federal University of Bahia (UFBA), Salvador - BA, Brazil

The global research agenda for mitigating antimicrobial resistance from World Health Organization (WHO) points to the urgency in investigate antimicrobial stewardship interventions. Traditional antibiotic delivery methods face challenges, including systemic toxicity and poor bioavailability. In this context, transdermal microneedle (TMN) arrays offer a promising alternative, as they control the amount of antibiotic delivered and bypass the skin barrier to enhance antibiotic penetration. TMN follows minimally invasive strategy to release active substances into circulation, improving patient compliance and minimizing systemic side effects. In this study, cellulose acetate/polyethylene oxide (CA/PEO) nanofibers (NFs) were engineered to encapsulate tetracycline (TC) and integrated with 3D-printed TMN to deliver antibiotics. Solution blow spinning was selected for nanofiber production due to its ability to rapidly generate robust mats with high loading capacity. The nanofibers were directly collected on the backside of the MN patch. The TMN, consisted of 18 hollow microneedles, shaped like hypodermic needles and arranged on a circular base, were designed using Blender 4.1. The 3D-printing was performed via digital light processing (DLP) using an Elegoo Mars 2 Pro LCD 3D printer (Elegoo, China), and using Prizma 3D Bio Guide photocurable resin. Nanofiber morphology was characterized using field-emission scanning electron microscopy (FESEM), while composition was analyzed by Fourier Transform Infrared Spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and contact angle measurements for wettability assessment. The antimicrobial activity of the composite system was assessed through in vitro bacterial inhibition assays, and the drug release profile was compared with that of standalone nanofiber mats. The results showed that integrating NFs with MN patches led to a more sustained release of TC and reduced burst effects. The antimicrobial effect was confirmed by the diffusion of TC through the MN-modified patch into the pierced medium, effectively inhibiting the growth of Escherichia coli and Staphylococcus aureus.

#### SIMPLIFIED METHODOLOGY TO OBTAIN TEMPO-OXIDIZED CNFS FROM SUGARCANE BAGASSE AND PILOT-PLANT UPSCALING

Authors: BERNARDES, S.J. $^1$ ; ALONSO, S.T.P. $^2$ ; NASCIMENTO, M.D. $^2$ ; MASSUCATO, P.B. $^2$ 

Institution: <sup>1</sup>LNNano/CNPEM, Campinas, Brasil <sup>2</sup>LNano/CNPEM, Campinas, Brasil

Cellulose nanofibrils (CNF) are sustainable materials with applications in numerous fields. However, processes to isolate them from plant-based biomass are often highly time, energy and water-consuming, mainly due to lignin removal and mechanical defibrillation steps. As a result, large-scale commercial production and application of CNFs are still at an early stage. Previous studies demonstrated that TEMPOcatalyzed oxidation with sodium hypochlorite (NaClO) can simultaneously remove lignin and fibrillate the cellulose. Additionally, sugarcane bagasse was also found to be a less recalcitrant material, allowing chemical defibrillation to reach nanoscale dimensions without requiring intensive mechanical treatments. In this work, we developed a simplified one-pot TEMPO-oxidation methodology to obtain CNFs from sugarcane bagasse. High CNF yields (up to 30%) were obtained by adjusting NaClO dosage (10-50 mmol/g of fibers) and applying a low energy mechanical treatment (Ultra-Turrax Homogeneizer, at 20.000 rpm). AFM imaging revealed nanofibers with diameters below 5 nm and lengths around 1 µm at all oxidation levels above 20 mmol/g. Conductometric titration showed varying carboxylate contents as oxidation increased, with a charge density of up to 1.1 mmol/g for higher NaClO content. Acetyl-bromide lignin measurements, FTIR and TGA characterizations, confirmed significant lignin removal, as low as 6 wt.% for the highest NaClO loadings. Aqueous suspensions (0.5 wt.%) of CNFs exhibited typical shear-thinning rheology for higher NaClO contents, typical of CNF hydrogels. To test its scalability, a batch of the developed process was successfully executed at Pilot-Plant scale, processing 2.8 kg of bagasse into 20 L of ~3% wt. CNF dispersion. AFM imaging, chemical characterizations, and rheology all confirmed the product to be highly charged (~1.1 mmol/g) cellulose nanofibrils (< 8nm diameter), confirming that TEMPO-oxidized CNF was obtained at a large scale. Overall, this study provides a simpler, scalable methodology to obtain CNFs from sugarcane bagasse, opening new opportunities for commercial-scale nanocellulose applications.

### SPRAY-DRIED FORMULATIONS CONTAINING CELLULOSE NANOFIBERS FOR BIOLOGICAL CONTROL

Authors: MIELI, M.J. $^1$ ; FERNANDES, P.A. $^1$ ; MASSIMINO, L.C. $^1$ ; ALMEIDA, J.E.M. $^2$ ; BARUD, H.S. $^1$ 

Institution: <sup>1</sup>University of Araraquara, Araraquara, Brasil <sup>2</sup>Biological Institute, Campinas, Brazil, Campinas, Brasil

One of the main challenges in advancing biopesticide technology is extending the shelf life of formulations while preserving the viability of microbial agents. Spray drying stands out as a scalable and efficient encapsulation technique that enables incorporation of biocontrol fungi into protective biopolymeric matrices. However, there remains a need to develop formulations that provide enhanced physical protection and structural stability to biocontrol agents, reducing reliance on synthetic pesticides and improving pest control efficacy. This study focused on developing advanced formulations based on maltodextrin, starch, and cellulose nanofibers (CNF) for encapsulating the entomopathogenic fungus Beauveria bassiana, processed by spray drying at an outlet temperature of 70 °C. Special emphasis was placed on the inclusion of 3% (w/w) CNF due to its nanoscale morphology, high surface area, mechanical strength, and exceptional ability to stabilize microparticle structures, which together provide physical protection to fungal conidia and reinforce the spray-dried matrix. Thermal analysis (TG/DSC) indicated stability up to 250 °C, with Tmax values ranging from 261 °C to 312 °C-the highest observed in the starch/maltodextrin/CNF system. Formulations exhibited greater final mass loss (~95-96%) at 800 °C, confirming successful encapsulation. FTIR spectra revealed strong molecular interactions between the encapsulants and fungal biomass, with characteristic bands at 3422, 2922, 1652, 1547, 1160, and 1027 cm<sup>-1</sup>. SEM images showed spherical particles with increased surface roughness in CNF-containing samples. Notably, viability tests demonstrated conidial survival of 2.9x109 CFU/g, highlighting the protective efficiency of CNF in maintaining fungal activity post-encapsulation. These findings underscore the multifunctional role of CNF as a critical excipient in biocontrol formulations. Future work will focus on optimizing polymer ratios and evaluating field stability to advance sustainable applications in agricultural pest management.

## T-COMPLEX BIOINGREDIENT FROM MARINE ASCIDIANS

# TAILORING CELLULOSE NANOFIBRIL FILM PERFORMANCE VIA ENZYMATIC TREATMENTS AND LIGNIN NANOPARTICLES INCORPORATION

Authors: COSTA, J.<sup>1</sup>; DAMASIO, R.<sup>2</sup>; GRASSELLI, S.<sup>3</sup>; REDMOND, E.<sup>2</sup>; JUNIOR, H.J.E.<sup>4</sup>

Authors: LAS-CASAS, B.1; ROJAS, O.2; ARANTES, V.1

Institution: <sup>1</sup>ESALQ/USP and Webtech, Piracicaba, Brasil <sup>2</sup>Department of Chemical Engineering, SUNY College of Environmental Science and Forestry, Syracuse, USA <sup>3</sup>Gea Mechanical Equipment Italia S.p.A., Parma, Italy <sup>4</sup>Departamento Ciências Florestais ESALQ/USP, Piracicaba, Brasil

Institution: <sup>1</sup>University of Sao Paulo, Lorena, Brazil <sup>2</sup>University of British Columbia, Vancouver, Canada

T-complex is a natural bioingredient derived from the tunicate Styela plicata. Major components are cellulose 53.7 %, protein 13.10 %, fat 4.42 % (C6:0; C10:0; C18:0; C12:0; C14:0; C18:11n-9c; C16:1; C16:0 and C15:0) and 28.8 % of ash. In this study, T-Complex was obtained through a newly proposed method for extracting nanocellulose from tunicates. The process involved washing and preparing the nanocellulose material, followed by two stages of mechanical fibrillation. The first stage utilized a Masuko Sangyo Masscolloider (model MKCA6-2, Japan) operating at 1500 rpm, 2% consistency, and 4 cycles. The second stage employed a GEA Niro Soavi Panda Plus tabletop UHP homogenizer at pressures of 700, 1200, and 1500 bar, for 10 cycles. Cellulose and proteins interact through residual sugars, forming protein-cellulose complexes known as PC-tun complexes. XRD analysis revealed a cellulose crystallinity of 85%.. At 700 bar, nanofibrillation progression was observed through light microscopy (LM) and visual inspection (VI) images, with the specific surface area (SSA) increasing from 265.8 m<sup>2</sup>/kg for the initial nanofiber homogeneity (NH) to 714.2 m<sup>2</sup>/kg after the 10th recirculation cycle. At 1200 bar, LM and VI images also demonstrated nanofibrillation progression, and the SSA increased from 265.8 m<sup>2</sup>/kg to 564 m<sup>2</sup>/kg after the 10th cycle. Finally, at 1500 bar, nanofibrillation progression was similarly observed, with the SSA increasing from 265.8 m<sup>2</sup>/kg to 573.2 m<sup>2</sup>/kg after the 10th recirculation cycle. T-complex suspension occurred between 35 to 1980 nm. In our results no protein-cellulose separation occurs after applying 1500 G at 4 °C during 15 min, indicating that the PC-tun complexes are well linked as described. As a bioingredient, T-complex holds significant potential due to its unique protein composition, oAering promising applications in health, personal care, and medical fields

Cellulose nanofibril (CNF) films have emerged as a promising alternative to synthetic polymers for packaging applications owing to their oxygen and grease barrier, transparency, and mechanical performance. The performance of CNF films is highly dependent on the intrinsic properties of the nanofibers, such as aspect ratio, crystalline structure, and chemical composition. Production steps, including mechanical defibrillation and preand post-treatments, may significantly alter these intrinsic properties, offering opportunities for customization. In addition, CNF films' performance can be further tailored by incorporating of bio-additives such as lignin nanoparticles (LNP), which can yield multifunctional films with antioxidant and UV-barrier properties for active packaging. This study employed three different strategies to customize CNF films for highperformance packaging: i) endoglucanase (EG)-assisted disc ultra-refining; ii) endoxylanase (EX) post-treatment; and iii) LNP incorporation. EG pre-treatment under optimized conditions enabled the isolation of CNFs with reduced energy consumption, resulting in films with higher transmittance, barrier performance, and lower surface wettability than the untreated samples. EX post treatment allowed the isolation of CNFs with varying xylan content, preserved crystalline structure, and various morphologies. EX-CNF films exhibited a smoother surface, improved thermostability, and excellent moisture barrier but became less strong and more brittle as xylan content decreased. The incorporation of LNP impart antioxidant and UV-barrier properties without compromising the mechanical, thermal, and barrier performance of the CNF films. LNP self-assembled on the surface of the films without affecting their physical properties and crystalline structure. Therefore, EG and EX treatments, as well as LNP incorporation, represent eco-friendly strategies for tailoring CNF film properties, showing great potential for developing high-performance, customizable biomaterials for active packaging applications.

# TAILORING THE SURFACE PROPERTIES OF CELLULOSE NANOFIBRIL FILMS THROUGH ENZYMATIC ESTERIFICATION AND LIGNIN NANOPARTICLES

Authors: TEIXEIRA, O.1; COSTA, G.1; ARANTES, V.1

Institution: <sup>1</sup>Applied Bionanotechnology Laboratory, Department of Biotechnology, Lorena School of Engineering, Universidade de São Paulo, Lorena, Brazil

Cellulose nanofibrils (CNFs) are promising biomaterials, but their pronounced hydrophilicity restricts their applicability, especially in interactions with apolar media or in products requiring water and vapor barriers. Surface modification of CNFs to reduce their water affinity is crucial for expanding their applications, enhancing dimensional stability, improving moisture resistance, and increasing compatibility with hydrophobic components. In this study, we developed and characterized thin CNF films with reduced hydrophilicity, focusing on three systems: (i) CNFs enzymatically esterified with butyric acid; (ii) CNFs containing lignin nanoparticles (LNPs); and (iii) CNFs both esterified and containing LNPs. Low-angle laser light scattering (LALLS) was employed to determine the nanofibril size distribution in suspension, thereby ensuring film homogeneity. X-ray diffraction (XRD) was used to determine the crystallinity index, while atomic force microscopy (AFM) and scanning electron microscopy (SEM) were employed to visualize the surface morphology and fibril entanglement. Fourier Transform Infrared Spectroscopy (FTIR) confirmed ester bond formation and lignin presence. Thermogravimetric analysis (TGA) assessed thermal stability, while contact angle measurements hydrophilicity reduction. Zeta measurements indicated surface charge and colloidal stability, shedding light on intercomponent interactions. This comparative analysis elucidated the impact of enzymatic esterification and LNP incorporation on the structural, thermal, chemical, electrokinetic, and surface properties of CNF films. The findings support the potential use of these optimized films as platforms for the transdermal delivery of hydrophobic drugs, such as ibuprofen.

# THERMAL-MECHANICAL EVALUATION OF HYDROXYPROPYLATED STARCH AS PLASTICIZER FOR CONVENTIONAL THERMOPLASTIC STARCH FILMS REINFORCED WITH CELLULOSE NANOFIBRILS

Authors: LOPES, H.  $^1$ ; COSTA, F.  $^2$ ; ALVES, L.  $^3$ ; PARRA, D.  $^2$ ; DUFRESNE, A.  $^4$ ; KOMATSU, D.  $^5$ ; MENEZES, A.  $^3$ 

Institution: ¹Technological College of Sorocaba (Fatec), Sorocaba, Brazil

<sup>2</sup>Nuclear and Energy Research Institute (IPEN-CNEN/SP), São Paulo, Brazil

<sup>3</sup>Federal University of São Carlos (UFSCar), Sorocaba, Brazil

<sup>4</sup>University of Grenoble Alpes, CNRS (Grenoble INP), Grenoble, France

<sup>5</sup>Pontifical Catholic University of São Paulo (PUC-SP), Sorocaba, Brazil

Cellulose nanofibril (CNF) films have emerged as a promising alternative to synthetic polymers for packaging applications owing to their oxygen and grease barrier, transparency, and mechanical performance. The performance of CNF films is highly dependent on the intrinsic properties of the nanofibers, such as aspect ratio, crystalline structure, and chemical composition. Production steps, including mechanical defibrillation and preand post-treatments, may significantly alter these intrinsic properties, offering opportunities for customization. In addition, CNF films' performance can be further tailored by incorporating of bio-additives such as lignin nanoparticles (LNP), which can yield multifunctional films with antioxidant and UV-barrier properties for active packaging. This study employed three different strategies to customize CNF films for highperformance packaging: i) endoglucanase (EG)-assisted disc ultra-refining; ii) endoxylanase (EX) post-treatment; and iii) LNP incorporation. EG pre-treatment under optimized conditions enabled the isolation of CNFs with reduced energy consumption, resulting in films with higher transmittance, barrier performance, and lower surface wettability than the untreated samples. EX post treatment allowed the isolation of CNFs with varying xylan content, preserved crystalline structure, and various morphologies. EX-CNF films exhibited a smoother surface, improved thermostability, and excellent moisture barrier but became less strong and more brittle as xylan content decreased. The incorporation of LNP impart antioxidant and UVbarrier properties without compromising the mechanical, thermal, and barrier performance of the CNF films. LNP selfassembled on the surface of the films without affecting their physical properties and crystalline structure. Therefore, EG and EX treatments, as well as LNP incorporation, represent ecofriendly strategies for tailoring CNF film properties, showing great potential for developing high- performance, customizable biomaterials for active packaging applications.

# THORIUM INTERACTION WITH ACTIVATED NANOCELLULOSE AND ITS INFLUENCE ON STRUCTURAL PROPERTIES

Authors: Carriello, M.G.<sup>1</sup>; Alves, R.L.<sup>1</sup>; Pegoraro, M.G.<sup>1</sup>; Janolla, A.T.<sup>1</sup>; Lopes, M.H.S.<sup>1</sup>; Assis, S.J.A.<sup>1</sup>; Flayeh, A.A.<sup>2</sup>; Freitas, R.M.R.<sup>1</sup>; Botaro, R.V.<sup>1</sup>; Rezende, L.M.<sup>3</sup>; Menezes, J.A.<sup>1</sup>; Mambrini, P.G.<sup>1</sup>

Institution: <sup>1</sup>Federal University Of São Carlos, Sorocaba, Brazil <sup>2</sup>Ministry of Oil – Middle Refineries Company – Karbala Refinery, Babylon, Iraq <sup>3</sup>José Crespo Gonzalez Faculty of Technology, Sorocaba, Brazil

The modification of nanocellulose has gained increasing attention due to its potential for advanced applications through the tuning of its physicochemical properties. Among various approaches, the incorporation of metallic elements is particularly promising, as it can impart specific functionalities to the material. Thorium (Th), however, remains largely unexplored within this context. Its selection was primarily driven by its comparatively large atomic radius and the limited number of studies addressing its integration into cellulose-based structures. This study aimed to investigate the interaction of Th with activated nanocellulose. Nanocellulose was activated using a 0.5 M NaOH solution in an equimolar ratio to the hydroxyl groups. Th(NO<sub>3</sub>)<sub>4</sub>·5H<sub>2</sub>O was then added in molar ratios of 1:1, 2:1, 4:1, and 8:1 relative to the activated OH groups. The samples were stirred at 50°C for 1 hour, washed, and analyzed using XRD, SEM-EDS, Raman spectroscopy, FTIR, TGA, DSC, contact angle analysis, and UV-Vis spectroscopy. Results showed that Th was successfully incorporated into the nanocellulose structure at 1:1 and 2:1 ratios, leading to a transition from crystalline to amorphous morphology and increased material brittleness. At higher Th ratios (4:1 and 8:1), the crystalline structure of α-cellulose remained unchanged, and Th was not detected, suggesting unsuccessful incorporation. This behavior is likely influenced by the acidic nature of Th and its interaction with residual NaOH in the reaction medium. The findings suggest that there is an optimal range for Th incorporation into nanocellulose and highlight the need for careful control of reaction conditions. Further research will include dynamic mechanical analysis (DMA) to better understand the viscoelastic behavior of Th-functionalized nanocellulose.

### TOCNF AND ALGINATE FILMS AS PH-RESPONSIVE ACTUATORS

Authors: AMARAL, G.1; MERCES, L.2; BERNARDES, J.2

Institution: <sup>1</sup>Federal University of ABC, Campinas, Brazil <sup>2</sup>Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brazil

TEMPO-oxidized cellulose nanofibrils (TOCNF) have gained attention as a promising material due to their biodegradability, high elastic modulus, low density, high aspect ratio, and the presence of carboxyl groups, which impart sensitivity to pH variations. When combined with alginate, an anionic biopolymer widely used for its biocompatibility and ability to form ionic gels, it becomes feasible to develop films suitable for use as smart actuators. pH-responsive actuators are of particular interest in fields such as biomedicine and soft robotics, where controlled changes in shape or volume in response to the chemical environment are desirable. This study aims to evaluate the pH responsiveness of polymeric films made of TOCNF, alginate, and the alginate-TOCNF combination. The films were first dried, then rehydrated for 24 hours and subsequently immersed in solutions with pH 1 and pH 9. Responsiveness was assessed based on dimensional changes in the films, aiming to understand their swelling and contraction behavior under acidic and basic stimuli. Preliminary results indicate that the films made only with TOCNF did not exhibit dimensional responsiveness. This may be due to the hornification of the fibrils during drying, which affects rehydration and conformation changes in response to pH. When alginate is mixed with TOCNF in a 5:1 mass ratio, the film exhibits volume contraction and expansion of approximately 2,6% at pH 1 and 4,4% at pH 9. In contrast, films composed solely of alginate have reduced mechanical properties. Therefore, the alginate-TOCNF composite film has potential as an actuator, combining pH responsiveness with mechanical stability.

# TUNING INTERFACIAL INTERACTIONS BETWEEN CATIONIC NANOCELLULOSE AND ANIONIC CLAYS FOR STABLE DISPERSIONS

Authors: POLEZI, G.1; BERTOLIM, F.B.1; BERNARDES, J.1

Institution: <sup>1</sup>Brazilian Nanotechnology National Laboratory (LNNano), Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brazil

Homogeneous and stable aqueous dispersions of nanocellulose and clays are essential for producing nanocomposites with enhanced mechanical, optical, thermal, and barrier properties. Therefore, it is essential to understand the interactions that govern the colloidal behavior of these systems. In this study, we investigated the interactions between cationic cellulose nanofibrils (cCNF) and anionic clay nanoplatelets — bentonite (MMT) and kaolin (KAO) — in aqueous media across a wide pH range. Zeta (ζ) potential measurements showed that cCNF suspensions remained positively charged throughout the pH range, peaking at +38 mV at pH 7. MMT and KAO suspensions were negatively charged, ranging from -27 to -36 mV (MMT) and -7 to -55 mV (KAO). At pH 7, mixing cCNF with 70-80 wt.% MMT caused a shift to negative ζ-potential, indicating charge inversion. No such inversion occurred in cCNF/KAO mixtures. Quartz crystal microbalance with dissipation monitoring experiments revealed strong interfacial interactions in cCNF/MMT and cCNF/KAO systems. At neutral pH, the increase in dissipation (ΔD) upon MMT adsorption onto cCNF was smaller than that observed for KAO, suggesting that cCNF/MMT forms a more compact or rigid structure, while cCNF/KAO forms a thicker or more viscoelastic layer. In cCNF/MMT systems, pH significantly influenced adsorption: at pH 12, a high  $\Delta D$  and large negative frequency shift ( $\Delta f$ ) indicated a thick, hydrated, and viscoelastic layer. At pH 1, minimal  $\Delta D$  and  $\Delta f$  changes suggested limited interaction and a thinner film. Moderate values at pH 7 reflected intermediate adsorption and viscoelasticity. These results highlight pH as a key factor in tuning interfacial assembly between cCNF and clays, with implications for designing bioinorganic nanocomposites. Further studies are needed to better understand the forces driving these interactions.

# REGENERATED CELLULOSE BEADS: PREPARATION BY DROPPING TECHNIQUE, CHARACTERIZATION AND TAILORED APPLICATIONS INTO STARCH-BASED MATERIALS – PRELIMINARY RESULTS

Authors: LIZANO, K.L.1; VIEIRA, I.A.2; CURVELO, A.A.S.2

Institution: <sup>1</sup>Department of Materials Engineering (SMM) - São Carlos School of Engineering (EESC) - USP, São Carlos, SP, Brasil <sup>2</sup>São Carlos Institute of Chemistry (IQSC), USP, São Carlos, São Paulo, Brazil

Cellulose is typically used in its fibrous or nanocrystalline forms. However, regenerated cellulose, obtained through dissolution and regeneration into its most stable allomorph, cellulose type II, offers versatility in shaping for tailored applications. Despite this potential, its use in combination with starch, particularly in bead form, remains underexplored. Both, cellulose and starch are polysaccharides composed of glucose units linked by glycosidic bonds, but with different structures. In this work, cellulose was derivatized to be dissolved, and a viscose solution was obtained, which was shaped into beads for targeted applications. Beads were produced via a dropping technique using syringes of varying diameters (0.3-0.55 mm). Regeneration occurred in a 2M H2SO4 bath, followed by washing to neutral pH and stored either in water or a 20% v/v glycerol solution. Samples were classified as fresh, air-dried, or freeze-dried. Characterization included optical microscopy (fresh and airdried beads), SEM (freeze-dried beads), size distribution (fresh beads), and thermogravimetric analysis for thermal stability and glycerol content. The effect of storage conditions on bead size was also evaluated. Fresh samples with and without glycerol exhibited diameters of ~2.5 mm, while air-dried samples without glycerol reduced to 1.25 mm. These increased to ~1.5 mm, upon re-exposure to glycerol. A porous morphology and glycerol content reaching around 70% per bead were observed. Preliminary applications showed the cellulose bead's capacity for encapsulating additives and for composites development, achieving integration with starch-based materials. Glycerolloaded beads were incorporated into thermoplastic starch (TPS) by extrusion at 0.5% and 1%, acting as reinforcements and plasticizer carriers. Additionally, starch granules were successfully encapsulated within the beads, confirmed by SEM. Inspired by this, starch beads were also produced to enhance powder flow in TPS extruder feeders, addressing clogging issues in TPS production as a technological solution. All these findings highlight the promising potential of cellulose beads for starchbased material applications.

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