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BOOK OF ABSTRACTS

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INFLUENCE OF BARK CHEMICAL COMPOSITION ON THE THERMAL STABILITY OF POLYPROPYLENE COMPOSITES

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ABSTRACT

Forest bark is a widely available by-product from the forest industry, yet its valorisation as a raw material remains underutilized, limiting its contribution to a circular economy but offering a promising source of functional fillers for the development of sustainable biobased composites. Its complex chemical composition including cellulose, hemicellulose, lignin and extractives, presents an opportunity to improve thermal performance in polymer matrices. This study explores the influence of different bark species as fillers in a polypropylene (PP) matrix, with a focus on the thermal stability and the fire behaviour in relation to their distinct chemical composition.

Three bark species were investigated: birch (*betula sp.*), atlas cedar (*cedrus atlantica*) and maritime pine (*pinus pinaster*). Bark residues were dried and ground into powders (<250µm). PP-based biocomposites were prepared by melt mixing followed by injection molding, incorporating 10 to 35 wt% bark filler. Thermal properties were carried out on micro-scale using thermogravimetric analysis (TGA) and microscale combustion calorimetry (PCFC) to assess thermal degradation, char formation and heat release. Initial thermal characterisation of raw bark powders revealed pronounced differences among species. Birch bark showed the highest thermal stability, with decomposition temperatures around 370°C and 440°C and the highest total heat release (THR ~ 30 $kJ.g^{-1}$). In contrast, atlas cedar bark and maritime pine bark decomposed at lower temperatures (~ 335°C and 365°C, respectively), with lower THR values (~ 5 and 6 $kJ.g^{-1}$) Residual char yields also varied strongly. Atlas cedar and maritime pine barks produced higher char fraction than birch bark (36 wt% and 37 wt% against 10 wt%, respectively).

TGA and PCFC analyses of the PP composites highlight that increasing bark content led to an increase in char residue and a decrease in THR, both trends following approximately a mixing rule. Additionally, it was observed that increasing the filler content progressively led to higher degradation temperature of the PP matrix across all species, indicating improved thermal stability at higher bark loadings. Moreover, composites containing birch bark delayed polymer degradation more effectively than atlas cedar or maritime pine composites. The observed variations in residual char yields among species highlight that the efficiency of thermal stabilisation appears to mostly depend on the chemical structure of the formed char rather than its quantity. Ongoing chemical analyses aim to establish the correlation between the bark distinguished chemical composition and their thermal behaviours.

KEYWORDS

Bark, thermal stability, chemical composition, polypropylene/bark composites materials

PICKERING EMULSION-TEMPLATED ULTRA-POROUS FILMS FROM BIOPOLYMERS AS SUPPORTS FOR ENZYME IMMOBILIZATION

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ABSTRACT

Pickering emulsions (PEs) have attracted much attention due to their high stability. These emulsions exhibit unique advantages in film fabrication, particularly because of the adjustable porosity. Here, we prepared hydrophilic, ultra-porous films from two biopolymers, sodium alginate and chitosan, using cellulose nanocrystals (CNC) as stabilizers for oil-in-water PEs. Our tailored PE formulation allows for precise control over the resulting pore architectures. By tuning CNC concentration and oil volume fraction, pore structure can be adjusted predictably. To counter the loss of robustness at high porosity, ionic cross-linking is optimized in two different strategies by varying parameters such as crosslinker concentration and cross-linking time. FTIR indicates characteristic functional groups upon cross-linking, and SEM reveals a regular, open porous network. The PE-based films show improved wet integrity compared with the non-crosslinked ones. Beyond structural control, these films offer a rationale for prospective enzyme immobilization: the interconnected porosity is expected to provide high specific surface area and short diffusion pathways beneficial for enzyme loading and mass transfer, while the functional groups on the film surface, such as hydroxyl, carboxyl or amino groups, provide potential interaction/coupling sites via adsorption, ionic binding, or covalent attachment after suitable activation. Overall, our PE-templated films represent a versatile platform for future functional films integrating enzymes and other catalytic components.

KEYWORDS

Pickering emulsion, biobased materials, porous films, enzyme immobilization, biocatalysis

CIRCULAR UPCYCLING OF TEXTILE WASTE INTO NANOCELLULOSE AEROGELS FOR WATER REMEDIATION

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ABSTRACT

The extraction of high-performance nanomaterials from textile waste represents a promising strategy for resource valorisation. Simultaneously, the textile industry constitutes a significant environmental concern due to the release of hazardous dyes and heavy metals into aquatic ecosystems, leading to biodiversity degradation and the necessity for costly wastewater treatment processes[1].

In this study, nanocellulose-based aerogels derived from three types of waste fabrics (cotton-based recycled fabric, viscose-based waste fabric, and a polyethylene terephthalate (PET)/cellulose blend) were developed and systematically evaluated for their effectiveness in the removal of heavy metals, dyes and emerging contaminants (PFAS) from contaminated water.

Various chemical pre-treatments were investigated to enhance the extraction of cellulose nanofibers (CNF) from textile waste[1,2,3]. The resulting CNF were employed as fundamental building blocks for aerogel fabrication and were combined with a cross-linker and a co-crosslinker to obtain a stable formulation[4,5]. CNF-based aerogels were subsequently produced through freeze-drying and thermal treatment. The resulting materials were thoroughly characterized, and their contaminant removal performance was assessed, showing outstanding sorption properties towards target contaminants (Figure 1).

This approach aims to promote a circular and environmentally sustainable strategy for water treatment. The upcycling of textile waste into high-performance nanomaterials establishes an effective circular approach, which not only mitigates environmental pollution but also supports the sustainable reuse of a waste as an innovative and environmentally responsible solution for wastewater treatment in the same process chain.

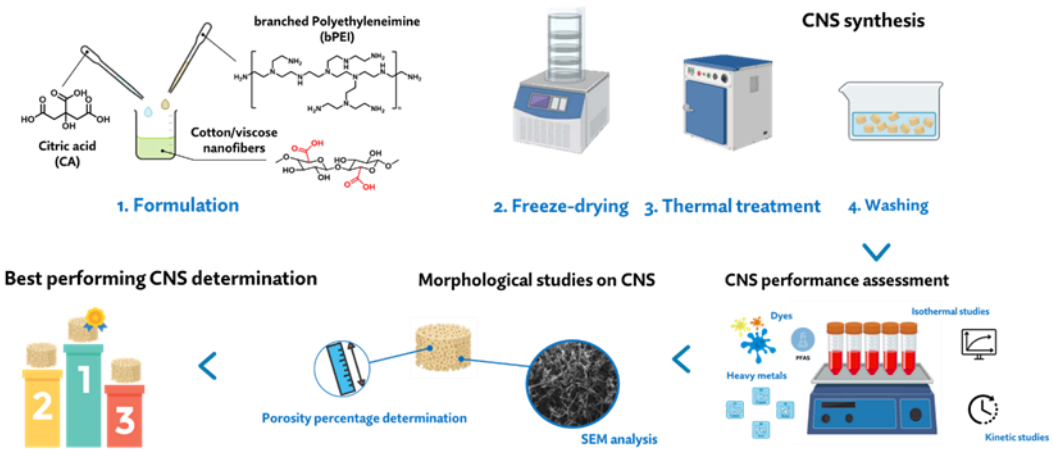

FIGURE 1 GRAPHICAL ABSTRACT

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KEYWORDS

Nanocellulose, aerogels, remediation, wastewater treatment

IN-SITU LASER-INDUCED CHEMICAL FORMATION OF HEXAGONAL BORON NITRIDE COATINGS ON WOOD

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ABSTRACT

Objectives: A great hindrance in the transition to sustainable timber construction is the inherent flammability of wood. While hexagonal boron nitride (h-BN) is an ideal candidate for fire protection due to its specific microstructure, its direct synthesis requires extreme temperatures, which are incompatible with heat-sensitive substrates such as wood. The objective of this study was to develop a rapid, scalable, and localized synthesis method for functionalizing wood surfaces with protective h-BN coatings without compromising the substrate's structural integrity.

Methods: The coatings were fabricated via a laser-induced chemical transformation of a precursor mixture containing urea and boric acid. The precursors were melted and applied to native poplar wood. A commercial CO_2-laser (10.6 µm) was employed to trigger the *in-situ* conversion to h-BN, at varying laser powers to investigate the effect of energy density on h-BN formation. Following laser treatment, samples were hot-pressed to remove unreacted residues. Structural and chemical characterization was conducted using Raman spectroscopy, SEM, and HR-TEM, while fire performance was evaluated through direct flame exposure tests.

Key Results: Raman spectroscopy and HR-TEM confirmed the successful formation of h-BN across the wood surface. HR-TEM analysis revealed nanocrystalline domains with a (002) lattice spacing of 0.33nm, consistent with high-quality hexagonal stacking. An optimal laser power was identified, yielding high h-BN conversion while maintaining the wood's structural integrity. Single-flame tests demonstrated the exceptional efficacy of the ceramic coating: while native wood ignited immediately, the h-BN-functionalized wood remained structurally unchanged and showed no signs of combustion after 30 seconds of direct flame contact.

Conclusion: This research introduces a novel approach for a direct fire-retardant treatment of timber. By achieving precise control over the chemical reaction, the laser-induced synthesis provides a pathway to create high-performance h-BN coatings. This rapid and precise functionalization method offers a scalable solution for enhancing the safety and durability of wood-based materials in the modern construction industry.

KEYWORDS

Hexagonal boron nitride, laser-induced synthesis, wood functionalization, fire retardancy, ceramic coatings

REVALORIZATION OF TOMATO AGRI-FOOD RESIDUES AS A SOURCE OF CELLULOSE AND CELLULOSE NANOCRYSTALS (CNCS)

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ABSTRACT

Cellulose is the most abundant biopolymer in the biosphere and the main component of the plant cell wall. Due to its high availability, it constitutes one of the most widely used raw materials at the industrial scale, with applications in sectors such as medicine, cosmetics, and food packaging. Traditionally, cellulose is obtained from woody plants specifically cultivated for this purpose. On the other hand, tomato (*Solanum lycopersicum*) is one of the most relevant crops in Spain, with an annual production of around 4.5 million tonnes. Agri-food residues generated during its cultivation and processing represent a promising raw material for the production of cellulose and cellulose nanocrystals (CNCs).

In this context, the present work focuses on the valorization of tomato-derived residues as a source of cellulose, within the framework of a circular bioeconomy strategy. To this end, cellulose was isolated and purified from three tomato fractions (leaf, stem, and fruit peel) using a subsequent chemical protocol with toluene:ethanol, $NaClO_2$ under acidic conditions, and KOH to remove extractives, lignin, and hemicelluloses and pectin, respectively. The obtained cellulose fraction was characterized by attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR), thermogravimetric analysis (TGA), and size exclusion chromatography (SEC). The results showed that cellulose from the stems exhibited longer polymer chains and higher thermal stability, whereas cellulose from the peels presented shorter chains and lower thermal stability. In a second step, CNCs were obtained from the isolated cellulose via acid hydrolysis and characterized by ATR-FTIR, X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and light scattering. The results

indicated that CNCs from tomato peels were smaller, more homogeneous and with higher colloidal stability, whereas stem-derived CNCs were larger, more heterogeneous, and less stables. In conclusion, tomato agri-food residues were demonstrated to be a promising source of cellulose and CNCs. Furthermore, the results highlight that cellulose properties strongly depend on the plant fraction, which is a key factor to consider in the design and development of cellulose-based materials.

KEYWORDS

Cellulose, Cellulose nanocrystals, Circular bioeconomy, Tomato agri-food residues.

FUNCTIONALIZED MICROCRYSTALLINE CELLULOSE AS A REINFORCING AGENT FOR TOMATO POMACE-BASED COATINGS

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ABSTRACT

Metal food packaging is essential for preserving food over long periods and protecting it from external agents. To prevent the direct contact between metal and food and the potential corrosion and release of toxic substances, metal is coated with lacquers typically made of Bisphenol A (BPA) as a protective barrier. However, the use of BPA has been associated with adverse health effects and, for this reason, it has been banned in the European Union, creating the need for safe alternatives.

In this work, we propose the surface functionalization of microcrystalline cellulose (MCC, a food-contact-approved additive) with different acyl chlorides (palmitoyl and oleoyl chlorides). This surface modification was achieved under reflux with toluene, pyridine, and the corresponding acyl chloride. The resulting products were then purified using Soxhlet with acetone and water, obtaining final yields of about 80%. In a second step, palmitoyl and oleoyl modified MCC were mixed separately with the lipid fraction of tomato pomace (the main residue of the tomato processing industry) and coatings were prepared by electrospray and melt-polycondensation. After that, the effect of the addition of modified cellulose was studied by determination of the polymerization kinetics, thermal stability through thermogravimetric analysis, and surface morphology by Scanning Electron Microscopy and Optical Microscopy. Finally, the suitability of these new bio-based coatings as food packaging materials was assessed by sterilization and migration tests with different food simulants. In view of the results, the addition of functionalized MCC to the lipid fraction of tomato pomace represent a promising and sustainable alternative to BPA-based coatings for metal food packaging.

KEYWORDS

Functionalized Microcrystalline Cellulose; Metal food packaging; Bio-based coatings; Tomato Pomace.

LOW-TEMPERATURE ELECTROSTATIC SPRAY DRYING OF ESSENTIAL-OIL-LOADED EMULSIONS STABILIZED BY FOOD WASTE-DERIVED CHITOSAN NANOPARTICLES

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ABSTRACT

Each year, millions of tons of residual seafood shells are generated worldwide, creating a largely underutilized waste stream rich in valuable biopolymers. Here, chitin-derived chitosan extracted from lobster shells was investigated as a Pickering emulsifier for producing essential-oil-loaded powders. Nanoparticles with controlled degrees of deacetylation stabilized oil-in-water emulsions that were converted to powders using electrostatic spray drying (ESD) compared to conventional spray drying (SD) as benchmark. ESD uses a strong electric field to atomize charged droplets and enable rapid solvent separation under inert, low-temperature conditions, producing dry powders while minimizing thermal stress. We compared encapsulation efficiency, droplet size on reconstitution, powder morphology, and chemical preservation of volatiles, alongside accelerated storage tests. Across formulations, powders produced by ESD consistently outperformed SD: ESD powders showed higher encapsulation efficiencies, finer and narrower droplet-size distributions after reconstitution, and markedly improved powder morphology with fewer fused or collapsed particles. Crucially, GC–FID analysis revealed significantly greater retention of key volatile compounds and fewer thermal degradation markers in ESD powders, indicating superior preservation of essential-oil quality. Accelerated stability studies confirmed prolonged retention of oil content and functional activity for ESD samples. These results demonstrate that lobster-derived chitosan nanoparticles combined with low-temperature ESD form a scalable, sustainable route to shelf-stable powders that better preserve labile essential oils, an attractive platform for food, cosmetic and agrochemical applications where volatile retention is critical.

KEYWORDS

Electrostatic spray drying, chitosan, Pickering emulsion, essential oil, food waste

THE CHEMICAL FATE OF GALLIC ACID AND ITS AMIDE DERIVATIVES USED AS STABILIZERS UNDER LYOCELL PROCESS CONDITIONS

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ABSTRACT

The lyocell process is an environmentally friendly and sustainable industrial technology for high-performance man-made cellulosic fiber production. It is based on the direct dissolution of up to 15 wt% cellulose in aqueous *N*-methylmorpholine *N*-oxide (NMMO), a non-toxic, biodegradable, and >99.8 % recyclable solvent, followed by regeneration of cellulosic fibers in a water bath *via* dry jet-wet spinning.

Despite its advantages, NMMO is a strong oxidant and promotes undesired side reactions in the spinning dope under lyocell conditions. These include autocatalytic decomposition of NMMO, the formation of reactive intermediates such as free radicals, formaldehyde, and *N*-(methylene)morpholinium ions. These processes can negatively affect process stability and fiber quality, making the use of stabilizing additives essential.

Gallic acid propyl ester, *i.e.*, propyl gallate (PG), is currently used as a stabilizer due to its activity against radicals and reactive intermediates but also transition metal ion complexation. However, PG also contributes to secondary reactions, leading to the formation of additional chromophores, such as ellagic acid (EA) and its corresponding quinone derivatives, which cause discoloration of the spinning dope, ultimately reducing fiber brightness and increasing the need for fiber bleaching.

To address these limitations, gallic acid and gallic acid amides have emerged as promising alternative stabilizers. While maintaining comparable stabilizing efficiency to PG, their reduced chemical activity could limit chromophore formation and associated side processes. Consequently, their application could enhance fiber brightness and reduce dope contamination, offering a viable strategy for improved overall stabilization of the lyocell process.

In the current work, the stabilizing effect and chemical behavior of gallic acid and its amide derivatives compared to PG have been studied under lyocell conditions using calorimetry and HPLC-MS methods. The color generation in the spinning dope and during thermal and chemical degradation of NMMO was evaluated using UV/Vis spectroscopy. In addition, the solubility of EA and its oxidation products, their

adsorption onto cellulose fibers, as well as chemical behavior under peroxide bleaching conditions were studied.

KEYWORDS

cellulosic fibers, chromophores, lyocell process, stabilizers

REGENERATED CELLULOSE-CARBON ALLOTROPE COMPOSITES FOR SUSTAINABLE TRIBOELECTRIC NANOGENERATORS

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ABSTRACT

The increasing number of electronic devices in the era of Internet of Things (IoT) has led to a significant rise in energy demand, which conventional energy sources are struggling to meet. Therefore, developing sustainable, self-powered, and portable energy sources has become an urgent task for researchers.

Triboelectric nanogenerators (TENGs) have emerged as a promising technology for converting mechanical energy from friction, motion, or vibrations into electrical energy via contact electrification and electrostatic induction. In principle, a TENG operates using a pair of materials with different charge affinities, generating charges through contact electrification. When two materials come into contact, one donates electrons and becomes tribopositive, while the other accepts electrons and becomes tribonegative. Selecting an appropriate material pair is crucial, as it is one of the key factors affecting TENG efficiency. While much of the research in this field has focused on synthetic, oil-based polymers, cellulose is gaining attention as a sustainable alternative.

Although cellulose has shown good potential as a tribolayer, various chemical and physical modifications have been applied to further enhance its triboelectric properties. Since both cellulose and carbonaceous nanoparticles can act as electron donors in TENGs, it is worth investigating the performance of cellulose-carbon nanoparticle composites.

In this study, cellulose pulp was dissolved in a cold-alkali aqueous solution and used as a matrix to incorporate different carbon allotropes, including nanodiamonds, multi-walled carbon nanotubes (MWCNTs), and graphene nanoplatelets, to systematically evaluate their effect on triboelectric performance.

The results showed that graphene-, MWCNT-, and nanodiamond-based composites improved the power output by up to 76%, 51%, and 40%, respectively, compared to pure cellulose (34.3 W m^{-2}). Physical and structural analyses, including dielectric spectroscopy, electrical conductivity measurements, scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD), FTIR, and Raman spectroscopy, were employed to investigate the structure–property relationships of the regenerated cellulose-carbon allotrope composites. This comprehensive study paves the way for the use of regenerated cellulose-carbon allotrope composites in advanced applications.

KEYWORDS

Triboelectric Nanogenerators (TENGs), Cellulose-Composites, Carbon Allotropes, Dielectric Properties.

CIRCULAR AND SCALABLE LEATHER ALTERNATIVES FROM FUNGAL FRUITING BODIES

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ABSTRACT

Fungal leather alternatives intended to imitate animal leather are popular with fashion designers. Most fungal leather alternatives rely on 'as-grown' mycelium networks, which are time-intensive to grow and difficult to scale. We present a leather alternative designed for scalability, utilising mushroom residues and papermaking methods. Flexible chitin-glucan sheets were produced from *Agaricus bisporus* mushrooms with tensile strengths up to 20 MPa, values approaching those of synthetic and bovine leather (~13 MPa and 10–31 MPa, respectively), and strain-to-failure of 16–18% (compared to 15–60% and 31–69%, respectively). Sheet aesthetics and mechanical properties were tailored through modified fungal pulp extraction, dyeing, and reinforcement with cellulose-based fibres to achieve tear strengths comparable to bovine leather (104–107 N/mm compared to 42–106 N/mm). Mushroom residue-based sheets demonstrated a solution aligned with the principles of circularity, converting underutilised by-products into wearable garments to reduce waste and resource use in the fashion and textile industries.

KEYWORDS

Leather alternatives, bio-based, chitin, textile

UNDERSTANDING AND CONTROLLING GRAFTING ONTO CHITOSAN THROUGH STRUCTURE-PROPERTY RELATIONS

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ABSTRACT

Chitosan is an abundant and renewable waste that can be used in different fields, such as agriculture, medical or food industry, etc.[1] However, its natural variability, the difficulty to characterize it, and its poor solubility currently limit its broad use.[1]

The objective of this work is to establish relationships between chemical composition and hydrodynamic volumes of chitosan on the one hand and chemical compositions and hydrodynamic volumes of these chitosan after chemical modification. To fulfill this objective, the aim is to develop new characterization methods for chitosan and chemically modified chitosan.

To determine the hydrodynamic volumes and compositions (degree of acetylations (DA)) of different chitosan batches, Taylor dispersion analysis (TDA),[3] free solution capillary electrophoresis (CE)[2], and NMR spectroscopy (^{1}H, ^{13}C, DOSY, etc.)[4] are explored and compared to size exclusion chromatography (SEC) results. SEC is the most widely[4] used separation and characterization method for chitosan. However, filtration can alter the sample.[5] It is mandatory before SEC experiments but not before CE and TDA ones. Therefore, our first task was to compare the chemical compositions and hydrodynamic volumes of filtered and non-filtered samples using CE and TDA.

One of our secondary objectives is to enhance the solubility of chitosan at neutral pH. To achieve this, we envision several strategies to graft hydrophilic polymers onto the chitosan's backbone.[6] The resulting grafted chitosan will be fully characterized by means of ^{1}H NMR, CE, TDA, etc.

KEYWORDS

Chitosan, Filtration, Capillary electrophoresis (CE), Taylor dispersion analysis (TDA), Grafting

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LABELING OF CELLULOSE NANOCRYSTALS; AN ATOMISTIC VIEW

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ABSTRACT

Cellulose nanocrystals (CNCs) are notoriously difficult to visualize at the molecular level because they are composed of light elements and are highly beam sensitive. Recent cryo-STEM work demonstrated that exchanging CNC counterions for high-Z cesium can "label" surface sulfate groups and dramatically enhance electron contrast, enabling direct structural readout from CNC hierarchies [1].

Building on this experimental premise, we use all-atom molecular dynamics to ask a complementary question: which alkali ions are most likely to act as robust, information-rich labels at the CNC–water interface? We compare Li^+, Na^+, K^+, and Cs^+ through a unified set of descriptors spanning interfacial residence times, binding and rebinding statistics, and their coupled impact on hydration structure (water density modulation and local molecular ordering). Finally, adsorption free-energy calculations provide a thermodynamic baseline for ranking ion affinity.

Together, these measures converge to a coherent picture in which ion size and hydration competitiveness govern labeling propensity, highlighting conditions under which heavy-atom tagging can be both persistent and minimally disruptive, thereby guiding the rational choice of ionic labels for CNC imaging and interface engineering.

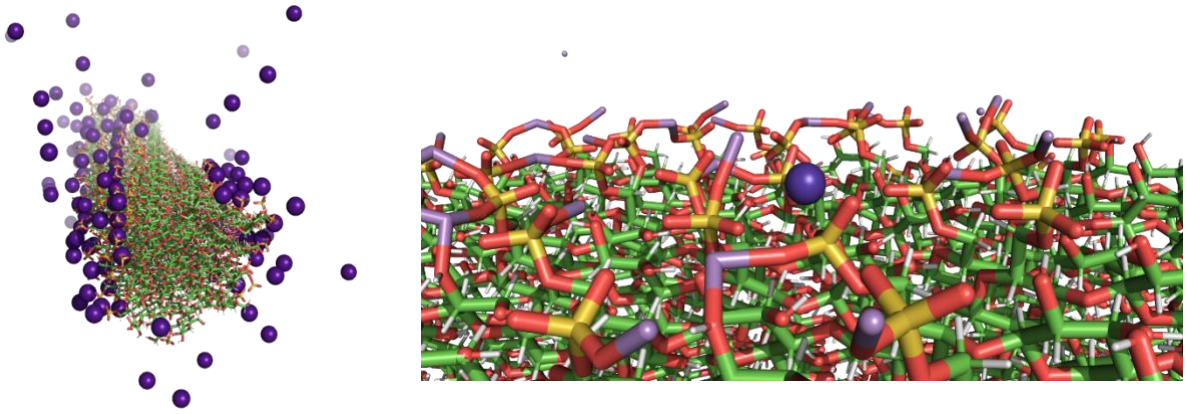

Figure 2 Front view (left) and close view (right) of a sulfated CNC labeled with Cs^+ counterions. Cs^+ shows reversible adsorption near C6 hydroxymethyl groups, highlighting continuous binding–unbinding at the CNC–water interface.

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KEYWORDS

CNC Labeling, Adsorption Behaviour, Molecular Dynamics Simulations

THE EFFECT OF THE TREATMENT HISTORY ON THE GELATION OF NANOCELLULOSE HYDROGELS

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ABSTRACT

Introduction: TEMPO-oxidized cellulose nanofibrils (TCNF) are promising materials for biomedical applications due to their high aspect ratio, tunable surface chemistry, and ability to form hydrogels at very low solid contents. Although surface charge density, hornification, and mechanical treatments are known to influence TCNF network formation, their combined effect on gelation behaviour is not well understood.

Objectives: In this study, we examine how TEMPO-oxidized cellulose nanofibril (TCNF) surface charge density (degree of carboxylation) and sonication jointly affect hydrogel gelation behaviour and viscoelastic transitions at low solid contents.

Methods: Three TCNFs with low, medium, and high charge densities (705 - 1205 mmol kg^{-1}) were prepared from dried cellulose pulp and analysed in non-sonicated and sonicated states. Oscillatory rheology (frequency sweeps) was performed for 0.1–1.0 wt% suspensions to identify the transition from viscoelastic liquid to viscoelastic solid, using frequency-independent loss tangent (tan δ) as the critical-gel criterion. Network morphology was assessed by light microscopy, SEM, and AFM.

Key results: A concentration range (rather than a single point) with frequency-independent tan δ was observed, indicative of self-similar relaxation behavior characteristic of a critical gel. Low-charge TCNF reached the critical gel state at lower concentrations, consistent with enhanced fibrillar aggregation, while higher surface charge shifted gelation to higher concentrations due to stronger electrostatic repulsion. Sonication increased the critical gel concentration for medium- and high-charge TCNF, with weaker effect on low-charge samples.

Conclusion: Microscopy (light microscopy, AFM, SEM) confirmed continuous fibrillar networks with embedded aggregates across all samples. The observed self-similarity is attributed to irreversible hornification in dried cellulose across multiple length scales, with gelation governed by a balance between electrostatic interactions and fibril entanglement; increasing charge density and (for medium/high charge) sonication both shift gelation to higher solid concentrations.

KEYWORDS

TCNF, gelation, hydrogels, self-similarity

FROM BY-PRODUCT TO HIGH-PERFORMANCE MATERIALS: ELECTROSPUN CASHEW GUM-BASED BLENDS FOR SUSTAINABLE PACKAGING AND AGRICULTURE APPLICATIONS

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ABSTRACT

Cashew gum (CG), an abundant polysaccharide obtained as an exudate from *Anacardium occidentale* trees, represents a promising by-product of the cashew nut industry with strong value-adding potential [1]. This study aimed to explore the feasibility of converting CG into functional materials using electrospinning, a scalable technique for producing continuous micro- and nanostructured polymer networks [2]. Since pristine CG does not exhibit adequate spinning behavior [1], its electrospinnability was achieved by blending it with suitable supporting polymers, yielding a set of tailored blends that enabled stable electrospinning. The resulting materials were characterized using various techniques, including scanning electron microscopy, tensile testing, and biodegradability analyses. The continuous films, obtained by annealing the fiber mats, were also characterized for their oxygen and water vapor barrier properties. Across the different formulations, the electrospun materials after annealing exhibited a combination of elasticity, with very high elongation at break (>250%), and excellent barrier properties (OTR < 5 $cm^3 \cdot m^{-2} \cdot day^{-1}$ at low relative humidity), consistent with the formation of densely packed polysaccharide-based networks. Preliminary biodegradation tests further confirmed an improved environmental profile, consistent with the gum's renewable origin. Overall, the findings demonstrate that CG can be effectively transformed into advanced electrospun and continuous structures with functional properties comparable to those of conventional polymers used in agricultural and packaging applications. By converting an existing agricultural by-product into high-value material, this work supports the integration of CG into circular bioeconomy strategies and highlights electrospinning as a versatile processing technique for developing next-generation sustainable materials.

KEYWORDS

Cashew gum, electrospinning, biopolymer fibers, stretchable materials, agro-industrial applications.

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ACKNOWLEDGMENTS

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MULTIMODAL AI–DRIVEN VIRTUAL SCREENING FOR TENSILE DESIGN OF CELLULOSE NANOFIBER–REINFORCED RUBBER COMPOSITES

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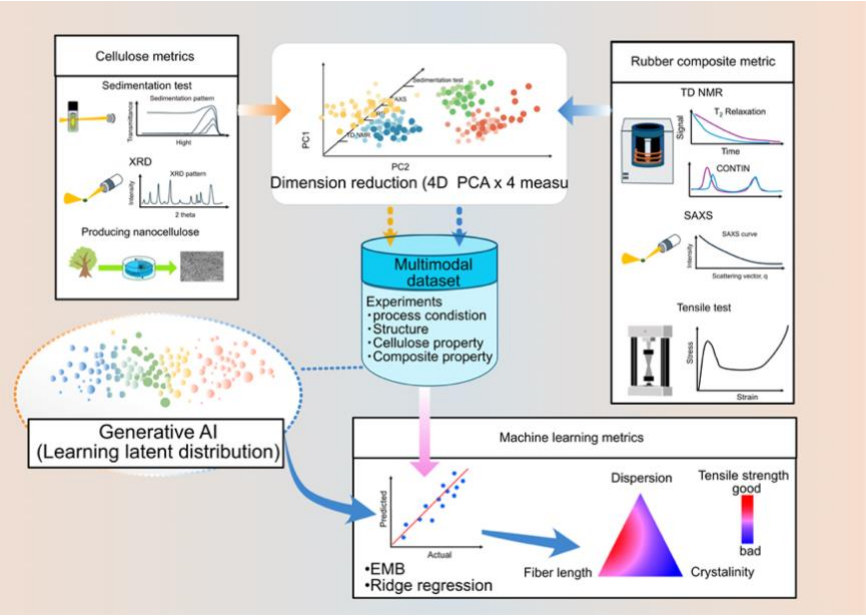
ABSTRACT

Cellulose nanofiber (CNF)–reinforced rubber composites can deliver high strength and toughness, yet rational formulation and processing remain difficult because the governing structure–property relationships are multiscale and nonlinear. Here we introduce a multimodal machine-learning framework that integrates heterogeneous characterization signals into unified descriptors to predict the tensile performance of CNF/natural rubber composites.

The reduced multimodal features are combined in a regression model that achieves reliable prediction across multiple tensile metrics. To explore material states beyond the measured set, we build modality-specific conditional generative models to create realistic pseudo-experimental datasets from material and processing parameters; models trained on these generated data reproduce prediction trends consistent with real measurements, indicating functional equivalence. The proposed workflow enables large-scale virtual experimentation for rapid screening and provides an interpretable, scalable strategy to accelerate tensile design of CNF-based rubber composites.



KEYWORDS

Cellulose nanofiber (CNF), Rubber composites, Multimodal machine learning, Virtual experimentation, Structure–property relationship, Generative modeling

MECHANOSORPTIVE CREEP RESISTANCE OF CORRUGATING-MEDIUM PULPS: THE ROLE OF HYGROEXPANSION AND NETWORK BONDING

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ABSTRACT

Mechanosorptive creep (MSC) — the accelerated time-dependent deformation of paper under combined mechanical load and cyclic humidity — is a primary cause of compressive failure in corrugated containerboard boxes during transport and storage. Although MSC has been studied for decades, its prediction remains difficult because the relative contributions of fiber-level hygroexpansion and network-level bonding are still debated. This study systematically compares the cyclic-humidity creep performance of four industrially relevant corrugating-medium pulp types: ammonium-base neutral-sulfite semi-chemical pulp (A-NSSC), sodium-base NSSC (Na-NSSC), eucalyptus high-yield kraft pulp (HY-KP), and recycled old corrugated containerboard (RP).

Laboratory handsheets were prepared at two controlled conditions: a fixed-freeness set (Schopper–Riegler 25°) and a fixed-density set (approximately 700 kg/m^3), achieved through PFI refining. This dual approach allows the separation of fiber-property effects from network-bonding effects on MSC. Compressive creep tests were conducted under cyclic humidity (50% ↔ 90% RH) until specimen failure. Hygroexpansive strain, sheet density, water retention value, and wet zero-span tensile strength were measured for each pulp–beating combination.

A-NSSC exhibited the highest MSC resistance across both beating sets, attributed to its low beating resistance and consequent ability to form denser, better-bonded fiber networks at moderate refining levels. MSC time to failure correlated strongly with sheet density (R^2 = 0.87 for virgin pulps), confirming that fiber–fiber bonding governs creep resistance. In contrast, hygroexpansive strain showed no positive correlation with MSC; pulps with the highest hygroexpansion (A-NSSC) in fact exhibited the longest creep times. This finding contradicts earlier literature that reported a direct positive link between hygroexpansion and MSC acceleration, and can be explained by the well-known co-dependence of hygroexpansion on sheet density: denser sheets expand more but simultaneously resist creep more effectively through superior bonding.

These results demonstrate that for corrugating-medium pulps, network bonding quality is the dominant factor controlling MSC, while hygroexpansion alone is not a reliable predictor. Practical improvement of creep resistance should therefore focus on enhancing fiber–fiber bonding and sheet densification rather than solely minimizing hygroexpansion.

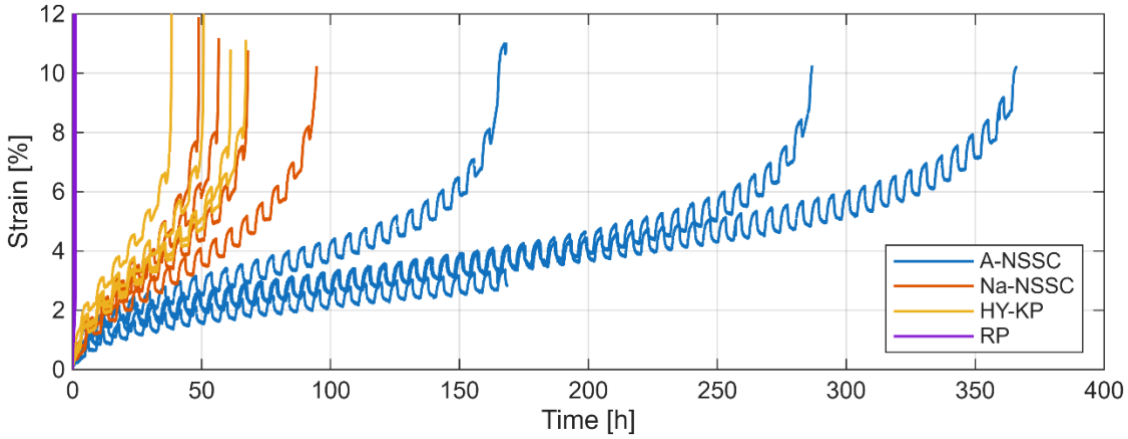

FIGURE 3 COMPRESSIVE CREEP STRAIN DEVELOPMENT UNDER CYCLIC HUMIDITY (50% ↔ 90% RH) FOR THE FIXED-FREENESS (SR 25°) SET. EACH CURVE REPRESENTS ONE SPECIMEN. A-NSSC (BLUE) SUSTAINED LOADING FOR UP TO 370 H, SUBSTANTIALLY OUTPERFORMING NA-NSSC (ORANGE, ~50–100 H)

KEYWORDS

mechanosorptive creep, corrugating medium, hygroexpansion, fiber bonding, cyclic humidity

LINKING CELLULOSE MICROFIBRIL ANGLE TO MECHANICAL BEHAVIOR IN SINGLE WOOD PULP FIBERS

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ABSTRACT

Cellulose-based materials are gaining increasing attention as renewable and recyclable alternatives to single-use plastics. To improve their performance in technically relevant applications, a better understanding of the mechanical behavior of the underlying cellulosic fibers is required. A key structural parameter is the microfibril angle (MFA), which strongly influences fiber stiffness, strength and failure behavior. However, reliable MFA determination at the single-fiber level remains challenging and comprehensive datasets linking morphology and mechanics are still scarce.

This work presents an experimental approach for the systematic characterization of single unbleached, unrefined kraft pulp fibers. The approach combines structural, geometrical and mechanical measurements to link fiber morphology with mechanical response. The MFA is determined using wide-angle X-ray scattering (WAXS), while the fiber geometry is obtained using an established microtome-based method. Furthermore, single fiber tensile testing will be applied to study the mechanical response of the fibers until failure and enable the direct linking of MFA and mechanical performance.

The proposed approach is designed to provide a more comprehensive experimental description of single-fiber behavior than currently available. By combining structural and mechanical characterization, it aims to establish a consistent dataset for a better understanding of paper-based materials and support more accurate modeling approaches of paper products.

KEYWORDS

Microfibril angle, WAXS, cellulose fibers, mechanical characterization

SELECTIVE FRACTIONATION OF CELLULOSE FROM TEXTILE WASTE VIA CARBAMATION AND NAOH DISSOLUTION

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ABSTRACT

In fiber-to-fiber recycling of textile waste, the presence of degraded fractions in recovered polymers will limit the implementation of efficient circular material strategies. This present work investigates a selective method for refining cellulosics recovered from textile waste by combining chemical modification and controlled dissolution.

The proposed strategy involves pre-swelling and carbamation of cellulose fibers using a deep eutectic solvent (DES) based on choline chloride:urea (ChCl:urea), under varying thermal conditions and reaction times. DES systems are considered environmentally favorable due to their low toxicity, low volatility and tunable solvent properties. In addition, these systems enable carbamation under milder conditions and may result in a reduction in overall energy demand. Subsequent dissolution in NaOH enables selective solubilization of low-molecular-weight cellulose carbamate fractions, while higher-molecular-weight fibers remain insoluble and can be recovered. The process is evaluated using gel permeation chromatography (GPC) and inverse size exclusion chromatography (ISEC) to determine changes in molecular weight distribution and porosity, while attenuated total reflectance (ATR) spectroscopy and nitrogen analysis measurements are applied to monitor chemical modification and the extent of carbamate formation.

The preliminary findings suggest that DES-assisted pre-swelling enhances fiber accessibility, thereby facilitating more targeted carbamation and controlled dissolution. Moreover, this approach enables the selective removal of lower-molecular-weight fractions without extensive degradation of the remaining fiber structure. The work involves both native and regenerated cellulosics, represented by cotton and viscose samples, to understand the influence of fiber type in process design.

In conclusion, the presented strategy aims for a more energy-efficient and adaptable recovery of cellulose from textile waste. The selective recovery of higher-molecular-weight fiber fractions will support their reuse in recycling processes and contributes to the development of more practical and scalable route for recovery of resources from textile waste.

KEYWORDS

Fractionation, textile waste, carbamation, cellulose

FUNCTIONALIZED NANOCELLULOSE IN MARINE SYSTEMS: ENVIRONMENTAL SAFETY, BIOLOGICAL RESPONSES, AND INTERACTION WITH VENLAFAXINE

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ABSTRACT

Objectives

The increasing use of nanocellulose-based materials raises concerns regarding their potential release into aquatic environments and the need for comprehensive ecotoxicological assessment. This study aims to evaluate the environmental safety of different nanocellulose systems (cellulose nanofibers (CNF), TEMPO-oxidized nanofibers (TOCNF), and cellulose nanocrystals (CNC)) using the marine bivalve *Mytilus galloprovincialis* as a model organism, including scenarios of single and combined exposure with the pharmaceutical contaminant venlafaxine (VEN).

Methods

Fluorescently labeled nanomaterials (Rhodamine B-functionalized CNF/TOCNF and CNC) were employed to investigate uptake, biodistribution, and biological responses in adult mussels under environmentally relevant (1 μg L^{-1}) and acute (1 mg L^{-1}) exposure conditions. Sub-lethal effects were assessed through biomarkers including lysosomal membrane stability, P-glycoprotein (P-gp) activity, and cholinergic enzyme activity. In parallel, embryotoxicity assays were conducted to evaluate the effects of single and combined exposure to nanocellulose and VEN.

Key Results

Both CNF and TOCNF were internalized by adult mussels, accumulating in gills and hemolymph. Sub-lethal effects included lysosomal membrane destabilization, inhibition of P-gp activity (more pronounced for CNF), and alteration of cholinergic enzyme activity. However, no significant oxidative stress or biotransformation responses were observed. CNC exhibited similar behavior, confirming a consistent pattern of low acute toxicity across different nanocellulose morphologies.

In embryo assays, CNF and TOCNF alone did not significantly affect development, except for a slight reduction in embryo size. In contrast, VEN strongly impaired larval development, likely through interference with serotonin-regulated pathways. In combined exposure scenarios, VEN-driven effects predominated, with no evidence of synergistic or antagonistic interactions, suggesting limited adsorption or carrier effects of nanocellulose in filtered natural seawater.

Conclusion

Nanocellulose materials, regardless of morphology or surface functionalization, exhibit low acute toxicity toward *M. galloprovincialis*. Conversely, co-existing contaminants such as venlafaxine pose a greater ecological risk and dominate mixture effects. These findings improve the understanding of nanocellulose behavior in marine environments and support future risk assessment under realistic exposure conditions.

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KEYWORDS

Nanocellulose, Ecotoxicology, *Mytilus galloprovincialis,* Marine environment.

THE STRUGGLE WITH LIGHT TRANSPORT IN LUMINESCENT TRANSPARENT WOOD

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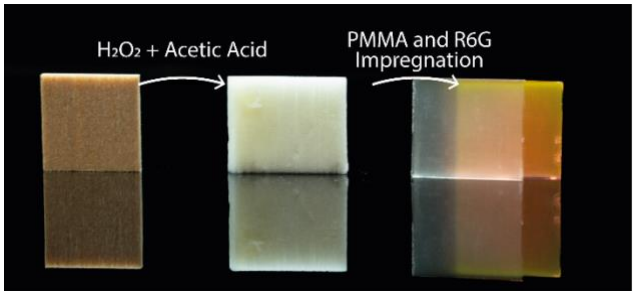
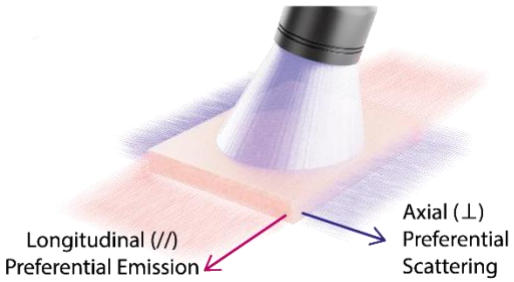
ABSTRACT

Luminescent transparent wood (TW) is a promising candidate for sustainable building-integrated photovoltaics, yet its complex anisotropic light transport behavior remains poorly understood. We report a Rhodamine 6G-functionalized TW luminescent solar concentrator (LSC) that challenges the prevailing assumption that longitudinal guided emission dominates performance. By resolving the interplay between anisotropic scattering and light transport, we demonstrate that for small device dimensions, light is mainly directed perpendicular to the fiber axis as transverse scattering outcompetes longitudinal waveguiding of emission. The cellulose scaffold however does show a substantial protective effect on the fluorophore, enhancing its photostability, while the non-functionalized TW control has no clear degradation after 500 h.

Beyond optical performance, a critical life cycle assessment (LCA) estimated a global warming potential of 17 kg CO_2 eq.·kg^{-1} TW, highlighting the burdens posed by the fossil-based matrix in TW.

This study thus exposes the limitations in TW composites that must be acknowledged and tackled, and provides the necessary framework for sustainable and efficient energy generation with TW devices.





KEYWORDS

Transparent Wood, Cellulose, Light Transport, Anisotropic Properties, Reproducibility

OPPORTUNITIES AND CHALLENGES OF HEMICELLULOSE RICH LYOCELL-TYPE TEXTILE FIBERS

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ABSTRACT

Man-made cellulose fibers (MMCF) such as Lyocell and Ioncell® are gaining attention as sustainable alternatives to synthetic fibers. However, most research has focused on cellulose-rich feedstocks, neglecting the role of hemicellulose—a key natural component of lignocellulosic biomass. This project explores the untapped potential of hemicellulose-rich kraft pulps in MMCF production, aiming to understand how hemicellulose influences fiber structure, dyeability, and mechanical performance. By incorporating higher hemicellulose content in the spinning dope and producing fibers via dry-jet wet spinning, the study investigates improvements in dye uptake, reduced fibrillation, and their implications for downstream processes like yarn spinning and fabric finishing. Preliminary results suggest that hemicellulose may offer functional advantages over conventional cellulose-only approaches. In terms of spinning, hemicellulose-rich raw materials may offer reduced energy demand and lower raw material costs, while expanding the range of suitable feedstocks and maintaining comparable mechanical fiber properties. Additionally, higher hemicellulose contents do not appear to negatively affect dyeability or color fastness. Future work will investigate the localization of hemicellulose and its potential effects on downstream fiber and fabric properties, such as fibrillation. This research contributes to the development of more sustainable and versatile textile fibers by leveraging the full chemical potential of wood-based raw materials. The anticipated insights could redefine material selection in MMCF production and support industry efforts to reduce reliance on synthetic fibers while enhancing textile quality and process efficiency.

KEYWORDS

Hemicellulose, Lyocell fibers, Man-made cellulose fibers, Sustainable textile, Fiber dyeability

MECHANOCHEMISTRY FOR BIOMASS VALORIZATION IN THE BIOECONOMY

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ABSTRACT

Lignocellulose source, as a high-potential renewable feedstock, holds a significant promise for the development of sustainable thermoplastic, bio-composite and textile materials. Among the available modification strategies, esterification is particularly important for introducing functional groups and tailoring material properties. However, conventional solvent-based modification methods often require large quantities of solvents and high temperatures, posing safety risks and increasing costs. In contrast, mechanochemical modification via ball milling has demonstrated its effectiveness in covalently modifying cellulose with target functional groups while minimizing solvent use and eliminating the need for heating. Building on these advantages, our work focuses on using mechanochemistry to modify lignocellulose materials for packaging applications and as additives in biocomposites. Beyond material functionalization, the reduced solvent demand and external heating highlight the potential of this approach for more sustainable processing. From an industrial perspective, this approach also considers the future scale-up of mechanochemical lignocellulose modification as a promising route toward the production of value-added bio-based materials.

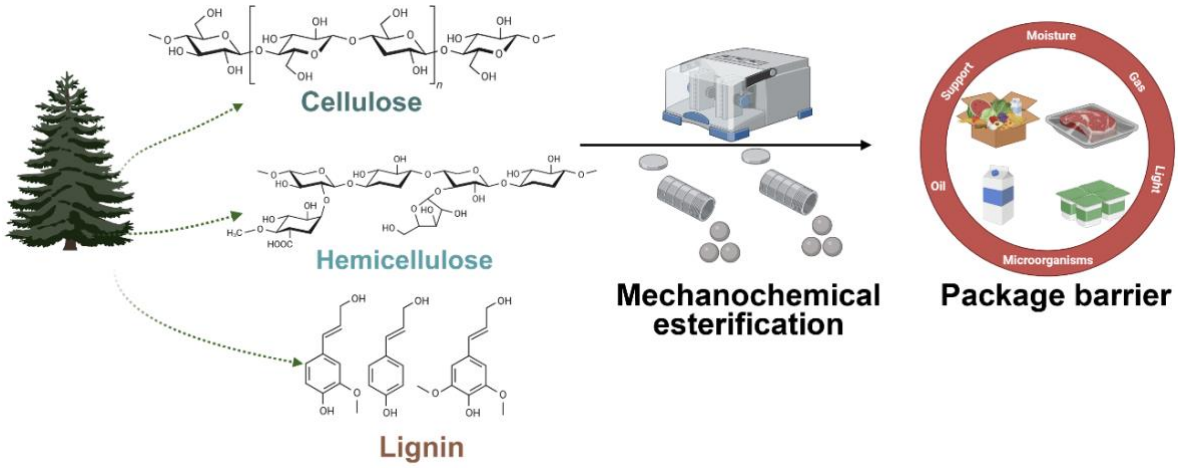

Figure 1. The roadmap of mechanochemical modification of biomaterial

KEYWORDS

Mechanochemistry, Lignocellulose chemistry

A NOVEL FUNGAL HYALURONATE LYASE ENABLES THE PRODUCTION OF STRUCTURALLY DIVERSE BIOACTIVE HYALURONAN OLIGOSACCHARIDES

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ABSTRACT

Hyaluronic acid (HA) is a key glycosaminoglycan whose biological functions are strongly dependent on its molecular weight, driving interest in the controlled production of defined hyaluronan oligosaccharides (HAOS) and HA fragments. However, enzymatic tools for HA depolymerization remain limited, particularly in fungal systems, where HA-degrading enzymes are poorly characterized. Thus, the aim of this work was to identify and characterize new fungal enzymes involved in HA degradation and to elucidate their mode of action.

A hyaluronate-degrading activity was identified in the secretome of *Purpureocillium lilacinum*, and the corresponding enzyme was heterologously expressed in *Pichia pastoris*. Sequence similarity network (SSN) analysis revealed that this enzyme does not cluster with any known polysaccharide lyase (PL) families in the CAZy classification, supporting its assignment as the founding member of a new PL family. Biochemical characterization using high-molecular-weight HA showed high catalytic efficiency and an unusual product profile. Reaction products were analysed by a combination of mass spectrometric and chromatographic techniques, revealing a heterogeneous mixture of predominantly unsaturated oligosaccharides with different degrees of polymerization, including both even-numbered and rare odd-numbered species. This distribution is inconsistent with classical exo-processive mechanisms and supports a non-canonical endo-processive mode of action. Notably, the resulting oligosaccharide mixtures exhibited antioxidant activity, highlighting their potential for biomedical and well-being applications.

In conclusion, this work identifies a novel fungal hyaluronate lyase that defines a new CAZyme family and exhibits an uncommon depolymerization mechanism, expanding the current understanding of HA degradation and providing a new biocatalyst for the production of structurally diverse and functionally relevant HAOS with potential applications in biomedical and well-being contexts.

KEYWORDS

Hyaluronic acid, hyaluronan oligosaccharides, hyaluronate lyase, antioxidant properties, fungal CAZymes

FUNCTIONAL FIBERS FROM BARK: TOPOCHEMICAL INVESTIGATION

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ABSTRACT

Forest-based biomass is uniquely positioned to supply sustainable materials through the higher-value use of side streams from pulp, paper, and sawmill operations. Among these side streams, pine bark is available at large scales and remains underutilized, often burned for heat and energy rather than valorized into advanced materials. Chemically, pine bark is rich in lignin, phenolic and lipophilic extractives, alongside cellulose, hemicellulose and pectin. Therefore, this material oKers an opportunity for a wide selection of functionalities.

Although decades of research on bark, the strategy of approaching it through the lenses of wood science where bark is narrowed down to just another lignocellulosic material, has overlooked its distinct anatomical, chemical and functional nature. Wet chemical analysis has shown big diKerences: while wood is chemically dominated by carbohydrates, bark is defined by lignin and extractives cross-linked with carbohydrate polymer matrix. The higher lignin content in addition to extractives, has been considered to make the fibers recalcitrant, but the origin and nature of this so-called recalcitrance has been barely touched upon. Characterization has been done to a high extent in bulk, but once the material is grinded and dissolved in a sequence of solvents, a whole dimension of data - the spatial information is lost. The distribution of bark's components at a cellular level, and its topochemical interlocking might hold the key to unlocking the full potential of this inherently multifunctional material. In this study we establish a multiscale investigation of native structure of bark fibers alongside its response to a selective removal of structural and non-structural components. The changes in chemical composition at a cellular level revealed by spectroscopic imaging are further linked to the three-dimensional structural impacts on morphological integrity by micro-computed tomography. Mechanical characterization showing how removal of diKerent components aKects load-bearing capabilities, is followed by a fiber liberation investigation. This perspective paves a way to harness the heterogeneity of bark fibres and build a valorization of the material based on its fundamental inherent structure.

KEYWORDS

Functional fibers, bark, topochemical analysis.

NANOCELLULOSE AEROGELS WITH CONTROLLED PORE ARCHITECTURES

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ABSTRACT

In this study, the pore structure of cellulose nanofiber (CNF) aerogels is tailored by controlling solvent crystallization. CNFs were produced from white corn cob as a sustainable cellulose source via mechanical nanofibrillation, yielding nanofibers with an average diameter of 11.1 nm. The resulting CNF suspensions were freeze-dried under controlled slow and fast freezing conditions, using aqueous tert-butyl alcohol (TBA, 0–40 wt%) to modulate crystallization behavior. Field-emission scanning electron microscopy (FESEM) showed that pore morphology of aerogels is strongly governed by solvent crystal structure, which depends on both freezing rate and solvent composition. Slow freezing produced large, open pores (up to 209 µm), while increasing TBA content significantly reduced pore size (down to 2.9 µm), reflecting altered crystal growth. In contrast, fast freezing yielded finer and denser structures (pore size 148–3.1 µm), indicating improved control over pore size distribution and structural uniformity.

KEYWORDS

Keywords: Cryogel, Cellulose nanofibers, Solvent crystallization, TBA.

ALL-CELLULOSE COMPOSITES AND CELLULOSE DERIVATIVES – AN APPROACH TO CLARIFY THE RELATIONSHIP BETWEEN PRECURSOR, ACTIVATED CARBON AND THE PERFORMANCE AS ELECTRODE MATERIAL IN SUPERCAPACITORS

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ABSTRACT

The high demand for energy storage systems means we need to develop new electrode materials that are highly abundant, easy to prepare and cost-efficient. Many different porous carbons produced from a high variety of biowaste materials, are used as electrode material in energy storage systems such as supercapacitors and lithium-ion batteries. However, the relationship between the precursor structure, the properties of the (activated) carbon, and the resulting performance as an electrode material has not yet been sufficiently clarified. The heterogeneity of various biowaste material in particular as well as the different activation and carbonisation routes makes i

t very difficult to establish clear correlations.

Therefore, we use cellulose in different shapes and sizes, combining them to create all-cellulose composites with defined precursor structures. These composites are made from pulp, microfibrillated cellulose, and cellulose microspheres. Cellulose derivatives with different functional groups are also used as a comparison for the precursor material.

The activated carbons derived from these cellulose composites reach specific surface areas of up to 2000 $m^2 g^{-1}$ and a high proportion of micropores, making them highly suitable for use as electrode materials in supercapacitors. In addition to the specific surface area and the pore structure, a large number of other factors influence the performance of the material in supercapacitors. The defined structures of the all-cellulose composite help identify the relevant parameters of the precursor material that influence the activated carbon and subsequent performance as an electrode material. Furthermore, it can be shown that composites prepared from materials with different structures exhibit enhanced electrochemical stability, particularly at higher current rates up to 10 A g^{-1}.

KEYWORDS

Cellulose composites, precursor structure, activated carbon, electrochemical energy storage

INFLUENCE OF ALKALI COUNTERIONS ON THE REFRACTIVE INDEX OF SULFATED CELLULOSE NANOCRYSTALS

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ABSTRACT

Cellulose nanocrystals (CNCs) are rod-like, highly crystalline nanoparticles. They are derived from cellulose, the most abundant biopolymer on Earth. Despite extensive work on CNCs, their refractive index (RI) is still not conclusively established. Reported values vary depending on the sample state and measurement method [1,2]. For sulfated CNCs, counterions are likely responsible for some of these variations. Counterions are bound to the sulfate half-ester groups and may modify the hydration environment at the CNC surface [6]. Understanding these interactions is relevant not only for RI measurements but also for fundamental CNC studies. Alkali ions can serve as labels in ultrastructural investigations, as we previously demonstrated [5].
This study examines how different alkali metal ions (from Li^+ to Cs^+, including H^+) as counterions on the CNC surface affect the apparent refractive index. To assess whether sample preparation contributes to the measured RI, several exchange procedures are compared. These include a dialysis-based route and two different cation-exchange resins [4]. For all procedures, pH is adjusted to 7, and ultrasonication is applied to reduce agglomeration, as this step highly influences the measured RI.
Two optical methods are used. In the first, an established dispersion-based approach measures CNC suspensions by refractometry at different concentrations. The data are then extrapolated to obtain an effective RI of the CNC in water [1,2]. In the second method, spin-coated CNC thin films are analyzed by surface plasmon resonance (SPR) in air and isopropanol. Unlike refractometry, SPR probes a substantially less hydrated state. A cellulosic layer was also probed as a binding layer between the gold substrate and the CNCs. This additional RI data on pure cellulose shows that CNCs have a lower RI than pure cellulose, as previously reported [3]. Our results further show that heavier alkalis, such as Cs-CNCs, exhibit a lower RI than H-CNCs and lighter alkali forms. Although the absolute RI values differ between refractometry and SPR, both methods show a comparable decrease of about 0.03 in the RI from H-CNCs to Cs-CNCs. In SPR, this relative difference appears at all three investigated wavelengths. Overall, these findings indicate that the absolute RI depends on the measurement environment, while

the relative counterion effect appears robust in the dataset. The different patches and exchange procedures only show minor variations in both measurement methods.

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KEYWORDS

CNC, Refractive Index, Counterions, Hydration, SPR

FLEXOELECTRICITY AS AN ELECTROMECHANICAL COUPLING IN SELF-STANDING CELLULOSE FILMS

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ABSTRACT

Flexoelectricity is a universal electromechanical coupling phenomenon arising from strain-gradient-induced polarization, making it attractive for mechanical-to-electrical energy conversion in next-generation functional materials. However, the flexoelectric behavior of natural polymers remains largely unexplored. Among these, cellulose is an abundant, low-cost, mechanically robust and biodegradable material, offering a promising platform for flexoelectric applications. Therefore, investigating the flexoelectric behavior of cellulose is an important step toward developing eco-friendly energy harvesters based on flexoelectricity. The main objective of this study is to understand the flexoelectric properties of self-standing films composed of a regenerated cellulose (RC) matrix and cellulose nanocrystals (CNC) with varying mass ratios. The flexoelectric response is investigated using a cantilever beam bending method in a trilayer configuration (top electrode/cellulose film/bottom electrode). In addition, the influence of CNC mass percentage on structural, morphological and dielectric properties is systematically examined. A phase analysis using XRD and FTIR shows that increasing the CNC content increases the cellulose I content; by comparing reflection and transmission XRD measurements, a strong in-plane orientation of the cellulose I phase is evidenced at higher CNC loadings. Morphological observations using SEM support these findings, revealing cross-sections of the RC/CNC composites with a layered structure at higher CNC loadings. The dielectric properties of the films show a low dielectric constant for bare RC films, while a significant increase is observed for RC/CNC composite films with optimal CNC content, which is $\sim 10^2$ times higher than conventional electroactive polymers such as PVDF. The same optimized composite also exhibits the highest flexoelectric coefficient, which is $\sim 10^3$ times higher than that of synthetic polymer dielectrics. These results demonstrate that incorporating CNCs into an amorphous RC matrix significantly enhances its flexoelectric response. This enhancement correlates well with the observed dielectric behavior, suggesting the formation of an optimized polarization network within the composite. Overall, this study highlights the flexoelectric behavior of cellulose films and their potential for application in electromechanical energy harvesting devices through flexoelectricity.

KEYWORDS

Cellulose, flexoelectricity, mechanical-to-electrical coupling

ROLE OF SEAWEED POLYSACCHARIDE ARCHITECTURES IN MODULATING DIGESTION BEHAVIOUR AND NUTRIENT BIOACCESSIBILITY

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ABSTRACT

Marine seaweeds are increasingly recognized as sustainable sources of proteins and structurally diverse polysaccharides. Although their amino acid profiles are comparable to those of conventional protein sources, nutrient bioaccessibility is often limited by the presence of complex cell wall matrices rich in non-digestible polysaccharides (dietary fibres). These polysaccharides, including cellulose, sulphated galactans, alginates, ulvans, amongst others, form hierarchical polymer networks that govern the physicochemical properties of the seaweed biomass and modulate digestion processes.

This study investigates how polysaccharide composition and architecture in three edible seaweeds from different groups (ULVA LACINULATA (GREEN), PORPHYRA DIOICA (red) and SACCHARINA LATISSIMA (brown)) influence protein digestibility. An adapted version of the INFOGEST *in vitro* digestion protocol was applied to better reflect the structural complexity of these matrices. Compositional analyses, including detailed monosaccharide profiling, were combined with spectroscopic and multi-scale structural characterization to elucidate structure–function relationships.

All species exhibited comparable amino acid profiles, with essential amino acids accounting for at least 29% of total content. However, protein digestibility varied significantly among species, ranging from ~60% in P. DIOICA to ~20% and ~10% in U. LACINULATA and S. LATISSIMA, respectively. These differences were closely associated with the nature and organization of the polysaccharide matrix. Galactan-rich networks in

P. DIOICA exhibited more permeable structures, whereas denser ulvan- and alginate-rich cell walls in U. LACINULATA and S. LATISSIMA limited enzyme diffusion, hindering protein digestion. Notably, polysaccharide-rich cell walls remained largely intact after digestion, as confirmed by spectroscopic and microscopic analyses, highlighting their resistance to enzymatic hydrolysis and their role as physical barriers.

Overall, these results demonstrate that seaweed polysaccharides are key components governing digestion through physical barrier effects and matrix interactions. Furthermore, the work provides key insights into the design of seaweed-based foods, highlighting the importance of tailoring polysaccharide architecture to modulate functionality and nutrient bioaccessibility.

KEYWORDS

seaweed polysaccharides, alginate, ulvan, porphyrans, microstructure

ECO-FRIENDLY AND AFFORDABLE APPROACHES TO MINIMIZE FIBRILLATION AND IMPROVE THE QUALITY OF LYOCELL FIBERS.

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ABSTRACT

In comparison to cotton fibers (Fibrillation: 2), Lyocell fibers tend to fibrillate significantly, with higher values reaching 4-6 with 6 being the maximum degree of fibrillation[1]. Fibrillation in Lyocell occurs when fibers experience mechanical stress, especially when wet, leading to microfibrils peeling off the fiber surface[2,3]. This tendency stems from the highly aligned cellulose crystals along the fiber's length and the amorphous regions between them, showing weak interactions with the crystallites[2,3]. Fibrillation can impact the fabric's appearance, causing reduced color vibrancy due to light scattering at the fibrillated surface[4]. Additionally, fibrillation contributes to fuzzing and pilling after washing, diminishing the garment's aesthetic appeal[3].

Crosslinking is a well-established method to prevent fibrillation, often using crosslinkers like 1,3,5-triacryloylhexahydro-1,3,5-triazine (TAHT) and dimethyloldihydroxy ethylene urea (DMDHEU) in the industrial manufacturing of low-fibrillating Lyocell fibers[5,6]. As these crosslinkers are toxic and release formaldehyde[7-9], alternative crosslinkers are explored to replace these chemicals, such as 1,2,3,4-butyltetracarboxylic acid (BTCA), citric acid (CA) and maleic acid. However, BTCA is more costly, citric acid causes yellowing, and maleic acid is less effective[8,9]. The yellowing concern of CA can be overcome by the usage of glycerol and xylitol and required to use softeners to preserve strength of fibers[8]. Yet the process required higher temperatures and pressurized reaction zone which is energy consuming[8].

In this study, we evaluate crosslinkers from different sources, such as pre-condensation methods with polycarboxylic acids, and achieve more controlled and effective fibrillation reduction under milder conditions. Moreover, the method of fibrillation analysis typically involves Scanning Electron Microscopy (SEM) and microscopy followed by image analysis to determine the Fibrillation Index, both of which often require enhanced manual effort[2]. Additionally, we aim to develop an automated, easy-to-use method for measuring fibrillation with the Valmet FS5 Fiber Image Analyzer.

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KEYWORDS

Fibrillation, cross-linking, pre-condensation

INVESTIGATION OF CHITIN-PROTEIN COMPOSITION IN CHITINOUS COMPOSITE MATERIALS OBTAINED FROM INSECTS

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ABSTRACT

Black soldier fly (BSF) farming represents a promising strategy for valorizing organic waste by converting it into high-value bioproducts, including proteins, fatty acids, and chitin, with applications in animal feed, agriculture, and the materials sector. Among these, chitin has attracted considerable attention as a versatile and sustainable resource; however, its extraction remains challenging due to intrinsically strong chitin–protein interactions in the insect matrix. This work investigates sustainable chemical treatments to selectively remove proteins from BSF biomass, yielding chitin-protein materials with tunable protein content. The chitin-to-protein ratio in these composites plays a critical role in determining their physicochemical properties and predicting performance in biomass-derived material applications. Quantification of both components therefore serves as a key indicator for tailoring the functional behavior of the resulting materials. ^{1}H NMR spectroscopy was employed to determine the chitin-to-protein ratio in the resulting materials. The composites were also investigated for material applications through the fabrication of papers and testing as adhesives. Mechanical and adhesion properties were evaluated to determine how differences in chitin-to-protein composition affect the performance of these insect-derived materials. This work supports the development of sustainable bio-based materials and contributes toward advancing a circular bioeconomy[1,2].

KEYWORDS

Acid hydrolysis, Chitin, Protein, ^{1}H NMR, Quantitative analysis

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SEMI-SYNTHESIS OF SULFATED β-GLUCANS: NEW CANDIDATES FOR IMMUNOLOGICAL STUDIES

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ABSTRACT

β-glucans are a class of homopolysaccharides widely distributed in nature, composed of a backbone of β-1,3 linked D-glucose (Glc) units. Many of these structures also feature D-Glc branches, typically via β-1,6 linkages. They exhibit several bioactivities including antioxidant, anticancer and immunomodulant properties. In particular, β-glucans are extensively investigated for their ability to activate and direct immune responses (1). Their potent immunomodulatory effect is mediated by interactions with key immune receptors and cells, which is closely related to differences in their structural characteristics (*e.g.* branching frequency, solubility, molecular weight, polymer charge, conformation in solution). Despite their therapeutic potential, the employment of native β-glucans in pharmaceutical field is often limited by some of their intrinsic properties (*e.g.* high molecular weight and poor water solubility) (2). Chemical modification arises as a useful strategy to overcome these limitations by introducing some functional groups to design new compounds with enhanced or novel functionalities. Through chemical modification, β-glucans with improved water solubility, reduced molecular weight and viscosity could be achieved. The most common methods of chemical derivatization include sulfation. Indeed, sulfated β-glucans result as promising bioactive compounds, with a number of studies demonstrating their antiviral, immunomodulatory, and drug delivery potential (3). Sulfated β-glucans are typically obtained through random sulfation reactions in which the regioselectivity of the sulfate groups cannot be controlled. In the recent years, several research efforts have been spent to develop methods for the regioselective sulfation of polysaccharides (4). To the best of our knowledge, regioselective sulfated β-glucans are poorly investigated; in this context, we developed different semi-synthetic pathways (5) for the sulfation of two different β-glucans to have access to a library of derivatives with unprecedented and well-defined sulfation pattern. We selected as starting materials two polysaccharides that, already in their native form, exhibit interesting bioactivities: curdlan, a linear β-glucan extracted from *Agrobacterium* strains and laminaran from *Eisenia bicyclis*, a branched β-glucan. Some of the obtained sulfated derivatives were also selected for a panel of immunological assays in order to define some structure–activity relationships (6).

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KEYWORDS

β-glucans, sulfation, regioselectivity, immunomodulant activity

CHITIN VALORIZATION VIA DEEP EUTECTIC SOLVENT FOR THE DEVELOPMENT OF ACTIVE FILMS

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ABSTRACT

In this work, chitin (Chi) was extracted from fruit fly pupae. The pupae were treated with NaOH (1 M) at room temperature, then filtered, and the solid fraction was decolorized using H_2O_2 at room temperature. After filtration, the solid was dried in an oven and milled to obtain a fine powder. In this chitin extraction procedure from fruit fly pupae, the demineralization process can be avoided, providing a more sustainable extraction method.

The extracted chitin was added to gelatin using a natural deep eutectic solvent (DES) composed of choline chloride and citric acid. The main objective was to valorize the chitin by overcoming its insolubility using a DES and to promote its reinforcing effect in active films. For that, chitin was dissolved in DES and Chi/DES was incorporated into gelatin at varying concentrations (20–80 wt %).

The resulting films were homogeneous, indicating effective chitin dispersion within the matrix. The incorporation of chitin significantly improved the film mechanical performance, revealing a transition from a brittle to a more ductile and flexible behavior.

In order to provide the films with bioactivity, grape marc extract (GME) was also valorized as a natural antioxidant and 1 and 2 wt % of GME were incorporated into the gelatin film forming solution with 40 wt % Chi/DES. The resulting active films demonstrated sustained release behavior, as shown in Figure 1, and significant radical scavenging activity (up to 73 %).

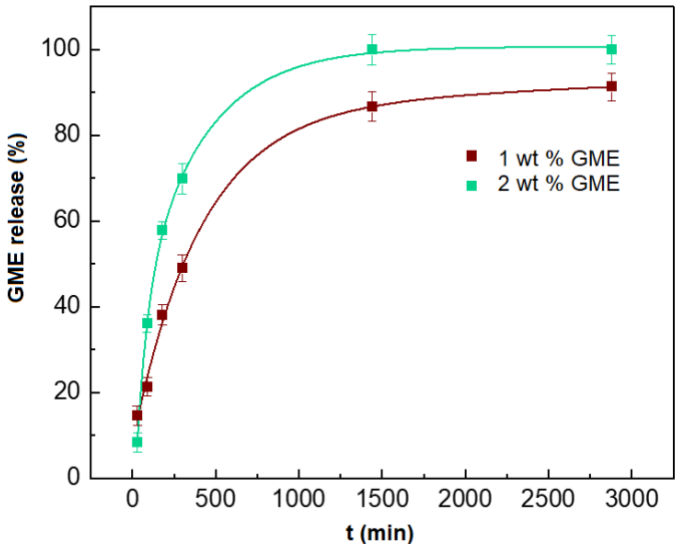

Figure 1. Grape marc extract (GME) release from gelatin films with 1 and 2 wt % GME.

KEYWORDS

Chitin, valorization, deep eutectic solvent, active films.

ELECTROSPINNING OF NANOCELLULOSE-STABILIZED GELATIN/DEXTRAN WATER-IN-WATER EMULSIONS: TOWARD STRUCTURED AND STABILIZED BIOPOLYMER NANOFIBERS

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ABSTRACT

Water-in-water (W/W) Pickering emulsions based on biopolymers offer a promising platform for fabricating structured, sustainable nanofibrous materials. Model systems such as polyethylene oxide (PEO)/dextran have been widely used to investigate phase behavior and emulsion formation. Building on this framework, we transfer the approach to gelatin (GEL)/dextran systems, where the introduction of a protein-based polymer enables additional intermolecular interactions and improved relevance for functional fibrous materials. The objective of this study is to investigate the electrospinning (ES) of GEL/dextran Pickering emulsions stabilized by cellulose nanocrystals (CNC), with a focus on emulsion stability, fiber morphology, and post-spinning stabilization. Systematic optimization has been initiated for GEL/dextran system at a 70:30 mass ratio, including adjustments to ES parameters to achieve continuous, defect-free fibers. Preliminary results indicate that stable emulsions can be obtained using CNC as a particle stabilizer, enabling the formation of electrospun fibers with tunable morphology. Confocal laser scanning microscopy is employed to visualize the phase distribution within the fibers and to distinguish between GEL and dextran domains, providing insights into core–shell or phase-separated structures. These observations are used to correlate emulsion properties with the resulting fiber architecture. To ensure structural integrity in aqueous environments, initial crosslinking of GEL-based fibers has been performed as a proof-of-concept, with ongoing efforts directed toward the development of non-toxic crosslinking strategies. Overall, this work translates insights from model W/W systems to GEL-based emulsions, enabling the development of stable and structured nanofibrous materials for potential biomedical applications.

KEYWORDS

Pickering emulsion; Gelatin; Dextran; Biopolymers; Electrospinning

IMPACT OF EXTRACTIVES ON THE ISOLATION OF HEMICELLULOSES FROM BIRCH AND SPRUCE

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ABSTRACT

Wood waste from forestry industry calls for sustainable valorization strategies. Hemicelluloses, are a major class of plant cell wall polysaccharides in wood, are gaining attention for applications in prebiotics and bio-based materials(Abik et al., 2023; Rao et al., 2023). However, their efficient isolation from lignocellulosic biomass remains challenging due to their association with lignin and cellulose, as well as the presence of wood extractives. These extractives, such resins, phenolics, fats, and other low-molecular-weight compounds, can hinder processing by limiting water accessibility and altering thermal reactions, particularly during subcritical water extraction (SWE).

Despite this, most studies rely on extractive-free materials, leaving limited understanding of how extractives, or their removal, affect hemicellulose extraction and quality. This study evaluates the impact of extractive removal on the yield, monosaccharide composition, and structural properties of hemicelluloses obtained via SWE, with a comparative focus on hardwood (birch) and softwood (spruce). Particular attention is given to how extractives influence extraction efficiency and whether the removal affects the structure of hemicellulose that is extracted out. To address this, both untreated and extractive-free wood samples were subjected to SWE under controlled conditions. The recovered hemicelluloses were characterized in terms of yield, monosaccharide composition, acetyl group content, and molecular weight distribution. Comparative analyses between birch and spruce were performed to assess differences related to biomass type.

The results demonstrate that the removal of extractives has only a minor impact on hemicellulose extraction performance. These findings suggest that the common practice of using extractive-free materials may not be necessary for efficient SWE-based hemicellulose recovery. Consequently, this study provides significant insights for optimizing extraction processes and supports the use of minimally processed biomass, which could simplify processing steps and improve the sustainability and scalability of hemicellulose valorization.

KEYWORDS

Xylan, Glucomannan, Extractives, Wood, Extraction.

References:

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HYDROPHOBIC CHITIN DERIVATIVE THROUGH STEARIC ACID GRAFTING FOR WOOD PROTECTION

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ABSTRACT

Chitin, a major component of the crustacean's exoskeletons, providing mechanical protection and structural support. Consequently, it is abundantly generated as a byproduct of the seafood industry. Despite its wide availability, its poor solubility in common solvents limits its processability [1]. Its conversion into chitosan is a common alternative. Strong alkaline treatments are applied to remove the acetyl groups leading to the formation of free amino groups, which are responsible of its water-soluble nature. In this study, we have sought for the chitin direct usage, taking advantage of its chemical hydrophobic nature, which leads to weak physical interactions with water. To improve its processability and enhance its hydrophobic properties, a bio-based hydrophobic coating was engineered by grafting apolar domains onto the hydroxyl groups of the polysaccharide (Fig. 1a). With this objective, shrimp-sourced chitin was modified through esterification with stearic acid while retaining the native acetyl groups promoting its inherent hydrophobic character. The grafting reaction was carried out at 60 °C using trifluoroacetic anhydride (TFAA) and methanesulfonic acid (MSA) as activating agents in the presence of stearic acid [2]. Different chitin:stearic acid molar ratios were tested by proportionally increasing the amounts of stearic acid, TFAA, and MSA. ^{1}H NMR analysis revealed a progressive increase in the degree of substitution (DS) with higher stearic acid content, reaching a maximum DS of 1.824 for the stearic acid-grafted chitin (SACH). Concurrently, the DDA increased from 8.23% to 38.25%, suggesting that higher MSA concentrations not only activate chitin hydroxyl groups but also promote partial deacetylation of the backbone. While the resulting free amine groups would be expected to increase hydrophilicity, this effect is outweighed by the increasing stearic acid substitution, which dominates the hydrophobic character of the material.

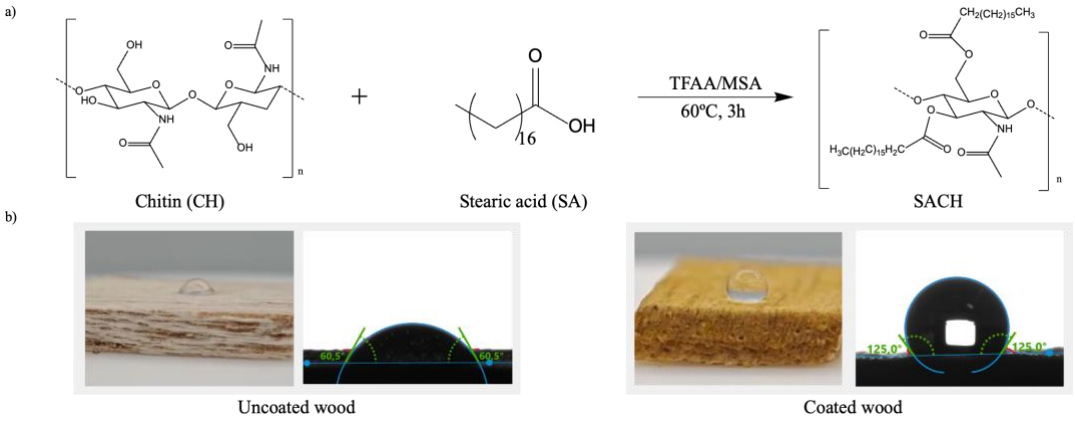

Figure 1. a) Grafting reaction of stearic acid on chitin. b) Wettability comparison between coated and uncoated wood (image and contact angle measurement).

The esterification reaction enabled the resulting SACH to be dissolved in DMSO, overcoming the long-standing processability challenge of the native chitin. Finally, the SACH/DMSO solution was applied onto balsa by dip-coating. The results from water contact angle measurements have indicated the successful conversion of the balsa wood surface into a hydrophobic one, increasing water contact angle from 61.83° to 121.96° (Fig. 1b).

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KEYWORDS

Chitin, Hydrophobic coating, Chitin functionalization, Surface modification, Esterification

TAILORING CYCLODEXTRIN–CURCUMIN INCLUSION COMPLEXES VIA ANHYDRIDE FUNCTIONALIZATION FOR IMPROVED PERFORMANCE

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ABSTRACT

Curcumin (CUR) is a natural polyphenol with well-documented antioxidant, anti-inflammatory, antimicrobial, and anticancer properties. However, its biomedical application remains severely hampered by poor aqueous solubility, low bioavailability, and rapid metabolism. In this context, cyclodextrins (CDs) have emerged as highly attractive drug carriers due to their cyclic oligosaccharide architecture, characterized by a hydrophilic external surface and a hydrophobic internal cavity that facilitates the formation of inclusion complexes with poorly water-soluble molecules [1]. Among these, β- and γ-cyclodextrins are particularly relevant due to their favorable cavity sizes and proven pharmaceutical applicability [2]. In this work, β- and γ-cyclodextrins were functionalized with maleic anhydride (β-/γ-CDM) or succinic anhydride (β-/γ-CDS) to investigate how external carboxyl groups influence their interaction with curcumin, as well as the physicochemical and biological properties of the resulting host-guest systems. The modified CDs were characterized by FT-IR, ^{1}H-NMR, TGA, and DSC, showing a degree of substitution ranging from 15% (for γ-CDM) to 40% (for β-CDS). Inclusion complexes were prepared via solvent evaporation and was evaluated through phase solubility studies, Job plots, and Benesi-Hildebrand analysis. The performance of these complexes was further assessed in terms of encapsulation efficiency, antioxidant activity, and antibacterial efficacy. Functionalization significantly improved the performance of the host-guest systems. While native β-CD increased curcumin solubility 11-fold, β-CDM and β-CDS yielded increases of approximately 25- and 70-fold, respectively. Notably, γ-CDs exhibited the most remarkable behavior, showing a linear solubility profile and a stability constant (K_s) of 1.66×10^4 M^{-1}, resulting in a 150-fold increase in curcumin solubility. Job plots and Benesi-Hildebrand analysis confirmed a predominant 1:1 stoichiometry. Solid-state characterization supported effective encapsulation, as evidenced by the disappearance of the curcumin melting endotherm. Inclusion efficiency peaked for γ-CD derivatives, reaching 25 ± 3% for γ-CDS_CUR. While maintaining antioxidant activity, the functionalized complexes exhibited significantly improved antibacterial activity, with MIC values reduced up to tenfold compared to free curcumin. Overall, these results demonstrate that succinylation is a superior strategy for enhancing the delivery and biological performance of curcumin, positioning γ-CDS as a promising platform for advanced therapeutic applications.

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KEYWORDS

Curcumin, Cyclodextrins, Inclusion complexes, Drug delivery, Functionalization

KINETIC INVESTIGATION OF CELLULOSE FIBRE CATIONISATION WITH CHPTAC

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ABSTRACT

Cellulose cationisation with 3-chloro-2-hydroxypropyl-*N*,*N*,*N*-trimethylammonium chloride (CHPTAC) is typically performed under alkaline conditions, with a CHPTAC-to-NaOH ratio of 1:2. This ratio provides the required alkalinity for the reaction and minimises the effect of alkaline hydrolysis, which is believed to be the main cause for the low reaction yield of less than 30 %. However, this explanation is inconsistent in some respects. For instance, starch cationisation exhibits higher reaction yields under comparable conditions with respect to temperature, alkalinity and the material-to-liquor ratio. Furthermore, the moderate to good stability of the reactive intermediate (epoxide) under alkaline conditions implies that rapid reagent hydrolysis is not the sole limiting factor in fibre cationisation. To advance beyond the current state of the art, it is therefore necessary to understand the role of the cellulose-NaOH interaction in the cationisation reaction. This raises the following key questions: is all the epoxide consumed at the end of the reaction and is NaOH uptake on the fibre decisive for the reaction yield?

For this purpose, the reaction rates of CHPTAC in the presence and absence of cellulose fibres were investigated by quenching the reaction at various time intervals and quantifying both the cationic species in solution and the CHPTAC bound to the fibre. An ion-exchange chromatography method was developed for quantifying the species in the treatment solution. Cationised fibres were analysed using ATR-FTIR, zeta potential analysis, and NMR measurements. Quantitative data were obtained from nitrogen analysis using the thermal combustion (Dumas) method.

The results indicate that fibre cationisation terminates early, with around 40% of the epoxide still available in the treatment solution. The early cessation of reaction is attributed to the consumption of active cellulose sites (i.e. deprotonated hydroxy groups) in the course of the reaction. Considering NaOH (on the fibre) to be consumed rather than acting as a catalyst improves the model fits and explains why the reaction yield increases at lower liquor ratios, since it was observed that the relative distribution of NaOH shifts towards the fibre when reducing the liquor content.

In order to improve the reaction yield, it is therefore necessary to increase the NaOH uptake on the fibre while minimising its concentration in solution. This can be achieved by reducing the liquor ratio. Other approaches, such as using a weaker alkali or additives, have not been successful in improving the yield.

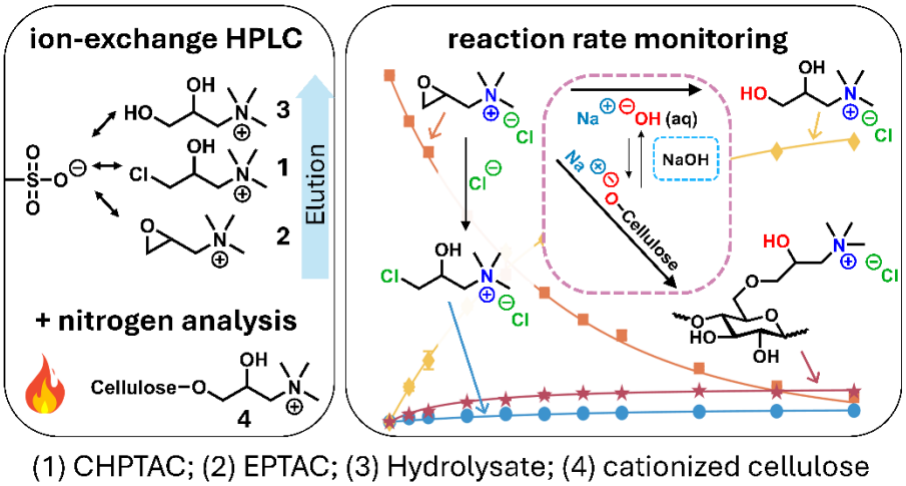

Figure 1. Graphical abstract.

Reference

Netzer, F., Manian, A. P., King, A. W. T., Bechtold, T., & Pham, T. (2026). Investigation of the reaction kinetics of 3-chloro-2-hydroxypropyl-N,N,N-trimethylammonium chloride (CHPTAC) with cellulose fibres. Carbohydrate Polymers, 379, 125021. https://doi.org/10.1016/j.carbpol.2026.125021

KEYWORDS

Cellulose, Cationisation, CHPTAC, Kinetic, IEX-HPLC

NANOSYSTEMS BASED ON HYALURONIC ACID AND CHOLESTEROL FOR THE DELIVERY OF ANTI-INFLAMMATORY THERAPEUTICS

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ABSTRACT

Rheumatoid arthritis (RA) is a chronic inflammatory autoimmune disease characterized by immune cell infiltration into the synovium and its hyperplasia. It leads to progressive cartilage destruction and bone erosion, resulting in joint ankylosis1. This project aims to design a novel nanogel (NHs) system based on an amphiphilic derivative of hyaluronic acid (HA) to deliver anti-inflammatory therapeutics for the treatment of RA. HA exploits the interaction with its receptor, CD44, which is over-expressed in activated macrophages involved in the inflammatory process. This strategy promotes better internalization of the drug into the cells and more effective treatment.

The HA backbone was derivatized with hydrophobic anti-inflammatory moieties and characterized by H-NMR, and FT-IR analysis. An MTT viability assay was performed on the macrophages RAW 264.7 to assess the safety of the NHs. Immune system cells were stimulated to induce inflammation using lipopolysaccharide (0.1 μg/mL), subsequently treated with the formulations. Cytokine levels (TNF-α and IL-6) were quantified by ELISA assay.

Successful functionalization of the polymeric derivative was confirmed by spectroscopic analyses. The derivatized polymer is capable of forming nanohydrogels via self-assembly2, with reduced size (163 ± 1 nm) and polydispersity index (PDI) (0.1). Cytotoxicity assays on the formulations demonstrated the cytocompatibility of the system up to a concentration of 200 μg/mL. The ELISA assay demonstrated the anti-inflammatory efficacy of the formulations and their ability to reduce intracellular cytokines during the early phase of the inflammatory response.

In conclusion, we show that the novel hyaluronic acid derivative NHs can deliver efficiently anti-inflammatory compounds and that the efficacy of the treatment in reducing the inflammatory cytokines is preserved when the drug is chemically linked to the polymer backbone in the NHs. Moreover, the presence of HA as the main component may represent a key advantage of the system, as its natural occurrence in the human body ensures high biocompatibility, while its specific interaction with HA receptor CD44 can promote receptor-mediated internalization of NHs into inflammation-activated immune cells. This feature highlights the potential of the developed nanocarrier as a safe and effective platform for targeted therapeutic delivery in rheumatoid arthritis.

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KEYWORDS

drug delivery, nanoparticles, hyaluronic acid, nanohydrogels, targeted delivery

STARCH HYDROLYSIS MODELLING: FROM FITTING IMPROVEMENTS TO THE FIRST PHYSICALLY CONSISTENT MODEL

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ABSTRACT

The enzymatic hydrolysis of starch, known as amylolysis, plays a fundamental role in both natural biological phenomena and in various industrial applications. Measuring the rate and extent of amylolysis has long been a subject of major interest in the research community, particularly due to its implications for human nutrition.

From a physicochemical perspective, amylolysis is a heterogeneous reaction, occurring at the interface between the solid phase formed by the starch granules and the liquid phase in which enzyme is conveyed. The most popular model to describe the amylolysis kinetics is the single exponential model

$$X_t = X_\infty\left(1 - e^{-k.t}\right), \qquad (Eq.\,1)$$

where X_t is the instantaneous hydrolysis extent at time t, X_∞ is the final starch fraction that is hydrolyzed by the enzyme, and k is the first-order reaction rate constant. While (Eq. 1) is simple, it fits very well hydrolysis curves (digestograms) with R^2-scores higher than 0.90 on average. However, this model does not take into account multiple mechanisms underlying the starch hydrolysis process, mainly that,

1. k is not constant through time,
2. at short term the dynamic is governed by surface-dependent factors,
3. k tends to a constant that reflect the inherent structure of starch.

Several models have been proposed in the literature to fit accurately the digestograms, unfortunately, the parameters of these models generally lack clear physical significance while slightly enhancing the R^2-scores of fits.

In our work, we highlight the importance of fitting combined instantaneous hydrolysis extent and its instantaneous reaction rate, denoted $k_a(t)$ and defined by

$$X_t = X_\infty\left(1 - e^{-k_a(t)\times t}\right) \Leftrightarrow k_a(t) = -\log\left(1 - \frac{X_t}{X_\infty}\right)t^{-1}, \qquad (Eq.\,2)$$

to easily distinguish models' performances and assess their physical consistency.

We develop and showcase, on 654 starch samples with several botanical origins and preprocessing, a new model that is physically consistent with amylolysis mechanisms that follows:

- Modelling explicitly a continuous change of the reaction rate coefficient over time.
- Being a generalization of (Eq. 1).
- Depicting both rapid-to-slow and slow-to-rapid, i.e. S-shape, kinetic patterns.
- Acknowledging that $k_a(t=0) = k_0 \in]0;+\infty[$ and $\lim_{t\to+\infty} k_a(t) = k_e \in]0;+\infty[$.

Our results show that our model is the only one that is physically consistent with the key mechanisms of starch hydrolysis, and that it better fits the hydrolysis curves, especially when examining both the X_t curve and the $k_a(t)$ curve, see (Fig. 1).

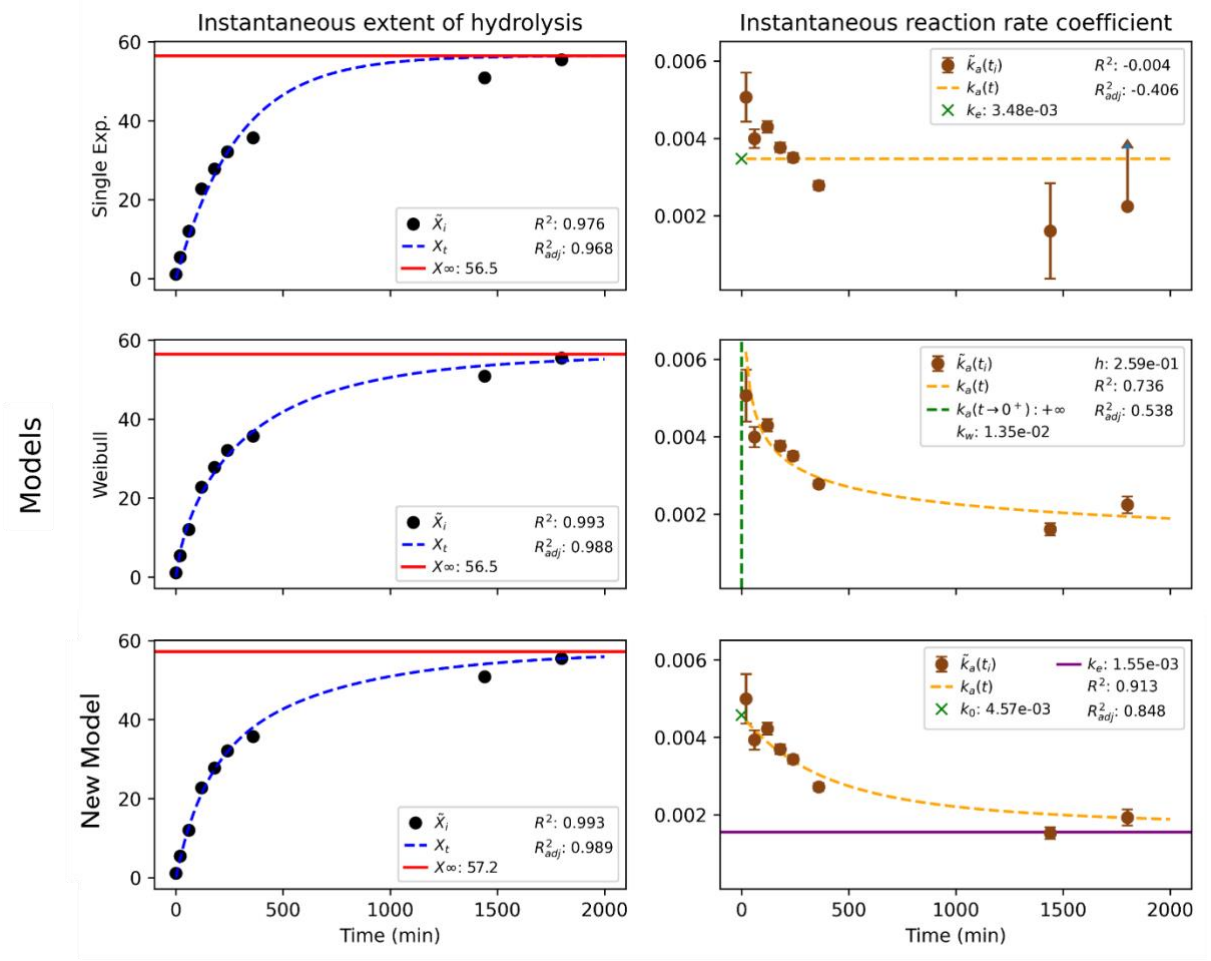

FIGURE 4 : EXAMPLE OF MODELS' FIT FROM A SAMPLE OF WHEAT STARCH. EACH ROW IS A DIFFERENT MODEL. THE LEFT PANELS DETAIL THE CALIBRATION BASED ON THE HYDROLYSIS DATA, WHILE THE RIGHT PANELS ARE ON THE INSTANTANEOUS REACTION RATE ASSOCIATED.

KEYWORDS

Amylolysis, Starch, Mathematical Modelling, Digestograms, Numerical simulations

EVALUATING ALTERNATIVE CULTURE MEDIA FOR ENHANCED BACTERIAL CELLULOSE PRODUCTION

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ABSTRACT

Bacterial cellulose (BC) is a promising biopolymer that has attracted significant attention in biomedical and advanced material applications due to its unique nanofibrillar structure, exceptional purity, mechanical strength, and biocompatibility [1]. BC is synthesized by non-pathogenic, aerobic Gram-negative bacteria, with KOMAGATAEIBACTER XYLINUS (*K. xylinus*) being the most widely studied and efficient producer due to its well-characterized metabolic pathways, stable cellulose production, and scalability [2]. To enhance BC production modifications of the conventional Hestrin–Schramm (HS) medium have been investigated. By contrast, studies combining HS with alternative microbiological culture media remains limited.

In this study, Lysogeny Broth (LB), Brain Heart Infusion (BHI), and de Man–Rogosa–Sharpe (MRS) media were evaluated as alternative culture media for *K. xylinus*. Each medium was tested individually, in different proportion mixtures with HS, and supplemented with 5% (v/v) glycerol as an alternative carbon source. BC production was quantified, and the resulting films were characterized for structural, physicochemical, and functional properties. Additionally, *K. xylinus* bacterial growth (CFU) and culture pH were monitored to evaluate their relationship with BC production.

Among the tested conditions, the HS:MRS medium supplemented with 5% glycerol yielded the highest BC production, achieving approximately a four-fold increase compared to the standard HS medium. Across all conditions, BC films retained comparable crystallinity, morphology, and chemical composition to those obtained under conventional conditions, indicating that material properties were not compromised (Figure 1). Glycerol supplementation consistently enhanced BC yield. Furthermore, the analysis of bacterial viability and pH evolution provided insights into the metabolic response of *K. xylinus* under different culture conditions and their relationship with BC production performance.

Overall, these findings demonstrate that alternative culture media, particularly when combined with glycerol supplementation, offer a robust and effective strategy to significantly enhance BC production without compromising material properties and performance.

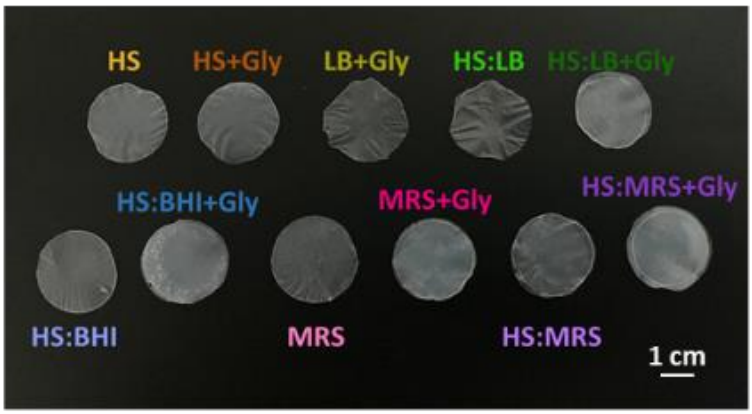

Figure 1. *Image of oven-dried BC samples in different media compositions showing different transparencies.*

KEYWORDS

Bacterial Cellulose, Culture Media, Biomaterials

CONTINUOUS CASTING OF GRAPE BYPRODUCT-BASED FILMS: SCALABILITY, MECHANICAL PERFORMANCE AND STABILITY UNDER SIMULATED SHELF CONDITIONS

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ABSTRACT

The development of biobased films from renewable resources has gained increasing attention as a sustainable alternative to conventional plastics. However, limitations related to mechanical performance, structural cohesion, and long-term stability still restrict their practical application. Incorporating lignocellulosic agro-industrial byproducts into polysaccharide matrices represents a promising strategy to tailor material properties and promote circular economy approaches. In this context, grape processing byproducts, rich in structural carbohydrates and phenolic compounds, stand out as multifunctional components.

This study investigated the structure–processing–property relationships of polysaccharide-based films incorporating grape byproducts, emphasizing scalability via continuous casting, mechanical performance, and stability under simulated shelf-storage conditions. Films were produced from grape pomace previously submitted to a mild acid pretreatment, combined with pectin (70 wt%) and carboxymethylcellulose (6 wt%), with glycerol (30 wt%) as a plasticizer, based on the total solids content of the dispersion. The film-forming dispersion was homogenized, deposited onto a moving polyester substrate, and dried under controlled conditions on a Mathis KTF-S-B continuous casting system.

Continuous casting enabled the production of homogeneous and cohesive films, demonstrating the feasibility of scaling up polysaccharide-based materials using an industrially relevant process. The developed films exhibited good mechanical performance, with tensile strength of 10 ± 2 MPa, elongation at break of 34 ± 5%, and elastic modulus of 39 ± 16 MPa, indicating a balanced combination of strength and flexibility. The materials also exhibited moderate surface hydrophobicity, with contact angles of 83 ± 5°.

Film stability was evaluated over 75 days under simulated shelf conditions, with samples exposed to ambient laboratory light to mimic retail display environments. Antioxidant activity decreased significantly over time, with DPPH inhibition dropping from approximately 49.7% to 18.7%, indicating degradation of phenolic compounds. UV-blocking properties remained stable in the UVB and UVC regions, while more pronounced changes were observed in the UVA range, accompanied by perceptible

color variation (ΔE > 3). These changes are influenced not only by light exposure but also by temperature, which plays a key role in accelerating the degradation of phenolic compounds and affecting the stability of the films under storage conditions.

Overall, the results demonstrate that incorporating grape byproducts into polysaccharide matrices enables the development of films with tunable properties. Continuous casting highlights the scalability potential of these materials, while storage conditions significantly impact their functional stability, underscoring the need to enhance the stability of bioactive compounds within the polymeric matrix to preserve functional performance over time.

KEYWORDS

Grape pomace, Scalability, bioplastics,UV-blocking, Shelf stability

STRUCTURAL ANALYSIS OF AMYLOSE *TRIS*(3,5-DIMETHYLPHENYLCARBAMATE) CRYSTALS. TOWARD ELUCIDATING THE CHIRAL RECOGNITION MECHANISM

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ABSTRACT

The physiological actions of bioactive compounds such as pharmaceuticals and agrochemicals are often chirality-dependent. Chiral resolution by high-performance liquid chromatography (HPLC) has thus been widely used to analyze the enantiopurity of chiral molecules, and cellulose or amylose phenyl carbamates [1] are commonly used chiral selectors (CSs) packed in HPLC columns. However, the detailed mechanism of chiral resolution via specific interactions between enantiomers and the CSs remains unclear. Only a few studies have investigated it at the molecular level [2,3]. A key step toward understanding these mechanisms is to investigate the crystallographic structure of CSs and their complexes with chiral compounds.

Amylose *tris*(3,5-dimethylphenyl carbamate) (ADMPC – Fig. 1a,b) with different degrees of polymerization (DP_n ≈ 30-1000) was crystallized for the first time from dilute solutions via controlled precipitation, using various combinations of solvents and precipitants. The resulting crystals were characterized using scanning and transmission electron microscopy (SEM, TEM), atomic force microscopy (AFM), optical microscopy (OM), as well as X-ray and electron diffraction (XRD, ED) (Fig. 1c,d).

Depending on the crystallization conditions, 3D flowerlike assemblies of lamellar crystals or individual hexagonal lamellae were observed. Long-chain ADMPC formed 40-nm thick lamellae, consistent with a chain-folded conformation, while decreasing DP resulted in thinner and more flexible structures. Two different structures were identified from powder XRD data, while ED patterns from dry single crystals suggested an orthorhombic or hexagonal unit cell. Although the temperature, concentration, and stirring primarily affected the morphology of the crystals and their 3D assemblies, the impact of the solvent and/or precipitant is still under investigation. In addition, cocrystallization experiments with enantiomers are underway to shed light on the molecular mechanisms of chiral discrimination.

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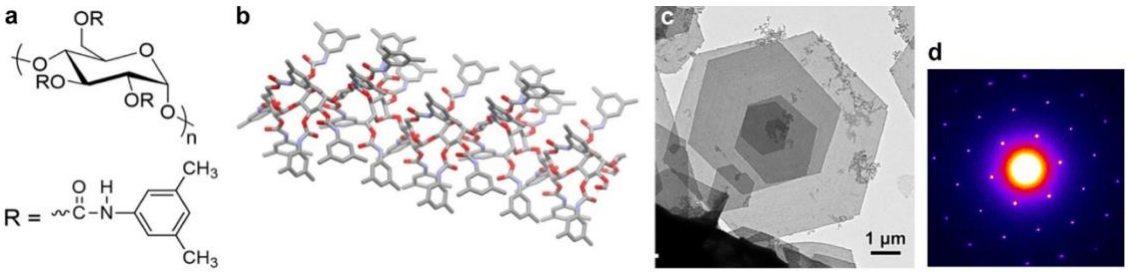

Figure 1. Chemical structure (a) and molecular model (b) [4] of ADMPC; c) TEM image of ADMPC single crystals (DP 1000 - solubilization in dioxane and precipitation with methanol); d) corresponding base-plane electron diffraction pattern.

KEYWORDS

Amylose *tris*(3,5-dimethylphenyl carbamate), Chiral selectors, Crystal structure, Electron diffraction, X-ray diffraction

SYNTHESIS OF FATTY ACID CELLULOSE ESTERS VIA REACTIVE EXTRUSION

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ABSTRACT

Cellulose is a renewable and abundant biopolymer with significant potential as a sustainable alternative to fossil-based materials. However, due to extensive inter- and intramolecular hydrogen bonding cellulose is insoluble in common solvents and lacks thermal transitions, which makes it unprocessable using conventional processing methods. Therefore, cellulose is typically chemically modified to achieve thermoplastic behaviour, commonly through esterification of hydroxyl groups. Reactive extrusion is a continuous mechanochemical process, in which the extruder acts as a chemical reactor under controlled temperature and shear conditions, representing a promising method for the rapid and solvent-efficient synthesis of cellulose esters. Furthermore, extruder design allows for the reagents to be introduced at different zones along the barrel, giving an opportunity of variation in feeding sequence.

The objective of this study is to investigate the effect of feeding sequence variability on distribution of key processes, degree of substitution (DS) and initial residence time during reactive extrusion of fatty acid cellulose esters (FACEs). It is hypothesised that changes in reagent introduction can majorly affect local mixing intensity and balance between dissolution and reaction stages.

Reactive extrusion is carried out using a co-rotating twin-screw extruder with systematically varied feeding configurations of solvent system and esterifying agent along the barrel, allowing further optimisation of FACE synthesis method. Feeding configurations include four distinct strategies: (I) delayed reagent addition after a swelling and dissolving stage, where reagent, solvent and cellulose are introduced separately from each other, (II) delayed reagent feeding into partially dissolved cellulose, (III) early-stage reagent introduction shortly after solvent addition, and (IV) simultaneous co-feeding of cellulose, solvent, and reagent in one feeder.

The resulting materials are characterised using Fourier-transform infrared (FTIR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, and rheological analysis.

Preliminary results indicate that feeding sequence strongly influences swelling of cellulose, reaction mixture thickening and homogenization along the screw profile. Earlier introduction of solvent and esterifying agent allows the reaction mixture thickening and homogenization stages to begin sooner, resulting in more uniform flow of material through the extruder die. Variation in the duration of

dissolution and reaction stages also significantly affects the homogeneity of final materials, which is reflected in rheological behaviour, where more homogeneous samples exhibit stable viscoelastic response and melt processability.

In conclusion, feeding sequence is identified as a crucial parameter in reactive extrusion of cellulose esters, affecting reaction progression and final material properties.

KEYWORDS

Reactive Extrusion, Twin Screw Extrusion, Cellulose Esters, Feeding Sequence, Degree of Substitution

HOPS TO SHEETS: TRANSFORMING BIOGAS RESIDUE INTO PAPER

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ABSTRACT

Humulus lupulus, commonly known as 'hops', is widely cultivated for use in brewing where it functions as a primary flavouring ingredient. However, only the plant's cones, which constitute 25 wt.% of the overall biomass, are used with the remaining 75 wt.% of stems and leaves typically incinerated or landfilled. Recent research has indicated that hops residues have high potential for energy generation in biogas reactors, presenting an obvious first valorisation step that utilises 40-70% of the initial total solids content. Volatile solids are largely removed, leaving low-grade ash, recalcitrant and inert solids with few valorisation paths. We sourced this biogas digestate and pulped it using acetosolv and traditional soda pulping processes to achieve long hop bine fibres, which were formed into sheets in analogy to papermaking. Pulping and papermaking technologies represent a viable method to valorise hops biogas digestates with otherwise limited scope of application and displace demand for traditional paper feedstocks, such as soft and hard wood fibre. In line with circular economy principles, sector coupling could see hops, a fast-growing plant with low land use requirements that does not compete with food production, utilised in brewing with its residues valorised in a two-step process, providing energy and paper products.

KEYWORDS

Hop, pulp, papermaking, hop bine fibers, biorefinery

PROMOTION OF PROLIFERATION AND DIFFERENTIATION OF PRIMARY HUMAN DENTAL PULP STEM CELLS ON SURFACE-MODIFIED NANOCELLULOSE SCAFFOLDS

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ABSTRACT

Dental pulp stem cells (DPSCs) have attracted much attention as a promising cell source for dental tissue regeneration; however, conventional *in vitro* culture relies heavily on animal-derived extracellular matrix (ECM) components, such as collagen and hyaluronan, which raise concerns regarding immunogenicity and lot-to-lot variability. To address these issues, wood-derived cellulose nanofiber (CNF) has emerged as an alternative scaffold material due to its structural similarity to ECM and tunable surface chemistry. Previous studies demonstrated that surface-modified CNFs can support cell adhesion and differentiation by structurally mimicking the ECM materials[1, 2]. Based on these findings, this study focused on evaluating the adhesion, proliferation, and differentiation of primary human DPSCs on surface-modified CNF scaffolds.

Unmodified CNF, TEMPO-oxidized CNF (TOCNF1.47, COO^-: 1.47 mmol/g), and phosphorylated CNF (P-CNF0.76 and P-CNF1.48, phosphate contents: 0.76 and 1.48 mmol/g) were used to prepare cast film scaffolds. Primary human DPSCs were cultured on these CNF scaffolds to evaluate the cell growth and differentiation behaviors.

After 72 hours of incubation, DPSCs failed to adhere to unmodified CNF and formed spheroids, whereas DPSCs adhered and proliferated well on surface-modified CNFs with appropriate amounts of functional groups. Notably, TOCNF1.47 supported the highest cell proliferation, suggesting that TOCNF provides a favorable microenvironment that structurally mimics the native ECM. Following 3 weeks of osteogenic induction, robust calcium deposition was observed on rat tail-derived collagen substrates used as controls. TOCNF1.47 exhibited only faint staining, whereas the P-CNF0.76 scaffold showed pronounced red aggregates with substantial calcium accumulation. No staining was observed under cell-free conditions, confirming that mineralization was cell-mediated. These results indicate that phosphate groups on P-CNF perform as nucleation sites for calcification and selectively promote hard tissue differentiation of primary human DPSCs. The present study demonstrates that surface functionalization of CNF critically modulates primary DPSC behavior and highlights the potential of CNF-based scaffolds for dental tissue engineering applications.

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KEYWORDS

Cellulose nanofiber, Surface modification, Cell culture scaffold, ECM mimicry

VALORIZATION OF INDUSTRIAL ALGAL RESIDUES INTO CELLULOSIC FIBERS: COMPARATIVE INSIGHTS FROM RED, BROWN, AND MICROALGAE

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ABSTRACT

Industrial processing of algal biomass produces cellulose-rich residues after the extraction of agar, alginate, or lipids. These side streams remain largely underutilized, and poorly characterized, despite representing a renewable, lignin-free, and structurally diverse source of fibrous materials. In this work, we investigate the composition, surface chemistry, and structure-property relationships of cellulosic fibers derived from three representative residues: red algae (*Gelidium corneum*, after agar extraction), brown algae (*Laminaria hyperborea*, after alginate extraction), and microalgae (*Nannochloropsis gaditana*, after lipid extraction). Mild chemical treatment was applied to remove impurities while preserving the fibrillar morphology and species-specific trace biomolecular components.

FTIR and compositional analyses confirmed cellulose as the primary constituent accompanied by residual biomolecules characteristic of each source such as sulfated galactans or uronic-rich polysaccharides. SEM revealed individualized micrometer-scale fibers with various topographies, and XRD indicated moderate differences in crystallinity and cellulose allomorph composition. Viscosity analysis provided approximate degree of polymerization. To examine how the residual non-cellulosic polysaccharides influence fiber behavior, their effects on dispersion and interfacial charge are evaluated through colloidal and zeta potential analyses, assessing surface functionality and stability in aqueous media. Mechanical testing and fracture imaging of the corresponding algal papers are conducted to relate fiber network integrity to compositional and morphological features, while moisture uptake and swelling experiments provide complementary insight into hydrophilicity and hygroscopic response.

Together, these comparative analyses aim to elucidate the species-specific characteristics of cellulose fibers from *Gelidium*, *Laminaria* and *Nannochloropsis* and show how trace polysaccharides from industrial seaweed residues might modulate the surface chemistry, dispersion behavior, and fiber-fiber interactions of algal cellulose. The results contribute to establishing a framework for understanding structure-surface-function relationships in marine-derived cellulosic fibers and support the development of sustainable, functional materials from algal side streams.

KEYWORDS

Cellulose, Algae, Structure, Property, Circular Economy

DESIGN OF FLEXIBLE BIODEGRADABLE BARRIER COATINGS BASED ON CELLULOSE NANOCRYSTALS

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ABSTRACT

Food packaging materials often require specific functional layers to ensure product preservation throughout their entire shelf life under controlled conditions. However, most of those commonly used layers are non-biodegradable or non-recyclable, becoming problematic once the packaging reaches the end of its life. In response to new regulations and environmental awareness, new strategies are being explored in the development of completely or partially bio-based packaging materials. One of the most promising of these bio-based coatings is cellulose nanocrystals (CNC), derived from cellulose, an abundant and biodegradable polysaccharide. Their very high oxygen barrier (oxygen permeability ≈ 10^{-21} $m^3.m/m^2.s.Pa$ at 0% RH, 23 °C[1]) and transparency once coated make them a promising candidate for food packaging applications[2]. Yet two significant limitations of CNC in this context are their moisture sensitivity that reduces their barrier properties after exposition and their high rigidity which leads to very brittle coating layers that are not suitable to replace conventional coatings on flexible materials.

The objective of the ongoing study is to identify ways to protect the CNC layer applied on flexible substrates to preserve the overall barrier of the structure even when exposed to mechanical constraints. To address this, our strategy consists in combining the CNC with a flexible biodegradable polymer, using multilayers coatings processes aiming at protecting the CNC from mechanical constraints and limiting cracks propagation, thus making it more suitable for food packaging applications.

To do so, poly(butylene adipate-co-terephthalate) (PBAT) was selected as the first polymer matrix to be tested due to its biodegradability, flexibility, and sealability. In this structure, the cellulose nanocrystals barrier layer is embedded between two PBAT layers, deposited on a polyethylene terephthalate substrate. First results indicate very significant reduction of oxygen permeance compared to the uncoated material, both prior to and following PBAT overcoating, indicating no significant degradation of the CNC layer performance through sequential coatings.

KEYWORDS

Packaging, Cellulose Nanocrystals, Barrier, Mechanical Resistance

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TOWARD ENERGY EFFICIENT CNF DESIGN THROUGH MACHINE LEARNING AND BAYESIAN OPTIMIZATION

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ABSTRACT

Cellulose nanofibers (CNFs) are one of the most promising advanced materials for supporting a circular bio-economy. However, their large-scale implementation remains constrained by the high-energy demand and lack of understating how processing conditions relate to material properties. Various routes for its production have been developed along the last decades, however these have been mostly studied or optimized mainly for the lab scale. For actual large-scale implementation, it is, therefore, of utmost importance to develop and establish data-driven strategies that are able to map complex process-property relationships and identify energy efficient operating conditions under pilot-scale production.

In this work, bleached eucalyptus kraft fibers were oxidized at three different levels (low, intermediate and highly oxidized) through TEMPO-mediated oxidation and subsequently fibrillated in a pilot-scale high-pressure homogenizer at three pressures (300, 600 and 900 bar) and four recirculation times (3, 6, 9 and 12 min). A total of 36 CNFs were produced and characterized in terms of optical transmittance, nanofibrillation yield and rheological properties. These data were then used to train a multioutput machine learning (ML) model based on an XGBoost regressor, using carboxyl content, pressure, and recirculation time as inputs and CNF properties together with energy consumption during fibrillation as outputs.

The final model showed excellent predictive performance, achieving test R2 scores above 0.92 for all seven predicted outputs. Bayesian optimization was then applied to the trained model to identify processing conditions that minimize the energy consumption while maximizing or minimizing targeted CNF properties, such as nanofibrillation yield and rheological behavior. The proposed approach enabled the identification of different energy efficient processing windows for producing CNFs with tailored characteristics, demonstrating the potential of machine learning assisted optimization as a practical decision support tool for more sustainable, large-scale CNF production.

Keywords

High-pressure homogenization, machine learning, nanocellulose, optimization

CHARACTERIZING THE USE OF CROP RESIDUES FOR BINDERLESS BOARD WITH SIMPLE MECHANICAL TEST

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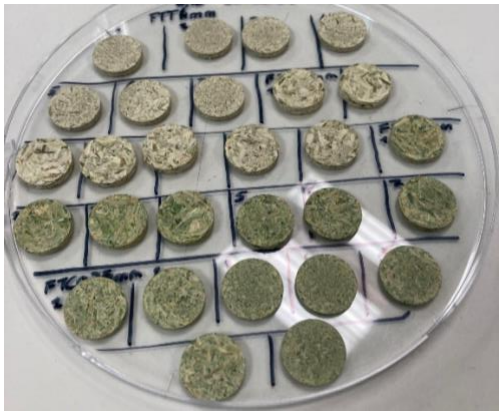
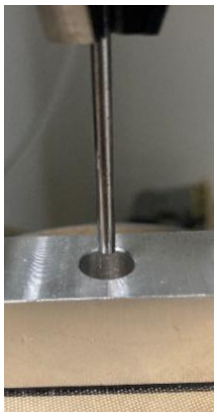
ABSTRACT

Particle boards used for furniture or insulation are made from wood fibres bound usually by synthetic resins such as urea-formaldehyde which raise concerns about health and environment. Binderless panels, composed solely of natural fibres, represent one way of limiting the use of synthetic resins. Producing such boards from under-valorized wastes would be pertinent, however the mechanical properties depend on the raw materials.

In our project, we investigate the feasibility of producing binderless boards from agricultural wastes. To this end, we have developed a rapid essay requiring little material to rank the starting materials suitability. The essay method involves a mechanical puncture test on fibre discs. Discs of 16 mm diameter and around 3mm thick, containing 400 mg fibre, are produced by hot-pressing (125 MPa at 150°C, duration 5 min). After 7 days storage under stable conditions (RH59%, 20°C) The discs are placed on a perforated tray (⌀10mm hole). A 3 mm diameter punch is driven into the disc at a rate of 2 mm/min. A stress-strain curve is obtained. This work reports results for three raw materials, tomato and cucumber stalks and wood particles, with different modalities of grinding and sieving before thermomoulding step. The materials composition was characterized through FTIR and particle size were determined through sieving. Initial results show that fibre composition plays a role in the mechanical properties of the discs rather than particles size.

Ongoing work aims to determine the ability of the puncture essay to predict the mechanical properties of end-products, using a larger set of boards with different particle-size, both with and without synthetic resin.





KEYWORDS

Binderless, panels, crops residues, mechanical properties.

FABRICATION OF FILMS BASED ON PECTIN DERIVED FROM APPLE POMACE FOR PACKAGING APPLICATIONS

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ABSTRACT

Pectin is an abundant biopolymer, derived from fruit processing residues, and a very promising biocompatible candidate for sustainable, degradable and water-soluble packaging. This study investigates the development of pectin-based films using industrial apple residues from the Saxony region (Germany). By systematically varying formulations and processing parameters, the work provides a detailed understanding of film development and presents a comprehensive overview of the resulting material. A sequential design of experiments (DoE) was applied to evaluate the combined effects of pectin and polyvinyl alcohol (PVA). Furthermore, citric acid (CA) was added as a crosslinker, as well as residual fiber fractions containing cellulose, hemicelluloses and lignin from the same biomass stream. The DoE was used to (i) screen pure and blended systems, (ii) optimize key film properties, such as tensile strength, water vapor permeability (WVP), and water vapor transmission rate (WVTR), as well as to (iii) assess the reinforcing effect of the added fiber residues.

Comprehensive characterization, including FTIR, rheology, TGA, DSC, and SEM, provided insight into structure–property relationships. The results demonstrate that the targeted combination of pectin with PVA, CA, and biomass-derived fibers significantly enhances the films performance, such as higher tensile strength or lower WVTR. Overall, this work establishes a systematic framework for designing pectin-based materials and highlights the potential of converting regional agro-industrial residues into high-performance, sustainable packaging solutions, supporting circularity and resource efficiency.

KEYWORDS

Pectin, Films, Sustainability, Biobased

EFFECT OF SUBCRITICAL WATER EXTRACTION PH ON HYDROGEL RHEOLOGY OF PECTINS EXTRACTS FROM INDUSTRIAL SUGAR PRODUCTION SIDE STREAMS

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ABSTRACT

Modern food production generates vast and often underutilised side-streams. This provides opportunities for upscaling and valorisation through, for example, generation of new materials. One major side-stream of industrial processesing of sugar beet for sugar production is the residual fibrous pulp. This pulp is rich in carbohydrates where roughly 30% of the total weight is comprised of pectin, a heteropolysaccharide compound of particular interest for side-stream valorisation due to exceptional functionality and well documented nutritional properties [1]. Sugar beet pectin (SBP) primarily contains two structurally distinct regions, a homopolymeric, linear region, homogalacturonan (HG), and a hetereopolymeric, heavily branched region rhamnogalacturonan (RG-I).

Ferulic acid, a phenolic substituent present in sugar beet pectin, provides distinct nutritional and functional properties. In particular, its radical scavenging potential can be utilised to form enzymatically coupled covalent crosslinks, forming a polysaccharide hydrogel i.e. generating a structural network capable of absorbing large amounts of water without dissociating [2, 3]. To be able to utilise this process, the preservation of the bound ferulic acid is vital and therefore an integral factor in the selection of extraction process. A method which utilises water at high pressure and temperature, subcritical water extraction (SWE), has been shown to preserve the ferulic acid substituents. However, cleavage of acetyl groups can lead to acidification and undesired autohydrolysis of the pectin backbone [4]. To mitigate this process, buffers can be used to maintain pH during the extraction process.

This project investigated the effect of extraction pH on a two-step fractionation extraction process and the resulting extract polysaccharide content and structural profile. The rheological properties, gelling kinetics and surface porosity of enzymatically crosslinked hydrogels formed from each fraction were subsequently studied. The selection of extraction pH had a significant impact on the structural profile of the extracted pectin, particularly, more acidic conditions and longer extraction times favoured the branched RG-I domain. This increase resulted in improved rheological properties in terms of increased storage and loss moduli as well as increase surface porosity. This indicates that by adjusting extraction conditions and times, the pectin domain profile can be selectively extracted, allowing for tuning of gelation time, surface porosity and rheological properties of the final gel. Tuneable hydrogels with inherent prebiotic and antioxidant properties produced utilising current industrial feedstocks to fit applications in food, pharmaceuticals and cosmetics promotes the upcycling of sugar beet side-streams to meet existing and future demands in industry.

KEYWORDS

Hydrogels, Subcritical Water Extraction, Pectin, Industrial Sidestreams, Rheological Properties

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COATED MATERIALS BASED ON FATTY ACID CELLULOSE ESTERS

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ABSTRACT

[Fatty acid cellulose esters (FACEs) are increasingly recognized as promising bio-based alternatives to petroleum-derived polymers for sustainable packaging applications. Their tunable chemical structure enables control over key properties such as hydrophobicity and mechanical performance. However, the broader application of FACEs in laminated paper structures remains limited by insufficient understanding of how synthesis route and processing conditions influence their chemical structure, surface properties, and mechanical performance.

The objective of this study is to investigate laminated materials based on FACEs produced via reactor-based synthesis and by reactive extrusion, with a focus on how synthesis technique, chemical composition, and lamination conditions influence morphological, surface, and mechanical properties of paperboards coated with FACEs.

Chemical characterization of the materials was performed using FTIR to identify chemical differences associated with the synthesis route. Surface hydrophobicity of the coated papberboards was evaluated through contact angle measurements. In addition, visual and morphological observations were conducted to assess coating uniformity and interfacial quality of the coatings. Mechanical properties were determined to evaluate the influence of synthesis route and material properties on mehcanical performance of the coated papberboards.

The results reveal that both synthesis method and chemical composition significantly impact the final material properties. Reactive extrusion–based FACEs exhibit distinct structural characteristics compared to reactor-synthesized materials, which are reflected in differences in surface morphology and mechanical behavior. Visual analysis indicated that optimized coating conditions promote homogeneous coatings with good adhesion, although variations in synthesis route influenced interfacial integrity.

In conclusion, this study demonstrates that the combination of synthesis technique and material properites plays a critical role in determining the performance of FACE-based coated materials. The findings provide important insights into tailoring chemical and physical properties through controlled synthesis and processing, supporting the development of durable, sustainable alternatives for paperboard-based packaging applications.]

KEYWORDS

Coated papberboard, fatty acid cellulose esters, paperboard-based packaging, reactor-based synthesis, reactive extrusion-based synthesis

MULTIFUNCTIONAL POLY(VINYL ALCOHOL) BIOCOMPOSITE FILMS FUNCTIONALIZED WITH (LIGNO)CELLULOSE NANOFIBRILS AND TANNIC ACID

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ABSTRACT

In recent years, biocomposite packaging films have been developed as sustainable alternatives to conventional plastics. This study investigates biodegradable poly(vinyl alcohol) (PVA)-based films reinforced with naturally occurring cellulose nanofibrils (CNFs), lignocellulosic nanofibrils (LCNFs), and tannic acid (TA) to achieve enhanced mechanical, thermal, surface, and antioxidant properties. CNFs and LCNFs serve as reinforcing agents, while TA provides antioxidant and antibacterial functionality, enabling the development of multifunctional materials with potential applications in packaging and biomedical fields.

To evaluate the individual effect of each component, two-component films (PVA–CNFs, PVA–LCNFs, and PVA–TA) were first prepared. Both CNFs and LCNFs significantly improved the tensile strength of the resulting PVA biocomposite films at all loadings, while generally reducing elongation at break. The greatest enhancement was observed in the PVA film containing 10% CNFs, which showed a 40% higher tensile strength compared to neat PVA. TA, primarily incorporated as a biologically active component, also improved tensile strength compared to neat PVA due to interactions within the polymer matrix.

Based on these results, three-component PVA-CNFs-TA films were subsequently developed and systematically studied. The film containing 2% CNFs and 10% TA exhibited a 30% higher tensile strength than neat PVA film and showed the lowest surface hydrophilicity, with a water contact angle approximately 20° higher than that of neat PVA. For three-component systems containing 4 and 6% CNFs combined with all investigated TA loadings, the onset degradation temperature increased by about 30 °C, indicating a pronounced synergistic effect on thermal stability. Additionally, an increase in glass transition temperature with increasing filler (CNFs, LCNFs, TA) content was also observed, reflecting restricted polymer chain mobility.

To improve environmental sustainability, CNFs were replaced by LCNFs as a more environmentally friendly alternative. The PVA film containing 2% LCNFs exhibited more than a 100% increase in

elongation at break compared to neat PVA, indicating improved flexibility. Incorporation of TA into the PVA-LCNFs system resulted in more water-resistant biocomposites. In particular, three-component films containing 2 and 6% LCNFs and 10% TA showed water contact angles more than 20° higher than neat PVA.

Antioxidant activity, evaluated by the DPPH method, was significant in all TA-containing films, while TA-free systems showed no or negligible activity. Overall, the results demonstrate a pronounced synergistic effect between nanofibrils and TA and highlight the potential of multifunctional PVA-based biocomposites for sustainable advanced material applications.

KEYWORDS

Poly(vinyl alcohol), (Ligno)cellulose nanofibrils, Tannic acid, Multifunctional biocomposite films.

CELLULOSE-BASED AND CIRCULAR INSULATION FOAMS FOR THE BUILT SECTOR

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[1]INRAE Nantes – UR 1268 – BIA: Biopolymers, Interactions and Assemblies

ABSTRACT

To mitigate the environmental impact of the construction sector, replacing petro-based materials with sustainable alternatives is decisive. The CIRCBUILT project (Horizon Europe, New European Bauhaus) addresses this challenge by developing and scaling up to TRL5 four innovative solutions derived from agricultural and forestry waste: thermal insulation panels, construction panels, adaptive cooling windows, and indoor acoustic panels. As part of this project, this study focuses on high-performance, cellulose-based thermal insulation panels.

The method relies on a surfactant-free process patented by INRAE, using a liquid foaming technique followed by simple oven-drying at 80°C. The formulation is based on a methylcellulose matrix that provides critical foaming properties and undergoes thermogelation during heating, preventing collapse of the cellular structure during drying. Secondary raw materials, such as wood bark, are milled to 100 µm and then integrated in the initial liquid formulation as biobased mechanical fillers.

The resulting panels present tunable densities ranging from ultralight to lightweight foams (5-70 kg/m^3) and exhibit competitive thermal conductivity (0.033-0.050 W/m.K). To meet building standards, mineral fillers can be added to ensure fire resistance. Combustion characterization revealed that these foams generate from 20 to 40 times less smoke than commercial extruded polystyrene while presenting a lower heat release by a factor of 1.7 or 2.7. Ongoing research also focuses on enhancing moisture resistance through polyphenol addition, evaluating the recyclability rate of the panels, and scaling up the entire process.

These findings highlight the potential of upcycling bio-waste into circular, upscalable, and high-performance insulation, offering a viable solution for decarbonizing the European building stock.

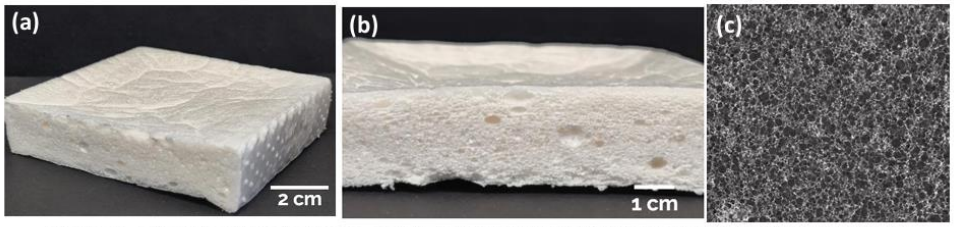

Figure 1: *(a)(b) Macroscopic images and (c) X-Ray Tomography image of a thermal insulation foam*

KEYWORDS

Insulation, foams, lightweight, circular, scale-up

COHESION MECHANISMS IN BINDERLESS LIGNOCELLULOSIC MATERIALS: THE IMPACT OF TIME AND TEMPERATURE COMBINATIONS ON THERMOCOMPRESSION

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ABSTRACT

The objective of this study was to better understand and quantify the mechanisms responsible for cohesion during the binderless thermocompression of lignocellulosic materials. To this end, a wide range of processing conditions was investigated using sugarcane bagasse, with temperatures ranging from 80°C to 240°C and processing times from 5 min to 2 h, in order to identify potential time–temperature equivalences and optimal processing conditions. The materials were characterized in terms of chemical composition using the NREL method, microstructure by scanning electron microscopy (SEM), density, flexural strength at break using a three-point bending test, and water absorption (WA) after immersion in water for 24 h at 25°C. Chemical composition analysis revealed the progressive degradation of hemicelluloses into extractable compounds, including xylan oligomers and monomers, as well as furfural, with increasing temperature. The degradation kinetics accelerated with temperature: it was observed at 120°C after 45 min, but was already detectable at 240°C after only 3 min of processing. Increasing temperature also promoted material densification, with densities reaching up to 1500 kg/m^3. The results highlighted a clear time–temperature equivalence, indicating that short processing times at high temperatures can substitute for longer treatments at moderate temperatures (e.g., 120°C). Flexural strength exhibited a bell-shaped trend, increasing to a maximum of 35–40 MPa before decreasing at higher temperatures and longer processing times. In parallel, water absorption decreased and reached a plateau of approximately 25% under severe processing conditions. Improvements in mechanical properties and water resistance were mainly attributed to densification and structural rearrangements, likely involving the formation of covalent bonds. At elevated temperatures (>160°C), thermal degradation—particularly hemicellulose hydrolysis—led to a reduction in flexural strength. However, degradation by-products may further react to form new covalent bonds, contributing to the stabilization of water absorption. Overall, four main cohesion mechanisms were identified: densification, structural rearrangement, covalent bonding, and thermal degradation. These findings provide a deeper understanding of binderless consolidation and offer valuable insights for optimizing processing conditions in the development of fully bio-based materials.

KEYWORDS

Thermocompression, Lignocellulosic materials, Thermal degradation, Cohesion mechanisms

EFFECT OF CELLULOSE NANOCRYSTAL SURFACE CHEMISTRY ON BIOMINERALIZATION OF CALCIUM PHOSPHATE

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ABSTRACT

Nanocellulose–hydroxyapatite (HAp) nanocomposites are attractive for bone repair because HAp mimics the inorganic phase of bone while nanocellulose provides a porous, collagen-like network. This study investigates how surface chemistry of cellulose nanocrystals (CNCs) influences calcium phosphate (CaP) and HAp nucleation and growth in simulated body fluid (SBF). Ultrathin films of carboxylated, phosphorylated, and sulfated CNCs were deposited on SiO_2 quartz sensors and incubated in 1.5× SBF at 37 °C for 96 h. Mineralization was monitored in situ by quartz crystal microbalance with dissipation (QCM-D) and characterized ex situ by atomic force microscopy and scanning electron microscopy.

QCM-D revealed the greatest frequency decrease for carboxylated CNCs, indicating the highest CaP deposition. Phosphorylated CNCs also showed substantial frequency reductions, consistent with significant precipitation, whereas sulfated CNCs exhibited only minor changes, suggesting limited mineral growth. All samples displayed high dissipation increases, indicative of soft, viscoelastic layers. Combined with microscopy observations, these data support the formation of amorphous calcium phosphate rather than crystalline hydroxyapatite under the tested conditions.

These findings demonstrate that CNC surface functional groups regulate CaP nucleation in SBF, with carboxylation most strongly promoting deposition and phosphorylation providing an intermediate effect, while sulfation is comparatively ineffective. Tailoring nanocellulose surface chemistry can thus be used to steer early-stage mineral formation, informing the design of bioactive, bone-mimetic nanocomposites and coatings that promote mineralization at implant interfaces. Of these three types of CNCs, the carboxylated one, showing the highest capacity to induce calcium phosphate growth, is likely the best candidate for bone healing. The next step is to develop mineralized nanocomposites from cross-linked, carboxylated CNCs and hydroxyapatite.

KEYWORDS

Cellulose nanocrystals, quartz crystal microbalance (QCM-D), thin film, biomineralization

MILD PRODUCTION OF HOLOCELLULOSE FIBRES WITH HIGH HEMICELLULOSE CONTENT

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ABSTRACT

Lignocellulosic fibres are attractive materials that can replace traditional fossil-based alternatives due to their superior mechanical, thermal, and chemical properties [1]. These fibres are biological materials precisely organized from the nano to the macroscale to fulfill their biological functions, providing stability and strength against mechanical and osmotic stress to plant cells. The release of lignocellulosic fibres from the recalcitrant woody feedstock typically involves harsh chemical processes to degrade the lignin [2], resulting in a concomitant loss of carbohydrates, hemicellulose in particular, that could be used for diverse applications. For example, hemicelluloses can enhance bonding strength between cellulose fibrils, improving the mechanical properties of cellulose fibrillar networks [3]. Some hemicellulose chains can tightly interact with cellulose chains in rigid conformations, whereas other hemicellulose domains are more flexible and available for further modification [4]. In this context, wood-based holocellulose fibres retaining most of the hemicellulose around the cellulose fibrillar structure, emerge as a high-quality sustainable building block both in terms of enhancing the mass balances for holofibre production and improving their performance.

Amongst delignification methods, peracetic acid (PAA) shows high selectivity towards lignin removal [5]. In this work, we propose a method to produce holocellulose fibres with high hemicellulose content via PAA treatment, preserving the rigid and flexible hemicellulose domains. We have investigated the

effect of different temperature and pH conditions, and comparing the compositional and structural properties of the derived holocellulose materials, including hemicellulose content, delignification efficiency, and fibre morphology to determine optimal conditions.

KEYWORDS

Holocellulose, hemicellulose, delignification, optimization

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IMIDAZOLE STRUCTURES AT REDUCING END GROUP SITES OF CELLULOSES FORMED THROUGH A MAILLARD REACTION CASCADE

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ABSTRACT

A recently reported facile one-step isolation of cationic cellulose nanocrystals (CNCs) was achieved by high temperature treatment (120 - 160 °C) of different cellulosic feedstocks in reactive eutectic media (REM) consisting of ammonium formate and high boiling organic acids [1]. The performed characterization suggested the introduction of charged nitrogen-containing moieties selectively to the isolated CNCs' reducing end groups (REGs). Given the operative reaction conditions, a reductive amination according to a Leuckart Wallach reaction was proposed. However, selectivity and underlying reaction mechanism could not be confirmed by any direct analytical technique.

This left room for scrutiny which motivated the current follow-up effort. The application of solution NMR measurements using a direct-dissolution electrolyte allowed further insights into the occurring reactions [2]. A modification predominantly restricted to the REGs was confirmed and no significant changes of the cellulose backbone shifts were apparent. In contrast to the initial proposal, no resonances corresponding to amine functionalities were present. Instead, peaks in the range of aromatic *N*-heterocycles were seen. Their constitution was established using a CNC prepared from a low molecular-weight cellulose model compound and the application of different 2D NMR experiments. Unexpectedly, the major product was identified as a furanosyl imidazole structure. This was further confirmed by high-resolution mass spectrometry. The proposed furanosyl imidazoles, in monomeric form, are known in literature. They were prepared from glucose in a high temperate reaction with formamidine acetate [3]. Under the conditions prevailing during CNC preparation, the same structures are likely to be formed over a multistep Maillard reaction cascade – involving the imidazole ring closure over a Weidenhagen or Bredereck type reaction. To the best of our knowledge, this represents the first account of a neat Maillard reaction product formation directly on the REGs of cellulose.

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KEYWORDS

Cellulose Nanocrystals, NMR, Maillard Reactions, Reducing End Groups

ELECTROSPINNING OF BIO-BASED CHITOSAN/SUBERINIC ACID COMPOSITE NANOFIBERS

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ABSTRACT

The development of sustainable, fully bio-based nanostructured materials remains a significant challenge, particularly when combining processability with functional stability. Chitosan is a promising biopolymer for hemosorbtion and biomedical applications; however, its electrospinning is limited by poor spinnability and instability in water. In this work, we demonstrate, for the first time, the fabrication of electrospun nanofibrous composites based on chitosan and suberinic acids derived from birch bark, using polyethylene oxide (PEO) as a processing aid.

Chitosan (degree of deacetylation >90%, Mw ~430 kDa) was used as the primary structural polymer, while suberinic acids served as a bio-based functional component stabilizing the fibrous structure. PEO (Mv ~600 kDa) was added in minor amounts to enable fiber formation. The compositions were systematically varied, resulting in nine formulations grouped by chitosan:PEO:suberinic acid ratios of approximately 4:1:5–10, 10:1:14–28, and 15:1:20–40, covering a wide compositional range.

Electrospinning was performed using a wire-based, needle-free setup (Nanospider™ technology) under optimized conditions. Uniform, bead-free nanofibers were obtained for selected compositions, demonstrating that stable electrospinning is feasible even in partially immiscible systems. Fibers with an average diameter of 246 nm and a narrow size distribution were confirmed by scanning electron microscopy (SEM) and statistical analysis.

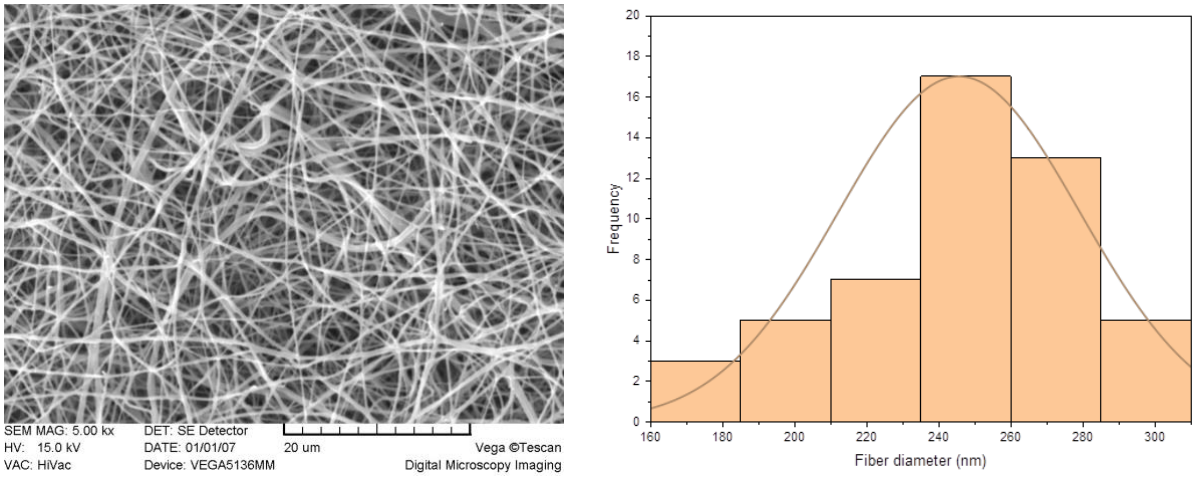

Fig. 1. Morfology of sample chitosan:PEO:suberinic acid 4:1:5.

Post-spinning thermal treatment (150 °C, 15 min) produced water-insoluble fibrous mats, indicating effective interactions between chitosan and suberinic acids. This eliminates the need for synthetic crosslinkers and highlights the potential of suberin-derived compounds as natural stabilizing agents.

Although the materials did not exhibit antibacterial activity, their biodegradability and fully bio-based composition suggest potential for biomedical applications. Considering the reported bioactivity of chitosan and suberin-derived compounds, the developed nanofibrous composites show promise for filtration applications, supported by their high surface area and chemically active functional groups, while also demonstrating potential for wound healing and hydrophobic coating applications.

Overall, this work introduces a novel class of fully bio-based electrospun materials and demonstrates the feasibility of integrating suberinic acids into nanofibrous architectures, providing a new pathway for the valorization of suberin-rich biomass.

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KEYWORDS

Chitosan, suberinic acids, ectrospinning, bio-based nanofibers

IN VITRO COLONIC FERMENTATION OF LOW METHOXYL PECTINS: THE INFLUENCE OF DELIVERY FORM

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ABSTRACT

Dietary fibers (DFs) are carbohydrate polymers that resist digestion in the human gastrointestinal tract (GIT) but are fermented by our gut microbiota, an essential microbial community that influences the individual's nutrient absorption, health, and disease prevention [1]. Among DFs, pectin is abundant in plant products, and it is widely used both in the food industry and the pharmaceutical sector [2]. Fermentation of DFs is influenced by various aspects, such as concentration, monosaccharide

composition or molar mass (M_W). This research aims to study the unexplored difference in fermentation between delivering a low methoxyl pectin (LM-pectin) as a calcium (Ca^{2+})-induced gel or a dispersion into an *in vitro* human colonic fermentation (HFC) system.

LM-pectins were characterised by SEC-MALS, monosaccharide, uronic acid analysis, and rheology. Ca^{2+}-induced gels were prepared from the same LM-pectins by addition of $CaCO_3$ and glucono-δ-lactone (GDL) and were characterized using small and large deformation. The fermentability of the LM-pectin dispersions and Ca^{2+}-induced gels was explored in an *in vitro* HCF setup by following gas and metabolites production, pH and changes in microbiota. The results show that more gas and short chain fatty acids (SCFAs) were produced upon fermentation of LM-pectin gels compared to their dispersions. PCoA analysis of microbiota compositions corroborated the differences in metabolites, showing difference in microbiota genomics between dispersions and gels, presence or absence of GDL and fermentation time. The results show the relevance in pharma and food of the delivery form of a DF as pectin, which modifies fermentation rate, metabolites production and shapes microbiota composition.

KEYWORDS

Pectin, dietary fiber, colonic fermentation, microbiota, fermentability.

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PRODUCTION AND CHARACTERIZATION OF A HIGHLY SULPHATED, FUCOSE-CONTAINING POLYSACCHARIDE BY MARINE PIGMENTED BACTERIUM *PARACOCCUS ZEAXANTHINIFACIENS* RA19

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ABSTRACT

Marine ecosystems are vast and diverse, harbouring microorganisms with unique metabolic strategies developed to survive in harsh conditions (i.e., high pressure salinity, oxygen, light, etc). Bacteria from such environments are sources of many useful molecules (exopolysaccharides, proteins, pigments, surfactants, etc.) with promising biotechnological applications in medicine, pharmaceutics, cosmetics, food and textile industries. In this work, the pigmented marine bacterium *Paracoccus zeaxanthinifaciens* RA19 isolated from a microbial mat in French Polynesia, was cultivated in mineral medium supplemented with glycerol to produce a highly sulphated extracellular polysaccharide rich in fucose (EPS RA19) and the yellow pigment zeaxanthin. Optimization of cultivation parameters was performed, where the best batch cultivation achieved 7.15 ± 0.42 g/L of pigmented biomass and 2.25 g/L of EPS. The polymer was composed mainly of fucose, rhamnose, and glucuronic acid, and high

levels of sulphur (15wt.%), determined by HPLC and elemental analysis, respectively. RA19 presented shear thinning behavior, increasing viscosity at higher polymer concentrations. The impact of temperature, pH and ionic strength was also assessed, indicating a flow behavior dependent on these parameters. The emulsification properties of EPS RA19 were evaluated using olive oil, paraffin oil and almond oil. Notably, EPS RA19 effectively stabilized emulsions with paraffin oil at an optimal oil-to-water ratio of 3:2 (v/v). This work shows that P. zeaxanthinifaciens could be a versatile marine microorganism to produce interesting biomolecules.

KEYWORDS

Polysaccharides, bacteria, pigment, rheology

INFLUENCE OF ACETYLATION AND FERULOYLATION ON CORN BRAN ARABINOXYLAN DEGRADABILITY BY *GANODERMA LUCIDUM*

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ABSTRACT

Corn processing from agricultural activities produces 10-13 million tons of bran annually, most of which remains underutilized[1]. Corn bran is rich in insoluble fiber, protein and phenolic compounds making it a promising renewable resource with high biotechnological potential for producing biofuels, biomaterials and bioactive compounds[2].

The predominant hemicelluloses in cereal cell walls are arabinoxylans, on which ferulic and *p*-coumaric acids are esterified. These phenolic acids form cross-links between polysaccharide chains, lignin and proteins, resulting in a complex structure that limits enzymatic accessibility[3].

White rot fungi (WRF) can efficiently degrade all lignocellulose components through the action of hydrolytic and oxidative enzymes. The adaptation of their enzymatic machinery is directly influenced by substrate composition and structure. In arabinoxylans, key features such as monosaccharide composition, degree of feruloylation and degree of acetylation significantly affect their degradability by fungi[4]. Despite extensive research on WRF-mediated lignocellulose degradation and enzyme synergies on arabinoxylan fractions, the specific roles of acetyl and ferulate substitutions to arabinoxylan recalcitrance during fermentation remain poorly understood.

This study investigates how acetyl and ferulate substitutions modulate arabinoxylans degradation by *Ganoderma lucidum*. Arabinoxylans were isolated from corn bran using subcritical water extraction and characterized in terms of monosaccharides composition, degree of substitutions and molecular weight distribution. Targeted enzymatic treatments were then applied to selectively remove acetyl and ferulate groups, generating substrates with defined substitution patterns. These modified arabinoxylans were subjected to fungal fermentation, and changes in substrate structure, sugar and phenolics release/consumption, and secretome composition were analyzed after 5 and 10 days using proteomics and enzyme activity assays.

Overall, this work provides new insights into fungus-substrate interactions during cereal bran degradation, particularly on the hemicelluloses fraction of the cell wall. Understanding how arabinoxylan structure influences fungal degradation strategies will support the development of more effective WRF-based pretreatment processes for cereal biomass valorization.

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KEYWORDS

Cereal by-products, Arabinoxylans, White rot fungi, Ferulic acid

EFFECT OF SOLVOLYSIS CONDITIONS ON THE RECOVERY, MECHANICAL AND SURFACE PROPERTIES OF FLAX FIBRES FROM ELIUM®-BASED COMPOSITES

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ABSTRACT

The recycling of bio-based composites reinforced with flax fibres represents a major challenge for the development of sustainable materials. Solvolysis appears as a promising route for fibre extraction and

recovery at end-of-life. This study aims to optimize solvolysis conditions to recover flax fibres from C195E Elium®-based composites for reuse in composite applications.

Solvolysis of Elium® was carried out in subcritical conditions at 250 bars in a lab-scale reactor, with water and water/ethanol mixture (50/50 v/v), with and without 0.2 M sodium hydroxide, at 175°C and 200°C, and at different flow rates (1.31 and 3.99 mL/min). High dissolution rates were achieved at 200 °C, whereas milder conditions (175 °C in water) effectively mitigated fibre degradation, with stiffness losses remaining below 10%, compared to up to 35% under more severe solvolysis conditions (Fig. 1) (Garat et al., 2021; Henry et al., 2016).

Tensiometric characterisation of flax fibres allowed the determination of their surface energy components (polar, dispersive, and total), highlighting differences between recovered fibres and virgin fibres. (Garat et al., 2021; Pucci et al., 2016). In addition, fibre/Elium® interfacial adhesion was evaluated using the Owens–Wendt method, based on the determination of the polar and dispersive components of the surface energies of both the fibres and the matrix. This analysis was further supported by interfacial shear strength (IFSS) measurements, enabling a direct evaluation of interfacial mechanical performance. Together, these approaches provide a comprehensive understanding of fiber/matrix affinity, highlighting the potential for reuse of solvolysed fibres.

Complementary characterisations (AFM, XPS, SEM) allowed to further investigate the surface topography and morphology of the fibres. Finally, a comparative study is conducted with carbon fibres, widely used in composite materials.

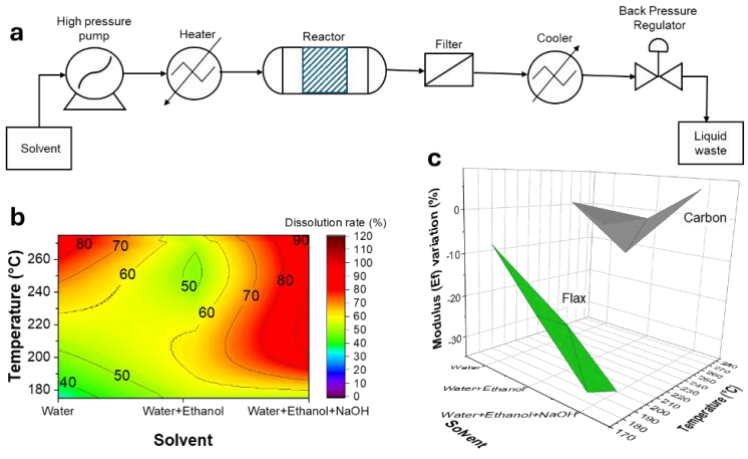

Fig. 1 : (a) Schematic representation of the lab-scale subcritical solvolysis system; (b) Dissolution rate (%) under the various temperature and solvent conditions tested ; (c) Variation in elastic modulus (%) compared to virgin fibres for extracted single flax and carbon fibres.

KEYWORDS

Recycled bio-based composite materials, Solvolysis, flax fibres, carbon fibres, Thermoplastic matrix (Elium®), Interfacial adhesion.

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ENHANCING WATER RETENTION IN CELLULOSE FIBRES VIA NON-IONIC SURFACE FUNCTIONALIZATION

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ABSTRACT

The reduced water retention of cellulosic fibres upon drying, known as hornification, poses many challenges for the recycling of fiber products. Hornification causes practically irreversible closure of pores within the cell wall and reduced fiber flexibility, which leads to poorly bonding, brittle fibres. A common strategy for improving the water retention of cellulose fibres is increasing their surface charge. However, charged fibres are generally sensitive to changes in pH and the ionic strength of the surrounding medium. Thus, in this study, we explore alternative strategies to increase the water retention of cellulose fibres, namely by attaching hygroscopic moieties to the fibre surface. Specifically, end-modified polyethylene glycol (PEG) was attached to a cellulose nanocrystal thin film, which serves as an experimental model representing the surface of cellulosic fibres.

The adsorption of amine-terminated PEG and subsequent swelling of the thin film was studied with a quartz crystal microbalance with dissipation monitoring (QCM-D). The attachment of amine-terminated PEG was achieved through both electrostatic interactions and covalent bonding. Our findings demonstrated a significant swelling of the cellulose model film upon end-wise attachment of PEG. The extent of swelling was found to depend on PEG chain length, with shorter chains inducing more pronounced swelling. Furthermore, we found that shorter oligomeric PEG chains can be attached by ionic interactions while longer chains require covalent bonds. This could be attributed to the interplay between the strong solvation of PEG in water and the driving forces required to adsorb these highly hydrophilic PEG chains onto cellulose. Finally, the equilibrium water content of the swollen films was evaluated, providing further insight into how hygroscopic moieties influence water retention in cellulose-based fibre networks.

KEYWORDS

Hornification, swelling, cellulose, adsorption

SEQUENTIALLY ELECTROSPUN BILAYER SCAFFOLDS CONTAINING HA-GLY-OH AS MEDICAL DEVICES

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ABSTRACT

Electrospinning enables the production of ECM-mimetic nano- and micro-fibers for tissue engineering. Bilayer asymmetric scaffolds, with a hydrophobic outer layer (simulating epidermis) and a hydrophilic inner layer (supporting cell activity), are promising for skin regeneration. Hyaluronan (HA) is attractive but difficult to electrospin; blending with synthetic polymers such as polyvinylpyrrolidone (PVP) overcomes this. This study aimed to fabricate bilayer scaffolds of PDLLA (outer) and PVP blended with either native HA or HA-Gly-OH (inner layer) to evaluate their physico-chemical properties, cytocompatibility, and release profile.

HA was coupled with glycine ethyl ester via carbodiimide chemistry, followed by global deprotection. Bilayer scaffolds were produced by sequential electrospinning: first pristine PDLLA, then a PVP blend containing 1% or 3% w/w of either native HA or HA-Gly-OH. Finally, the scaffolds underwent UV crosslinking to avoid complete dissolution of the inner layer in ultrapure water. Morphology, mechanical properties, cytotoxicity (fibroblasts), and cumulative release were assessed. Sequential electrospinning yielded intact bilayer scaffolds, whose morphology and mechanical behaviour differed between layers and were modulated by both polysaccharide type and concentration in a dose-dependent manner. Cytocompatibility assays showed that both layers individually were compatible with fibroblasts. However, higher concentrations (3% w/w) of either native HA or HA-Gly-OH induced a cytotoxic response. Conversely, the 1% w/w formulations, particularly with HA-Gly-OH, showed encouraging cytocompatibility. Cumulative release studies on the 1% HA-Gly-OH formulation demonstrated a slow-release profile, probably due to entrapment of polysaccharides within the crosslinked PVP network.

Bilayer PDLLA/PVP electrospun scaffolds incorporating either 1% or 3% w/w of pristine HA or HA-Gly-OH were successfully fabricated. The semi-synthetic HA-Gly-OH modulated scaffold properties without the complexity of peptide synthesis. The 1% formulation showed interesting cytocompatibility and cumulative release, highlighting its potential as a medical device for skin tissue engineering. Further *in vitro* and *in vivo* studies are needed to confirm therapeutic efficacy and long-term stability.

KEYWORDS

bilayer scaffolds, biocompatibility, electrospun scaffolds, glycine, hyaluronic acid

ALGINATE PRODUCTION USING *AZOTOBACTER VINELANDII* AND APPLE WASTE-LIKE MEDIA

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ABSTRACT

Alginate is a polymer produced in nature by algae and bacteria, and it is composed of β-D-mannuronic and α-L-guluronic acid as monomers. It has several applications in different fields, like in the food industry as a food-grade thickener or in the biomedical one due to the development of hydrogels. Biotechnological production of alginate in bacteria is more desirable than in algae because of the environmental impact, so for this goal it is used *Azotobacter vinelandii*. Furthermore, the objective of this research is to potentially study the production of alginate using hydrolysed apple waste as the carbon source to develop a sustainable process allied with the circular economy.

Firstly, cryopreserved *A. vinelandii* is cultured in Petri dishes with a solid agar media composed of 20 g/L sucrose, 0,5 g/L yeast extract and salts for 72 hours. After that, the bacteria is inoculated in Erlenmeyer flasks using the same media but liquid. The culture is then maintained at 30 ºC and 200 rpm

during 24 hours prior to a new inoculation in fresh media in new flasks. This culture begins with an optical density at 600 nm of 0.5 and lasts for 24 hours at 30 ºC and 200 rpm. Finally, the culture is inoculated in a bioreactor with an initial optical density at 600 nm of 0.5. In this step, there is no addition of yeast extract, and it is used apple-waste model media composed of glucose + fructose and salts. The bioreactor is maintained during 72 hours at 600 rpm, 30 ºC, pH 7.0 and 1.5 L/min air flow. To study this bioprocess, samples are taken at different times from the bioreactor culture. For each one, it is studied the media viscosity, glucose and fructose consumption, alginate and polyhydroxybutyrate (PHB) production and biomass growth. PHB is a co-product with several applications like packaging, so it is interesting to also be studied. The physical and chemical characteristics of the alginate like molecular weight, viscosity, acetylation rate and guluronic and mannuronic rate are also studied.

The preliminary results obtained show a maximum production of nearly 5 g/L alginate which is similarly reported in literature and all sugar content is consumed. This bioproduct has also characteristics similar to the previously studied by other authors. Consequently, with a model apple-waste the production of alginate is comparable with the ones using *A. vinelandii* model media with sucrose.

KEYWORDS

Alginate, circular economy, biotechnology, bacterial culture

EFFECT OF PRE-TREATMENTS AND RETTING PROCESSES OF *SPARTIUM JUNCEUM* FIBERS FOR TEXTILE APPLICATIONS ON THEIR BIOCHEMICAL COMPOSITION AND MECHANICAL PROPERTIES

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ABSTRACT

Widely distributed in Mediterranean regions, Spanish broom is a plant resource of interest due to its high cellulose-rich fiber content [1]. Used in the textile industry until the 1960s, it was gradually drop out in favor of imported cotton, particularly from Asian countries. The efficient extraction of fibers from broom stems depends on the retting conditions after stems harvesting. Retting is a well-known biological and physico-chemical process that leads to the degradation of pectins [2] and other compounds responsible for the cohesion of fibers with surrounding tissues. In this context, the aim of this study

consists in better understanding the relationship between fiber biochemical composition and their mechanical properties.

Using year's stems of Spanish broom, harvested in October 2025 in northern Hérault, in the Occitanie region (France), it became apparent that, prior to the retting stage, the stalks needed to undergo a series of pretreatment steps to facilitate the stem's decortication, i.e., the separation of the epidermis from the internal tissues. A water steam treatment followed by a mechanical beating was specifically investigated. The epidermis obtained from these pre-treatments was subjected to two different retting conditions. The first one is performed by immersing epidermis in a maceration water or referred to as « microorganisms-rich water » in the Figure 5 (water resulting from the prior immersion of fresh stems for 7 days, whose physico-chemical characteristics are known). The second one is being implemented on the soil where Spanish broom has been cultivated with a control of the humidity at the surface of the heap.

After each retting, the mechanical properties and the biochemical composition by quantifying the main cell wall components (cellulose, hemicelluloses, pectins) were determined. These results will then be correlated with the evolution of the stem microstructure (optical microscopy) and of the microbial diversity at the level of the epidermis (characterization of bacterial and fungal communities using metabarcoding based on 16S and 18S rDNA, respectively) [3].

The results highlight both a decrease in the pectin and hemicellulose fractions leading to fiber individualization, and an improvement of fiber stiffness and mechanical strength, thanks to the selected pretreatments. Differences between the two retting conditions were also observed.

It should be noted that the knowledge acquired is part of an integrated approach to structuring a Spanish broom processing sector in France, integrating textile industry – more specifically in Occitanie – in a logic of promoting local plant resources and addressing economic, social, and environmental challenges of the region.

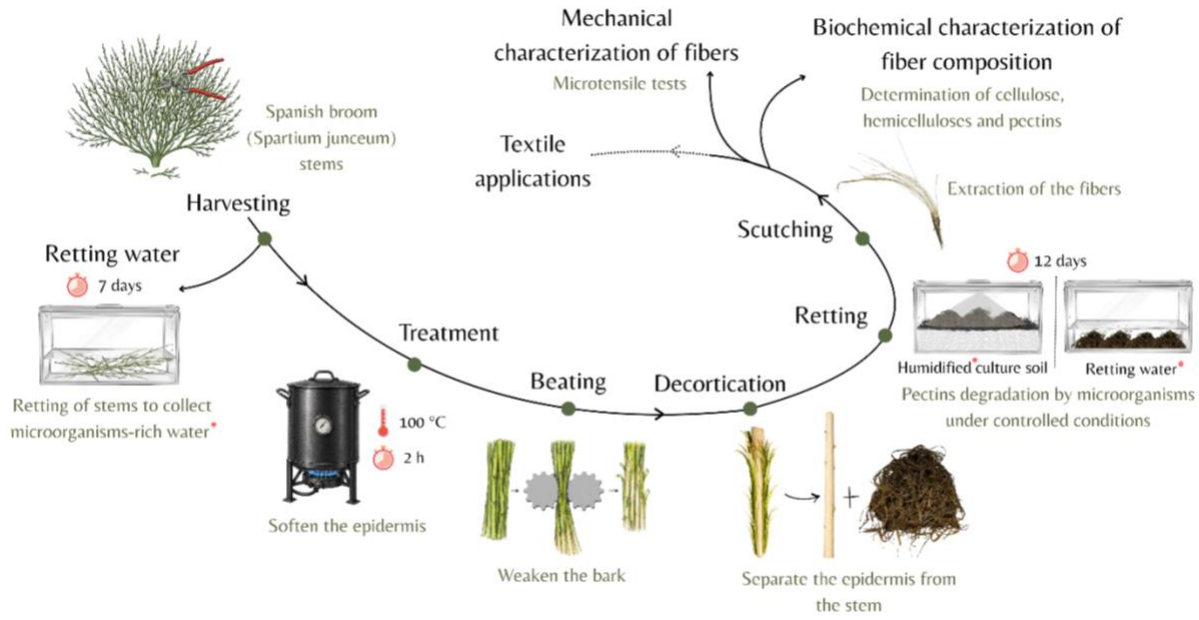

FIGURE 5 : METHODOLOGY OF EXTRACTING FIBERS FROM SPANISH BROOM STEMS (C. CAILLEAUD)

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KEYWORDS

Spanish broom fibers, biochemical composition, mechanical properties

HIGH-CONSISTENCY SYNTHESIS OF QUATERNIZED CELLULOSE NANOCRYSTALS FOR ANTIBACTERIAL APPLICATIONS

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ABSTRACT

The rising burden of bacterial infections across industrial and biomedical settings creates an urgent demand for sustainable, non-toxic antibacterial materials that prevent colonization without promoting resistance.[1] Quaternized cellulose nanocrystals (Q-CNCs) are an ideal candidate, combining the mechanical robustness of native cellulose with the antimicrobial properties of cationic surface groups. However, the etherification reactions typically used for functionalization yield low efficiencies (≤38%) due to competing side reactions,[2] severely limiting their industrial viability.

To further enhance the reaction efficiency, we employed a semi-dry process at 45 wt.% solids using a mechanical kneader.[3] This high-consistency mixing facilitated efficient mass transfer while preserving the intrinsic crystalline structure of the cellulose.[3] By optimizing the reaction medium with a water and isopropanol (IPA) co-solvent system, we successfully surpassed the 38% efficiency threshold, yielding a highly charged and colloidally stable suspension.

The reaction efficiency and degree of substitution were validated through elemental analysis and conductometric titration. Furthermore, chemical modifications and structural integrity were corroborated using NMR and X-ray scattering. Ultimately, this approach bridges the gap toward the scalable industrial production of Q-CNCs.

KEYWORDS

Quaternized CNC, antifouling, Semi-dry process

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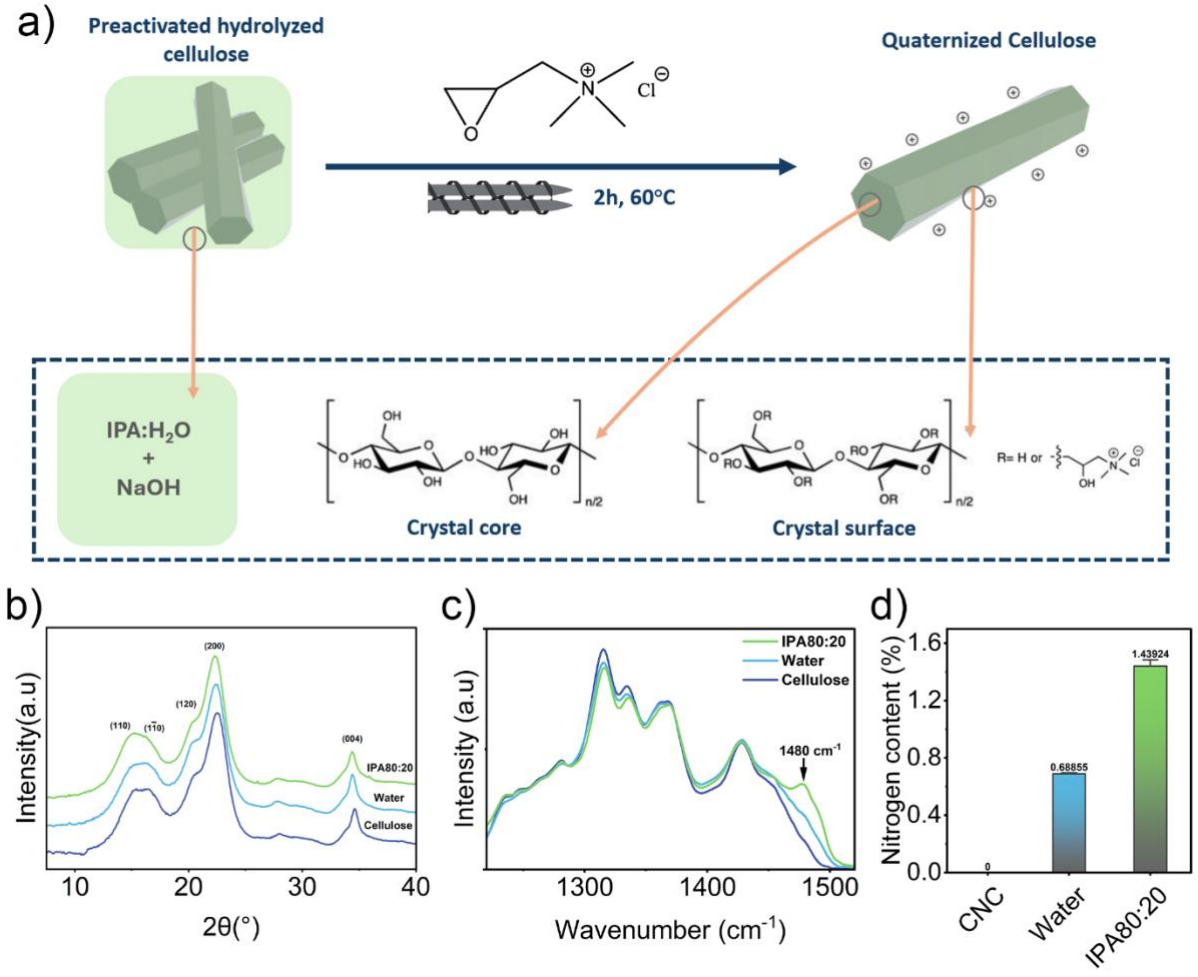

Figure 6. Semi-dry synthesis of Q-CNC. a) overall quaternization reaction illustration. b-d) WAXS, FTIR, and Nitrogen content of cellulose, Q-CNC using water and IPA/water as the reaction medium.

QUANTIFICATION OF TRACE AMOUNTS OF PET TO AID EFFICIENT RECYCLING OF POLYESTER-CELLULOSE BLENDS

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ABSTRACT

Owing to the growing textile consumption and the environmental concerns due to waste generation, there is an urgent need for more circular approaches to textile waste management. In particular, recycling of cellulose–polyethylene terephthalate (PET) blends remains challenging because it depends not only on efficient sorting and purification, but also on reliable analytical methods for composition control. This is especially critical for the precise determination of trace PET contents below 1%, for which no suitable methods are currently available. Existing approaches are largely based on gravimetry and thus lack the required precision. Yet even such small amounts of PET may already interfere with cellulose dissolution and the re-spinning process, making accurate quality control of recycled feedstocks essential.

This work therefore addresses the development of a suitable analytical method for precise PET quantification, thus aiding the recycling of cellulose-based textile blends. A system using basic hydrolysis of PET into its monomers and subsequent quantification of terephthalic acid (TPA) by HPLC-UV-ESI/MS is employed. This method enables the precise quantification of PET in cellulose–PET blends down to the ppm range. In addition, its comparatively simple setup facilitates straightforward scale-up and the high throughput required for industrial implementation. Thus, this method potentially supports the industrial recycling of cellulosic textile waste and contributes to improved quality control and more efficient closed-loop processing of textile blends.

KEYWORDS

Cellulose recycling, PET, textile recycling, polycotton

ONE-STEP PRODUCTION OF XYLOOLIGOSACCHARIDES FROM OAT HUSKS BY STEAM EXPLOSION

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ABSTRACT

Efficient conversion of lignocellulosic residues into high-value functional products remains a key challenge in sustainable biorefinery development. Oat husks, an abundant agricultural by-product, are rich in hemicelluloses, particularly xylan, which can serve as a precursor to xylooligosaccharides (XOS), a class of prebiotics with high functional efficacy. However, conventional XOS production typically relies on multi-step enzymatic processes, increasing costs and complexity.

In this study, a one-step steam explosion approach is proposed for the direct production of XOS from oat husks, eliminating the need for enzymatic treatment. The process was carried out at 200–205 °C for 5 minutes at 16 bar, during which autohydrolysis depolymerizes xylan into soluble oligosaccharides. This enables simultaneous biomass fractionation and the generation of functional compounds.

Two product pathways were evaluated. In the first, the entire processed material, including lignocellulosic residues, was used as a functional feed additive (BS). In the second, the oligosaccharide-enriched liquid fraction was separated and processed into a concentrated prebiotic product (XO). As shown in Table 1, the total XOS content increased from 80 mg g^{-1} in the bulk material to 256 mg g^{-1} in the purified fraction. The oligosaccharide profile ranged from XOS2 to XOS7, where the numbers denote the degree of polymerization (i.e., the number of xylose units), indicating effective and controlled depolymerization of xylan into low- and medium-length oligomers.

Table 1. Xylooligosaccharide composition determined by chromatographic analysis

Compound mg g^{-1}/ Sample	Xylose	Glucose	XOS2	XOS3	XOS3	XOS4	XOS5	XOS6	XOS7	XOS total
BS	5.92	6.25	7.99	7.40	14.26	13.98	12.67	11.57	12.32	80.18
XO	30.76	13.76	28.34	20.77	46.52	47.39	41.60	38.00	33.82	256.43

Compared with commonly used prebiotics such as inulin, XOS exhibits higher biological activity, allowing lower dosages to achieve similar effects. Their broader application has been limited by the cost of conventional production methods, whereas the presented approach offers a simplified and potentially scalable alternative.

Feeding trials with laying hens demonstrated improved laying performance, eggshell strength, and egg quality. While these results confirm functional potential, the primary significance of this work lies in developing an efficient, enzyme-free production route.

Overall, this study demonstrates a sustainable pathway for producing prebiotic oligosaccharides from oat husks, contributing to cost-effective biomass valorization.

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KEYWORDS

Xylooligosaccharides, steam explosion, oat husks, prebiotics, biomass valorization

GELATIN-BASED MICROGELS EMBEDDED IN ZWITTERIONIC GUAR GUM HYDROGELS AS A NOVEL BIOMATERIAL FOR BIOPRINTING AND CELL ENCAPSULATION

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ABSTRACT

Advances in 3D bioprinting have enabled the fabrication of cell-laden biomaterials for tissue engineering; however, bioink development remains limited by the narrow range of materials that can be reliably printed into complex three-dimensional structures. The performance of implanted bioprinted constructs is further compromised by biofouling and foreign body responses, which can lead to cellular overgrowth, fibrosis, and impaired transport of nutrients and oxygen to encapsulated cells. To address these challenges, zwitterionic polymers, known for their super-hydrated and charge-balanced surfaces, have emerged as promising candidates for the development of ultra-low-fouling biomaterials.

In this study, we developed zwitterionic guar gum (ZGG)-based bioinks with antifouling properties while maintaining suitable printability. ZGG was synthesised by reacting guar gum (GG) with 3-((3-chloropropyl)dimethylammonio)propane-1-sulfonate in an isopropanol/water mixture. To enable photocrosslinking, ZGG was further functionalised with methacryloyl groups via methacrylic anhydride, yielding ZGGMA. ^{1}H NMR confirmed successful synthesis of ZGGMA with a degree of substitution (DS) of 0.1 (Fig. 1a). Zwitterionic modification reduced the viscosity of GG, likely due to increased hydrophilicity and partial polymer degradation. To improve rheological properties, gelatin methacryloyl (GelMA) was initially added; however, significant phase separation occurred, compromising printability. To overcome this, GelMA was converted into microgels via fragmentation and extrusion through a 40 µm mesh (Fig. 1b). Incorporation of GelMA microgels (G-40 µm) at 2% and 4% (w/v) into ZGGMA (4% w/v) significantly enhanced the rheological properties, with storage modulus (G') exceeding loss modulus (G") (Fig. 2b). Mechanical testing revealed a substantial increase in compressive modulus for ZGGMA 4%/G-40 µm 2% compared to both ZGGMA 4% and bulk GelMA 5% hydrogels (Fig. 2c). Antifouling performance, evaluated via adsorption of fluorescein-labelled albumin, showed that ZGGMA hydrogels reduced non-specific protein adsorption by approximately 50% compared to GGMA (Fig. 2d). Bioinks were prepared in PBS with 0.1% (w/v) LAP photoinitiator, printed using an extrusion-based bioprinter, and subsequently photocured under 365 nm UV light. The developed bioinks demonstrated good printability following optimisation of composition and printing parameters. Confocal microscopy confirmed homogeneous distribution of GelMA microgels within the ZGGMA matrix (Fig. 2a). Cytotoxicity studies using C2C12 cells indicated good biocompatibility. Ongoing studies are investigating cell behaviour at the interface between the zwitterionic matrix and GelMA microgels, which may provide cell-adhesive cues.

In conclusion, ZGGMA-based composite bioinks exhibit antifouling properties, biocompatibility, and enhanced mechanical performance suitable for soft tissue engineering, demonstrating strong potential for cell encapsulation applications.

KEYWORDS

Zwitterionic guar gum, GelMA, Bioprinting, Biointerface

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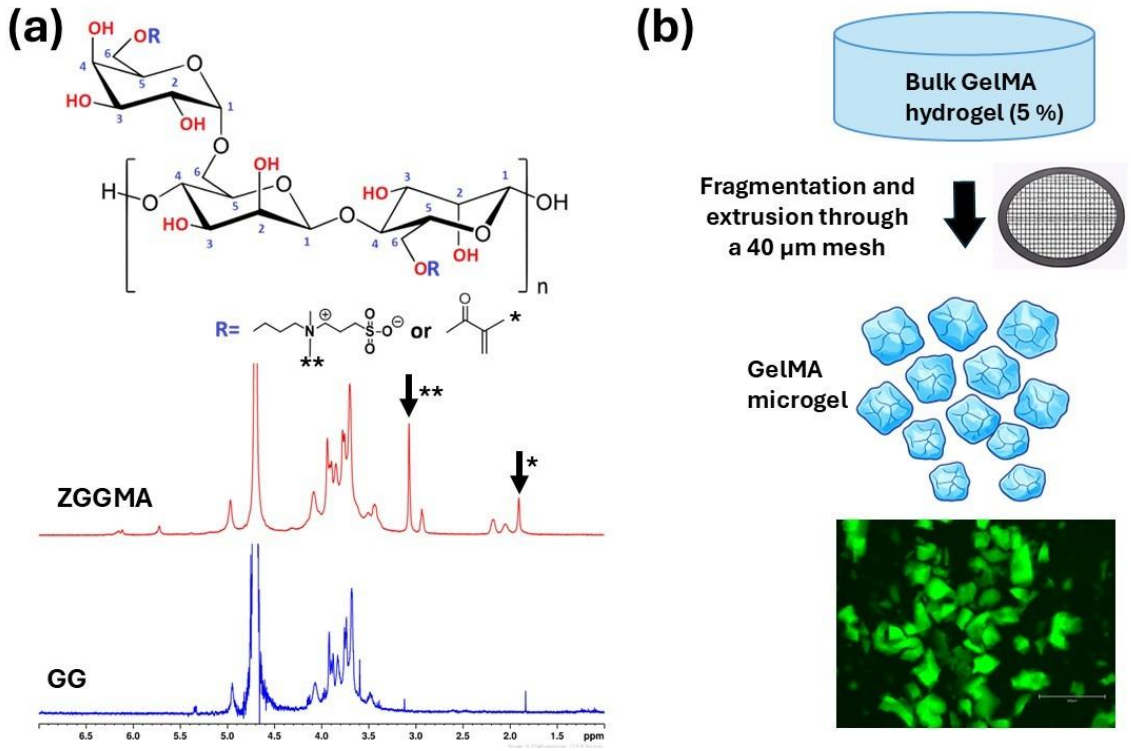

Fig. 1. (a) chemical structure of ZGGMA and ^{1}H NMR spectra; (b) schematic representation of GelMA microgel preparation supported by a CLSM image for the fluorescein-labelled microgels (scale bar = 300 µm)

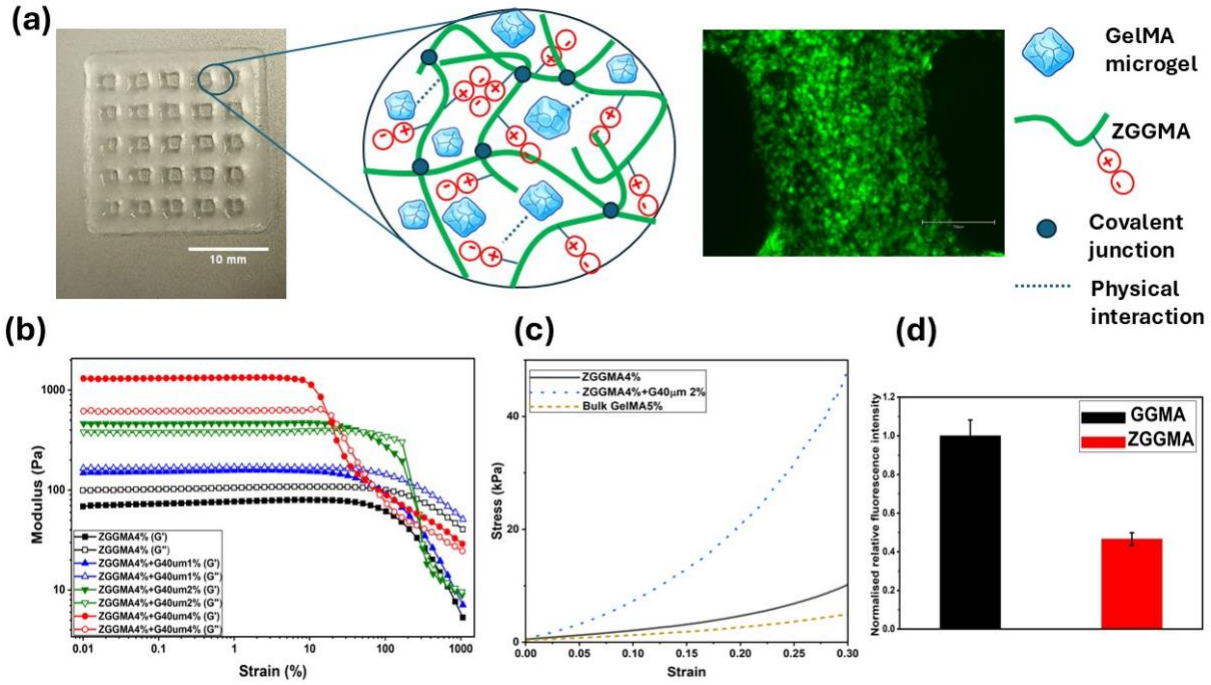

Fig. 2. (a) A3D printed grid using ZGGMA4%/fluorescein-labelled GelMA microgels 2% with a schematic representation of the hydrogel composite and CLSM image of part of the grid. (b) Amplitude sweep analysis (c) Stress-strain curves for ZGGMA 4%, bulk GelMA hydrogel 5% and ZGGMA4/GelMA microgel 2% composite; (d) Normalised anti-fouling behaviour of ZGGMA compared with GGMA (n=3)

FROM WOOD TO TEXTILES WITH IMPROVED WATER REPELLENCY

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ABSTRACT

Man-made cellulosic fibres are an environmentally friendly alternative to synthetic textile fibres like polyester, but they are limited by their high water absorbency. Current water-repelling treatments use chemicals, such as PFAS, that can be harmful to the environment and human health and are facing restrictions and bans worldwide. The treatments are typically applied on the surface of fabrics often leading to poor durability during washing. A potential improvement is the incorporation of non-toxic hydrophobising agents already during fibre production, integrating the chemical within the fibre structure. Alkyl ketene dimer (AKD) wax was added into a spinning solution of cellulose dissolved in ionic liquid prior to spinning into fibres using dry-jet wet spinning. AKD is a hydrophobic wax widely used in the paper industry since the 1950s. It is applied as a sizing agent to control the wettability of paper and board products by making cellulose fibres more hydrophobic. The addition of AKD allowed fibre spinning at similar conditions as a solution without wax. The resulting fibres exhibit increased hydrophobicity compared to reference fibres. The addition of the wax leads to a small reduction of the mechanical properties of the fibres but can still retain higher tenacity and elongation than some commercially available fibres such as cotton. Adding the hydrophobising agent during the fibre formation step would eliminate the need for hydrophobising post-treatment of fabrics, requiring less time, energy, and chemicals. Furthermore, introducing hydrophobicity already at the fibre-level could lead to a more durable treatment, as the hydrophobic agent is embedded inside the fibres as opposed to solely on the surface of fabrics. This would prolong the life of water-repellent textile products, decreasing their environmental impact.

KEYWORDS

Cellulose, textiles, hydrophobic, lyocell

EXPLORING COMPOSITE POLYSACCHARIDE HYDROGEL-BASED BIOINKS WITH BIOBASED MICRO- AND NANOSTRUCTURED ADDITIVES FOR THE 3D BIOPRINTING OF DIFFERENT CELL LINES

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ABSTRACT

Three-dimensional (3D) bioprinting has revolutionized biomedical research as a disruptive tool for the biofabrication of living 3D structures that may be used as analogues of human living tissues. This technique is characterized by precise layer-by-layer deposition of cell-laden materials, viz. “bioinks”, in a computer-controlled manner, originating living constructs with defined shape and size. Biopolymeric hydrogel-based bioinks are a popular choice for 3D bioprinting due to their favorable properties, like their facile crosslinking under mild conditions, resemblance to the extracellular matrix and biocompatibility. However, pristine biopolymeric hydrogels often fail to provide suitable properties for these endeavors, and precise adjustments to their characteristics are challenging. Micro- and nanostructured additives offer a promising solution: biobased nanofibers and particles like lysozyme amyloid nanofibrils (LNFs), nanofibrillated cellulose (NFC) or spherical cellulose-based particles have great potential to improve the properties of these bioinks, or even to grant new functionalities.

The present communication explores distinct studies from our research group (BioPol4fun at CICECO – Aveiro Institute of Materials) concerning the design and characterization of composite bioinks based on biopolymeric hydrogels of alginate, pectin, gellan gum, carrageenan, and cellulose. The addition of particles (including

LNFs, NFC or spherical cellulose particles) to the formulations lead to enhanced rheological properties and printability, increased stability of printed biostructures and in some cases, the development of new active capabilities (like drug-delivery potential or conductivity). All the bioinks were precisely tailored envisioning specific applications and type of tissues, and their potential for the 3D bioprinting of different cell lines, such as keratinocytes (HaCaT), cardiomyocytes (H9c2), chondrocytes (ATDC5), and melanoma cells (A375) was evaluated.

Overall, these studies confirm the potential of using biobased nano- and microstructures to design new and improved biopolymeric composite bioinks for 3D bioprinting, establishing a library of formulations suited for the biofabrication of 3D living structures with enhanced characteristics that may be used in biomedical contexts like tissue regeneration, pathophysiology studies or drug development.

ACKNOWLEDGMENTS

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KEYWORDS

3D bioprinting, biobased particles, composite hydrogel-based bioinks, natural nanofibers, polysaccharides

LONG-TERM BARRIER STABILITY OF POLYSACCHARIDE–BEESWAX COATINGS ENABLED BY TAILORED RECYCLED PAPER STRUCTURES

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ABSTRACT

The development of sustainable packaging solutions requires enhancing the barrier performance of renewable materials such as paper, which inherently lacks resistance to moisture, gases, and grease. This study investigates how engineering the physical structure of recycled paper through mechanical refining can optimize the performance of a novel bio-based barrier coating while ensuring long-term stability.

Recycled fiber pulp (fluting/liner ratio 35/65) was refined at varying PFI intensities (1500-10000 revolutions) to produce handsheets (120 g/m^2) with controlled porosity using a Rapid-Köthen former (ISO 5269-2). The substrates were coated with a beeswax-in-water Pickering emulsion stabilized by TEMPO-oxidized cellulose nanofibers, using a K Control Coater. Two application rods were employed to obtain coatings with different surface topographies: a smooth coating (S-coating) and a rough coating (R-coating), while also varying coating thickness. Coated samples were pre-dried with air at 50 ºC for 1 min and further dried for 4 min under pressure. Barrier properties (air permeability, water vapor transmission rate (WVTR), contact angle, and grease resistance) and morphology were evaluated. Microscopic images revealed that low-refined, highly porous papers allowed deep penetration of the coating into the fiber network, limiting the formation of a continuous surface layer. In contrast, highly refined and denser substrates exhibited reduced porosity, promoting coating retention at the surface and enabling the formation of a more uniform and continuous film. This improved film formation translated into significant enhancements in barrier performance. The synergy between high refining intensity and increased coating thickness resulted in up to a 94% reduction in water vapor transmission rate, over 90% decrease in air permeability, and an increase in water contact angle to 110º. Grease resistance was also substantially improved, reaching a Kit rating of 10. Besides, long-term performance tests conducted after more than six months of storage demonstrated that the barrier properties remained unchanged (see Figure 1), confirming the stability of the coating system and its suitability for practical applications.

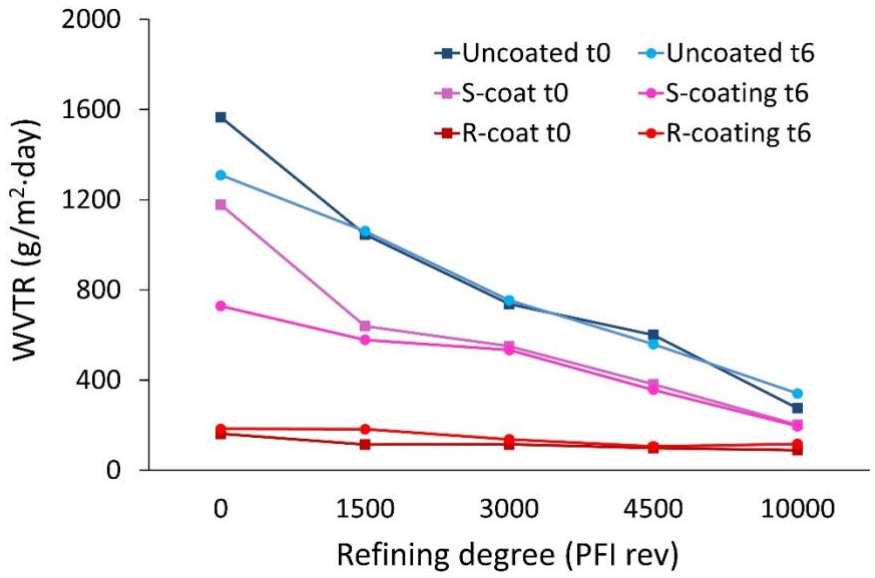

Figure 1. WVTR evolution with refining degree for uncoated and coated papers (S- and R-coating) at t0 and after long-term storage (t6), highlighting the stability of barrier performance over time.

In conclusion, mechanical refining is identified as a key strategy for tailoring paper structure to control coating distribution and maximize barrier efficiency, enabling the development of high-performance and sustainable packaging materials.

KEYWORDS

Mechanical refining, Polysaccharide-beeswax-coatings, Long-term stability, Barrier properties

A GREEN BINARY SOLVENT FOR THE ELECTROSPINNING OF CELLULOSE ACETATE

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ABSTRACT

Sustainable material development has intensified the need for environmentally benign solvent systems in polymer processing. In electrospinning, solvent selection plays a key role in determining both fiber properties and process sustainability. However, cellulose acetate (CA) electrospinning commonly relies on hazardous solvents, limiting its green potential. In this work, a sustainable electrospinning approach for CA is demonstrated using a binary solvent system based on dimethyl carbonate (DMC) and dimethyl sulfoxide (DMSO). CA solutions were prepared with varying DMC/DMSO ratios (100/0 to 50/50 w/w), and the effects of solvent composition on processability and fiber properties were investigated. The results show that solvent composition strongly influences fiber morphology and performance. Higher DMC content promotes the formation of porous fiber surfaces due to its high volatility, whereas increasing DMSO content results in smoother fibers. Additionally, the mechanical properties of the electrospun mats vary significantly with solvent ratio. These findings establish DMC/DMSO as an effective green solvent system for CA electrospinning, enabling tunable fiber properties and offering a promising route toward more sustainable production of fibrous materials for eco-friendly applications.

KEYWORDS

Sustainable production, fibers, biobased, green chemistry.

NANOCELLULOSE-BASED FILMS MODIFIED WITH A NATURAL ALDEHYDE FOR ANTIBACTERIAL WOUND DRESSING APPLICATIONS

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ABSTRACT

Owing to its attractive properties-including its origin from renewable natural resources, superior mechanical resistance, biocompatibility, biodegradability, good water retention capacity as well as availability for modification by various chemical and physical routes due to the numerous hydroxyl (OH) groups on its surface, cellulose has experienced a resurgence within the scientific community in the last decades, through its nanoscale form, commonly referred to as “nanocellulose” (NC). Compared to its microscopic counterpart, NC brings with it a higher specific surface area, higher number of accessible OH groups at the surface [1], better colloidal stability in water, as well as optical transparency [2]. While all these properties would render NC a promising candidate as material for wound dressings, the lack of antibacterial activity of NC limits its application for this purpose [1].

This work has as objective the chemical modification of NC films with a natural aldehyde with antibacterial properties to endow NC with antibacterial effect and obtain antibacterial wound dressings based on NC for biomedical applications.

NC was prepared by the microfluidization of an aqueous suspension of microcrystalline cellulose and the NC films were obtained by the vacuum filtration of the resulting NC suspension on a membrane followed by hot pressing. The covalent grafting of the natural aldehyde onto NC was performed by prior functionalization of the NC films with primary amino (NH_2) groups, followed by the reaction of these groups with the aldehyde (CH=O) groups of the modifying agent. The chemical structure of the modified NC films was investigated by Fourier-transform infrared (FTIR) and Raman spectroscopies, their morphology was examined by scanning electron microscopy, their thermal stability was studied by thermogravimetric analysis, their surface properties were examined by water contact angle

measurements, while their antibacterial properties against various strains were assessed by the disc-diffusion method.

The successful chemical modification of the NC films with the natural aldehyde was confirmed by FTIR and Raman spectroscopies, while antibacterial tests indicated that the obtained chemically modified NC films have promising antibacterial effect.

KEYWORDS

nanocellulose, microfluidization, aldehyde, wound dressings, antibacterial

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SULFATED POLYSACCHARIDE-NANOCELLULOSE FORMULATIONS FOR THE DEVELOPMENT OF COATINGS FOR ORTHODONTIC APPLICATIONS

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ABSTRACT

The petroleum-derived products have become a great part of our days, and have inflicted problems in many industries and, more importantly, in the quality of our lives. Research paths seek to find alternatives in natural sources, like biopolymers or bioactive compounds, to create new functional materials with extensive studies carried out for health-related applications. Fucoidan (F), a sulfated polysaccharide isolated from brown seaweed, can be attractive for obtaining coatings for biomedical applications due to its origin from renewable natural resources, water solubility, and, in particular, its documented antibacterial, antifungal, antioxidant, and antiviral activities [1]. However, aqueous solutions of fucoidan generally present low viscosity. This may constitute a limitation in its application as material for bio-coatings, as multiple deposition cycles would be necessary for achieving the desired coating thickness, it can lead to fragile or brittle coatings and increased risk of dissolution or wash off in aqueous environments, such as physiological fluids. Therefore, the incorporation of cellulose nanofibers (CNFs), unmodified or chemically modified by 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO)-mediated oxidation (oCNFs) may serve as an effective approach to enhance/adjust the rheological properties of fucoidan solutions and slow down the disintegration of the resulting F-based coatings under physiological conditions, while preserving the bio-based character of fucoidan[2][3]. In this work, various formulations based on F and CNFs/oCNFs with different compositions were prepared to obtain antibacterial coatings for orthodontic applications through the spray coating method. The materials were characterized by assessing their thermal stability through thermogravimetric analysis (TGA), morphology and other structural characteristics through scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR), and their resistance against selected bacterial strains.

KEYWORDS

Nanocellulose, fucoidan, coatings

Acknowledgments: This work was supported by a grant of the Ministry of Education and Research, CNCS - UEFISCDI, project number PN-IV-P1-PCE-2023-1557, contract 37PCE/2025 (WASTE2COAT) within PNCDI IV.

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FUNGAL BIOLUMINESCENCE IN LIGNOCELLULOSIC MATERIALS

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ABSTRACT

"Bioluminescence is a versatile tool for bio-marking and sustainable lighting. While bacterial and algal systems are well studied, fungal bioluminescence remains underutilized. Recently, our group achieved bioluminescent wood structures using the white-rot fungus *Desarmillaria tabescens*. In this presentation, the **research topic of bioluminescence by the colonization of D. tabescens of lignocellulosic materials is described.**

Upon colonizing balsa wood, the fungus produces luciferin via the caffeic acid cycle and glows when exposed to oxygen. However, light intensity in these systems is dependent on the wood's inherent structural variability and hygroscopicity, which limit oxygen diffusion and moisture consistency. Therefore, in a second recent work we explore in more detail the impact of wood density, species, anatomy, moisture content, and colonization period on the resulting bioluminescence. Such work hypothesises that the species-dependent wood decay, and specifically guaiacyl lignin consumption in pine wood by *D. tabescens*, increases the intensity and duration of the glow.

Notably, though, in our latest findings, we observed that the presence of wood as a substrate, and specifically lignin, is not necessary to obtain bioluminescence. Therefore, we are now exploring **nanocellulose hydrogels** as a tunable alternative to wood for sustaining *D. tabescens* activity. We hypothesize that the high moisture retention and structural versatility of nanocellulose can overcome the mentioned limitations. We aim to use material design and 3D printing—incorporating microfibrillated cellulose (MFC) and cellulose nanocrystals (CNC)—to fabricate porous architectures that optimize oxygen exposure and mycelial growth.

Preliminary results demonstrate that *D. tabescens* successfully forms bioluminescent **rhizomorphs** within the nanocellulose bulk. Moisture and oxygen cycling tests indicate a cumulative increase in glow intensity, suggesting luciferin accumulation.

These fungal composites offer potential for developing self-sustaining stimuli-responsive sensors and dim-lighting applications in moist environments. Ongoing research focuses on quantifying luciferin production and retention in the substrate, assessing the viability of 3D printing with photo-curable materials, and evaluating the impact of rhizomorph growth on material integrity."

KEYWORDS

Nanocellulose, Living Materials, Bioluminescence, 3D printing, Mycelium.

BANANA PSEUDOSTEM FLOUR-BASED POLYMERIC FILMS: MORPHOLOGICAL, THERMAL, AND UV-SHIELDING PROPERTIES

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ABSTRACT

The growing demand for sustainable materials has driven the development of biodegradable alternatives from agro-industrial residues. A polymeric film was developed from banana pseudostem flour, aiming to valorize this residue as a raw material for biodegradable materials with potential application in sustainable packaging. The pseudostem flour was subjected to hydrothermal treatment by dispersion in water (1:14 flour-to-water ratio) under stirring, followed by autoclave processing (121 °C, 30 min, 1.2 kgf cm^{-2}) and subsequent cooling in an ice bath. Glycerol (30%) and pectin (20%) were then added, based on the dry flour mass. The film-forming suspension was homogenized, vacuum degassed, and cast onto glass plates using a casting bar, producing films with an initial thickness of 2 mm, which were dried at 50 °C for 20 h. Morphological characterization by scanning electron microscopy (SEM) revealed a heterogeneous surface, with an irregular distribution of fibrous flour particles throughout the polymeric matrix. The micrographs showed relatively compact regions interspersed with pores and discontinuities, indicating partial interaction among the film components. Overall, these discontinuities are attributed to the heterogeneity of the lignocellulosic matrix, limited intermolecular interactions among formulation components, and stresses generated during drying, resulting in a less homogeneous structure with pores and microcracks. The cross-section exhibited a rough and stratified internal structure, characteristic of plasticized lignocellulosic materials. Thermogravimetric analysis (TGA) showed an initial mass loss below approximately 120 °C, associated with evaporation of water and volatile compounds. At intermediate temperatures (150–250 °C), degradation related to low thermal stability components and the plasticizer was observed, while the major weight loss occurred between 250 and 320 °C, corresponding to the degradation of the main organic constituents. Above approximately 350 °C, a final residue was observed, associated with carbonized and more recalcitrant fractions. The material presented a transparency of 37.82% in the visible region (400–780 nm), indicating moderate light transmission. In contrast, it showed high ultraviolet blocking efficiency, with S_UVA of 35.58%, S_UVB of 99.49%, and S_UVC of 99.65%. These results demonstrate excellent UV-shielding capacity, especially in the UVB and UVC ranges, highlighting its potential for sustainable packaging with photoprotective functionality.

KEYWORDS

Lignocellulosic materials, Biodegradable materials, Film casting, Microstructure, Thermal stability.

SEQUENTIAL EXTRACTION OF PECTIN AND CELLULOSE FROM LEMON PROCESSING RESIDUE FOR SUSTAINABLE FOOD PACKAGING

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ABSTRACT

The accumulation of agro-industrial by-products and plastic pollution are two major global challenges that require sustainable solutions. The use of industrial processing by-products to produce biodegradable food packaging materials is a promising strategy to address both issues simultaneously. Among various industrial residues, lemon processing waste generated after juice production is particularly valuable because of its high pectin and cellulose content, which can be recovered and converted into functional biomaterials. This study aims to valorize lemon processing residue through a cascade green extraction process for pectin and cellulose and to evaluate their application in edible food packaging films. For this purpose, pectin was extracted from dried lemon residues using microwave-assisted extraction. Cellulose was subsequently obtained from the remaining residue through enzymatic pretreatment (Viscozyme, pectinase, and their sequential combination), followed by high-pressure homogenization (HPH). The extracted pectin was characterized based on its monosaccharide composition, while the cellulose fraction was analyzed for its physicochemical, structural, and morphological properties using Fourier-transform infrared spectroscopy (FTIR), dynamic light scattering (Zeta-sizer), scanning electron microscopy (SEM), and X-ray diffraction (XRD). Edible composite films were then developed using lemon residue-derived pectin as the film-forming matrix and cellulose as a reinforcing agent. The films were evaluated for their mechanical strength, barrier properties, transparency, and water sensitivity to assess their suitability for food packaging applications. The characterization results confirmed that the extracted fractions were rich in pectin and cellulose and exhibited their typical properties, such as high galacturonic acid content in the pectin-rich fraction, high crystallinity in the cellulose-rich fraction, and good water dispersibility, which facilitated homogeneous distribution in the film-forming solution. By incorporating cellulose into pectin films, the tensile properties were improved, and water vapor permeability reduced in comparison to pectin-only films. These findings highlight the potential of converting citrus processing waste into sustainable packaging solutions that contribute to zero-waste and chemical-free processing, circular bioeconomy development, and decreased reliance on petroleum-based plastics.

KEYWORDS

Pectin extraction, Cellulose fibrillation, edible packaging films, Waste valorization

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THE VERSATILITY OF CHITOSAN ITACONATE IN COMBINATION WITH LIGNOCELLULOSIC MATERIALS

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ABSTRACT

This presentation demonstrates the versatility of aqueous chitosan itaconate solutions as a bio-based, multifunctional system in a variety of applications with lignocellulosic materials. Chitosan itaconate exploits the orthogonal reactivity of itaconic acid. While the carboxylic groups enable the protonation of chitosan amino groups to form a water-soluble salt, thermally initiated radical polymerisation transforms the salt into a water-insoluble interpolyelectrolyte complex. This yields on surfaces in a robust and durable transparent film.

The aqueous chitosan itaconate solutions exhibit shear-thinning and thixotropic behaviour, allowing for uniform application and deeply penetrates lignocellulosic substrates. Within the substrate, the functional groups of chitosan and the second, free carboxyl group of itaconic acid interact with the hydroxyl groups of cellulose through extensive hydrogen bonding. These interactions act synergistically, leading to strong adhesion of the interpolyelectrolyte complex to the substrate. On the surface, the system forms a thin, hydrophobic film with a water contact angle of approximately 93° and provides effective protection against UV-induced degradation. In addition, presence of chitosan itaconate significantly reduces flammability of lignocellulosic materials: a single application lowers the burning rate, while multiple applications can completely suppress combustion. When employed as an adhesive for solid wood, chitosan itaconate achieves high bond strengths. Tensile-shear tests reveal cohesive failure in the wood rather than cohesive failure in the joint or adhesive failure at the interface. Furthermore, fibreboards manufactured using chitosan itaconate as a binder exhibit bending strengths of up to 77 $N \cdot mm^{-2}$, exceeding those of solid wood (approximately 70 $N \cdot mm^{-2}$) and conventional oriented strand boards (approximately 25 $N \cdot mm^{-2}$).

Overall, chitosan itaconate combines easy application from aqueous solution with excellent mechanical performance, surface protection, and intrinsic flame-retardant properties. It therefore represents a powerful, sustainable alternative to conventional coating and binder systems and offers a tangible contribution to more environmentally friendly construction materials.

KEYWORDS

Chitosan, Adhesives, Binder, Coating, Wood

THE BONDING BEHAVIOUR OF CHITOSAN INTERPOLYELECTROLYTE COMPLEXES ON DISSIMILAR SUBSTRATES

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ABSTRACT

Commercial adhesives often face sustainability challenges, as many rely on petrochemical components and organic solvents. Our group has previously developed a fully bio-based, water-based wood adhesive that exploits the natural molecular affinities between chitosan itaconate and cellulose. While this system shows excellent bonding performance towards wood, their ability to bond non-polar substrates is limited.

In this work, we investigate fully aqueous chitosan-based polyelectrolyte adhesives for bonding wood to PMMA. The systems combine maleic acid as a PMMA-activating component with chitosan as a wood-compatible binder. We study how the structure of dicarboxylic acids, the chitosan chain length, the acid equivalent ratio and radical-induced polymerisation influence processability, network formation and adhesive performance. Polyelectrolyte complexes were prepared using long-chain chitosan and chitosan oligosaccharide with different maleic acid equivalents, with and without potassium peroxodisulphate (PPS) as radical initiator.

For chitosan-maleate systems, FTIR analysis confirms the formation of ionically linked and hydrogen-bonded network structures. In PPS-containing formulations, decreased C=C double bond signals and a more heterogeneous carbonyl region indicate partial consumption of maleic acid double bonds and the formation of oligomeric, multivalent maleate species. These structural changes correlate with increased network connectivity and improved cohesion. Reducing the chitosan chain length strongly lowers the viscosity of the formulations, enabling higher solid contents and improved processability.

Finally, we relate these network characteristics to adhesion in wood-PMMA bondings. It was found that aqueous maleic acid alone yields strong PMMA-PMMA bonds but poor wood-PMMA adhesion. In contrast, chitosan-maleate networks, especially with PPS, outperform maleic acid solutions and shift the failure from the adhesive layer into the wood. The combined rheological, spectroscopic and mechanical data demonstrate how molecular design of ionic chitosan-maleate networks tunes the transition from low-viscosity polyelectrolyte complexes to connected networks capable of bridging hydrophilic wood and hydrophobic PMMA.

KEYWORDS

Chitosan, Adhesion, Polyelectrolytes, PMMA

STRUCTURAL ELUCIDATION OF A RHAMNOSE-RICH EXOPOLYSACCHARIDE FROM *DIACRONEMA VIRESCENS* MICROALGAE FOR SUSTAINABLE PRODUCTION OF L-RHAMNOSE

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ABSTRACT

L-rhamnose is a rare monosaccharide with numerous applications in the food, cosmetics, and pharmaceutical industries. This sugar does not occur in its free form in nature but is instead found bound to other monosaccharides or aglycone compounds. Currently, it is mainly produced through the hydrolysis of rutin, a flavonoid extracted from citrus fruits and flowers such as *Sophora japonica*. However, this production route requires the use of aromatic, toxic, and corrosive solvents. The low yields of rutin in comparison with the total biomass and the cultivation of these terrestrial plants lead to the use of arable land and production of important quantities of coproducts. The French Agence Nationale de la Recherche (ANR) RAh project [ANR-23-CE43-0011] aims to develop alternative processes for rhamnose production based on novel sources, such as microalgal exopolysaccharides, combined with innovative enzymatic depolymerization and monosaccharide purification strategies by centrifugal chromatographic partition. In this context, the present study seeks to elucidate the structure of an exopolysaccharide produced by *Diacronema virescens* RCC 4037 microalgae using a combination of spectroscopic and chromatographic techniques, with the objective of identifying key enzymes required for its depolymerization. The exopolysaccharide obtained under phototrophic conditions in bioreactor was subjected to diverse chemical modification and analysis: FTIR, biochemical composition, monosaccharides composition, linkage analysis using the Hakomori method (Hakomori, 1964), desulfation, and NMR spectroscopy to determine its monosaccharide sequence. The results indicate that the polymer is a sulfated heterorhamnan composed of 63.4% rhamnose. The rhamnose residues are arranged in a repeating sulfated trisaccharide motif within the backbone, consisting of three rhamnose units linked through α-(1→3) and β-(1→4) glycosidic bonds. A transcriptomic study of the strain is planned to identify, through bioinformatic analyses, candidate genes associated with the biosynthetic pathways of this polymer. This study represents a first step toward the sustainable production of rhamnose and provides an additional contribution to the deeper understanding of the exotic polysaccharide structures produced by microalgae.

KEYWORDS

Microalgae, Exopolysaccharides, Structural elucidation, L-rhamnose

CANYACEL – REGENERATED TEXTILE FIBRES FROM *ARUNDO DONAX* WASTES OF URBAN REED MANAGEMENT

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ABSTRACT

Regenerated cellulose textile fibres (RCFs) have been known for over a hundred years; however, since 2000, interest in this material has grown exponentially again due to its potential to replace petroleum-based textile fibres, being considered a response to social and environmental pressures on the textile industry. Using wet-spinning technology, RCFs are produced from highly purified wood-based cellulose pulp (dissolving wood pulp, DWP), which is expected to be sourced from recycled textile waste, agricultural residues, and non-wood plants in the emerging "4th wave" of RCFs growth [1]. This shift promises to break the industry's dependence on virgin wood pulp and redefine what's possible for sustainable textiles. *Arundo donax*, a robust perennial grass, thrives with rapid growth and remarkable adaptability to cold or saline-alkali environments, and exhibits exceptional resistance to diseases and pests [2]. In Europe, this aggressive invader wreaks havoc on both ecosystems and economies, especially in urban environments.

The CANYACEL project explores the potential of *Arundo donax* waste from urban reed bed management to produce dissolving-grade pulp, which can be converted into valuable textile fibres via wet-spinning technology using an imidazolium-based ionic liquid as the solvent. Preliminary results of the cane waste organosolv cooking [3] showed that the unbleached DRP (dissolving reed pulp) composition (Figure 1) is close to that typical of DWP, except for the higher hemicellulose content (almost 2x higher). The DRP intrinsic viscosity (ISO 5351) was 916 mL/g, above the desired DWP range (400-600 mL/g). The results of optimising organosolv process parameters to obtain a dissolving-grade pulp, as well as studies on pulp dissolution and spinnability into textile fibres, will be presented and discussed.

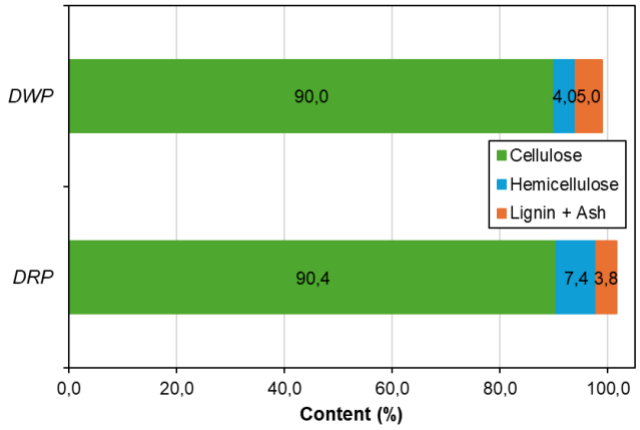

Figure 1. Comparison of the dissolving pulp prepared from reed residues (DRP) composition and a typical dissolving wood pulp (DWP) [4].

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KEYWORDS

Arundo donax, organosolv, dissolving pulp, wet spinning, regenerated cellulose fibre

ELECTRO-MEDIATED SYNTHESIS OF REACTIVE CELLULOSE DERIVATIVES

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ABSTRACT

The transition toward sustainable biobased materials is frequently hindered by the lack of regiospecific control in polysaccharide functionalisation. While cellulose offers a robust scaffold for advanced materials, its chemical modification is often compromised by the aggressive nature of stoichiometric oxidants. Conventional pathways typically demonstrate poor kinetic control, leading to the indiscriminate overoxidation of primary hydroxyl groups to carboxylates, thereby exhausting the potential for further specific ligation.

This study presents a unified electrochemical strategy to circumvent these limitations by utilising an ionic liquid as both a solvent and an electrolyte. This homogeneous medium facilitates the dissolution of the cellulose matrix, allowing for a TEMPO-mediated electrocatalytic process with enhanced mass transport and site accessibility. By regulating the electrochemical potential in a basic environment, we achieved selective oxidation at the C6 position, effectively stabilising the cellulose at the aldehyde stage.

Quantitative analysis confirmed a functionalisation of 300 µmol/g, with negligible carboxylic acid formation, demonstrating the potential to reach 500 µmol/g through parameter optimisation. This cogent evidence confirms the establishment of a robust synthetic route that operates with high fidelity within the ionic liquid phase. Ultimately, this work provides an efficient methodology for the derivation of renewable polysaccharides and supports the broader development of next-generation bio-based materials.

KEYWORDS

Electrochemical oxidation; C6-aldehyde cellulose; Ionic liquids

DOES BARLEY GENOTYPE MATTER? IMPACT ON THE FLAMMABILITY OF BARLEY STRAW-BASED PANELS AND COMPOSITES

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ABSTRACT

Within the context of the bioeconomy and circular economy, and aiming at valorising barley straw for higher value-added applications, the European BEST-CROP project is based on a collaborative framework involving academia, plant breeding companies, and biomass processing stakeholders to develop new high-yield barley varieties. The project strategy focuses on the development of these varieties through genetic engineering to: enhance biomass production while improving water and nitrogen use efficiency; increase protein content for the development of alternative biolubricants and animal feed sources; and control cellulose and lignin contents for the production of straw-based polymer composites and construction panels. This latter aspect constitutes the core research activity of the PCH team at IMT Mines Alès, whose objective is to investigate the influence of barley diversity, through its genotypic variability, on the thermal behaviour and fire reaction of raw barley straw and barley straw-based composites, such as insulating panels, for applications in the construction sector (**Figure 1**).

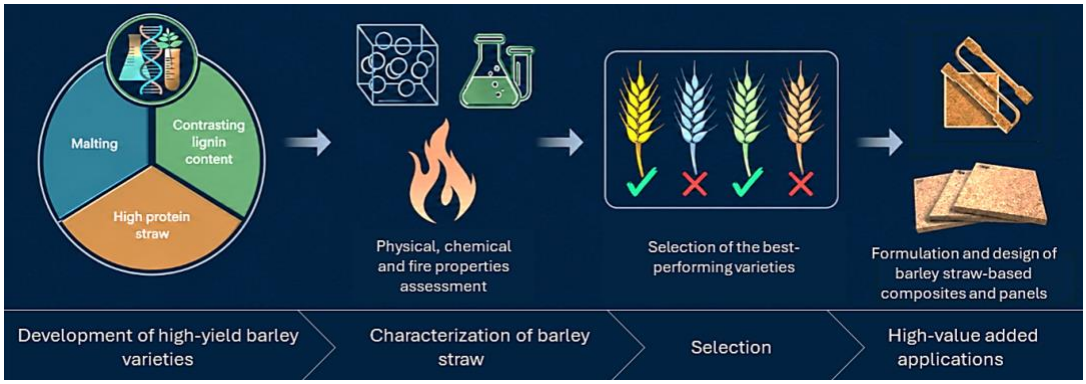

FIGURE 7 SUMMARY OF THE RESEARCH STRATEGY STEPS FOR BEST-CROP PROJECT.

The study first involves the characterization of the physical properties, such as density and moisture content, as well as the thermal properties of the straw using thermogravimetric analysis and micro-

scale combustion calorimetry. For the fire reaction assessment, the samples were submitted to the cone calorimeter test. Different varieties were compared considering their different characteristics, such as high protein content, contrasting lignin contents and malting varieties. For comparison purposes, wheat and rapeseed straws, which are currently highly used for insulation panels, were also investigated. One of the main parameters measured by the cone calorimeter test is the peak of heat release rate (pHRR), obtained from the curve of heat release rate (HRR) as a function of the time. **Figure 2** illustrates the relationship between the pHRR and the bulk density of the samples tested. By analysing these data, it is possible to observe that not only the bulk density of samples influences the pHRR, but also that the chemical composition plays an important role in the fire reaction of the different straw varieties.

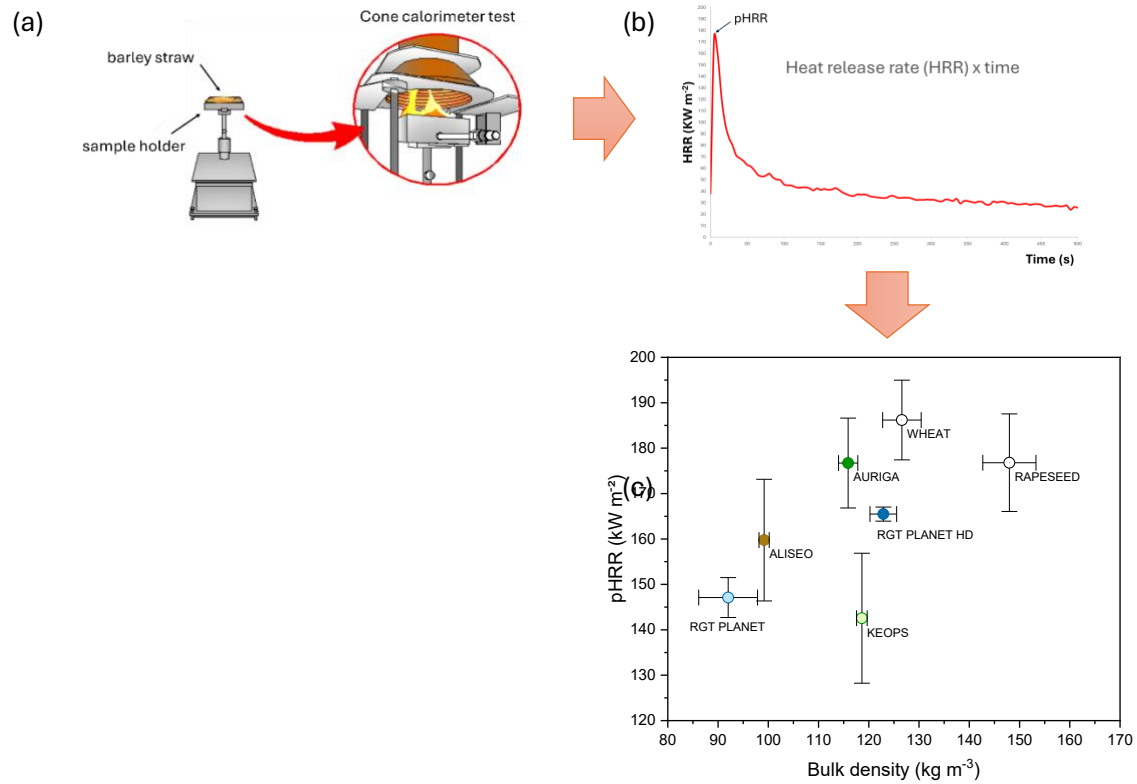

FIGURE 8 (A) SIMPLIFIED SCHEME OF A CONE CALORIMETER TEST; **(B)** AN EXAMPLE OF A HRR CURVE OBTAINED FROM THE CONE CALORIMETER TEST; **(C)** AVERAGE PHRR FOR SOME BARLEY STRAW VARIETIES TESTED AS FUNCTION OF THE BULK DENSITY. RGT PLANET: A MALTING VARIETY; ALISEO: HIGH PROTEIN STRAW; AURIGA: HIGH LIGNIN CONTENT; KEOPS: LOW LIGNIN CONTENT; RGT PLANET HD: RGT PLANET WITH A HIGHER BULK DENSITY. WHEAT AND RAPESEED

These parameters will be compared with those of other European straws and subsequently integrated into the formulation and design of barley straw-based composites. The flammability of these composites will then be evaluated and correlated with the properties of the studied barley varieties.

KEYWORDS

Barley straw, construction panel, fire reaction, thermal properties, flammability

EFFECTS OF THERMOMECHANICAL TREATMENTS PROMOTING HORNIFICATION ON THE MICROSTRUCTURE AND FUNCTIONALIZATION OF CELL WALL POLYSACCHARIDES FROM APPLE CO-PRODUCTS

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ABSTRACT

Apple pomace, the fibrous residue generated during juice production (comprising pulp, peel, seeds, and stems), is a major agri-food by-product rich in dietary fiber, phenolic compounds, and carbohydrates. Its valorization as a functional food ingredient represents a promising strategy to increase dietary fiber intake while reducing the environmental impact of agro-industrial waste. The functional performance of apple pomace in food products is closely linked to its hydration properties, which influence both sensory attributes (such as taste and texture) and potential gastrointestinal benefits. These properties are strongly dependent on the material's microstructure, which can be modified through processing. This study investigates the impact of thermomechanical processing, specifically extrusion, on the microstructure of apple pomace. The effects of processing were primarily assessed through hydration properties, namely water retention capacity and solubility, as well as microscopy analysis to characterize structural changes. To better understand hydration behavior, a kinetic approach was developed to quantify both water absorption and the release of soluble fractions over time. Hydration kinetics were evaluated by comparing untreated and extruded samples during water exposure. For extruded samples, the influence of processing parameters, particularly screw configuration, was also examined. The results show that water retention capacity kinetics differ significantly between extruded and non-extruded samples and are strongly influenced by extrusion conditions. These findings provide valuable insights into how processing-induced microstructural modifications affect hydration behavior. Overall, this work highlights the strong interplay between processing, structure, and functionality in apple pomace, contributing to the optimization of processing conditions and supporting sustainable strategies for the valorization of agri-food by-products.

KEYWORDS

Apple pomace, thermomechanical processing, hydration kinetics, microstructural modification, dietary fiber

IMPACT OF MW AND DS ON THE AGGREGATION AND EMULSIFYING PERFORMANCE OF OSA-MODIFIED HEMICELLULOSES

Nizam P A, Wangfang Deng, Martti Toivakka, Chunlin Xu

ABSTRACT

Softwood hemicelluloses are promising, renewable candidates for sustainable emulsion stabilisation. Although chemical modification with hydrophobic moieties is a known strategy, a fundamental understanding of how their inherent structural complexity affects emulsification remains limited. In aqueous environments, these polysaccharides often exist as a biphasic colloidal system, containing a mixture of dissolved chains, swollen coils, and suspended aggregates. This study investigates the impact of these structural characteristics on emulsion stability.

Three hemicellulose fractions of varying molecular weight (low, medium, and high Mw) were modified with octenyl succinic anhydride (OSA) across a range of degrees of substitution (DS). Preliminary results indicate a positive correlation between the degree of substitution and the extent of aggregate formation, as evidenced by dynamic contact angle measurements. These aggregates significantly influence the assembly behaviour at the oil-water interface, suggesting that the hydrophobic modification alters both the self-assembly of the polysaccharide and its surface activity.

The ongoing work involves characterising the size distribution and stability of these colloidal systems through systematic interfacial and rheological testing. By correlating the molecular weight, modification density, and aggregation behaviour with the resulting emulsion stability, this research aims to establish design principles for high-performance, hemicellulose-based emulsifiers.

KEYWORDS

Hemicellulose, galactoglucomannan, emulsion

ROLE OF PECTIN CHARGE IN MODULATING PECTIN-PROTEIN INTERACTIONS WITHIN THE PLANT CELL WALL

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ABSTRACT

Pectins are matrix polyanionic polysaccharides of plant cell walls whose physicochemical properties depend strongly on their degree of methylation (DM). They are the most abundant polymer in expanding cells. We showed previously that peptides of the RAPID ALKALINIZATION FACTOR (RALF) family contribute to drive the cell wall organization through their interaction with pectin [1] [2].

However, how charge variations associated with the DM influence pectin–protein interactions remains poorly understood.

This study investigates the impact of pectin charge on their interaction with proteins by comparing 3 pectins differing in their proportions of free charges. Pectin surface charge was assessed by zeta potential measurements as function of the aqueous medium (H_2O or sodium acetate buffer) and the ionic strength (achieved by adding sodium salt). Quartz crystal microbalance with dissipation monitoring (QCM-D) was employed to characterize pectin adsorption onto quartz surface and pectin-protein interaction in real time.

Our results show that DM and/or amidation alter the charge of pectin, significantly affecting protein adsorption behavior. QCM-D analysis reveals distinct interaction profiles depending on pectin charge, suggesting that electrostatic interactions play a major role in complex formation.

These findings provide new insights into the role of pectin charge in modulating biomolecular interactions and contribute to a better understanding of cell wall organization and its potential implications in plant growth.

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KEYWORDS

pectins, methylation degree, surface charge, protein interaction, QCM-D

ENGINEERING TRANSPARENT CELLULOSE NANOFIBRIL FILMS FOR RETINAL PROGENITOR CELLS CULTURE

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ABSTRACT

The in vitro expansion of human retinal progenitor cells (hRPCs) derived from induced pluripotent stem cells is commonly supported by Matrigel®, a murine tumour-derived matrix. Matrigel® presents limitations such as batch-to-batch variability, undefined composition, high cost and animal-derived components, restricting its suitability for translational biomedical applications[1]. Therefore, improved sustainable basement membrane matrixes are needed.

Cellulose nanofibrils (CNFs) are renewable polysaccharide nanomaterials with high water retention, nanoscale fibrillar architecture, optical transparency and tunable surface chemistry[2]. TEMPO-mediated oxidation introduces carboxyl groups, promoting nanofibrillation and providing reactive sites for biofunctionalization with cell-adhesive molecules. The aim of this work is to demonstrate that transparent biofunctional CNF hydrogels are good polysaccharide-based alternatives to Matrigel® for hRPC culture.

CNFs were obtained from bleached kraft eucalyptus pulp through TEMPO-mediated oxidation followed by high-pressure homogenization at 600 bar. Different oxidation conditions were explored by varying oxidant load and reaction time, and the resulting materials were assessed in terms of appearance, carboxyl group content, nanoscale morphology and optical transmittance. The selected CNF formulation showed a carboxyl group content of 2.01 ± 0.10 mmol/g. Nanofibril morphology and diameter distribution were analysed by transmission electron microscopy. CNF hydrogels were prepared at 0.2% and functionalized with gelatin or RGD peptide to introduce biological adhesion motifs while preserving transparency. Surface modification was evaluated by FTIR-ATR. For cell culture assays, hRPCs were seeded onto sterile hydrogels in non-treated plates at 40,000 cells/well and cultured in N2B27 medium at 37 °C and 5% CO_2, using Matrigel® as control. Cell adhesion, morphology and expansion were monitored by microscopy.

TEMPO-oxidized CNFs formed homogeneous and transparent hydrogel networks, with transmittance values above 97% at 600 nm and nanofibril diameters between 5 and 20 nm (Figure 1a). FTIR-ATR confirmed successful functionalization through amide-associated bands at 1560 cm^{-1} (Figure 1b). After medium preconditioning, the hydrogels reached physiological pH. hRPCs adhered from the first day, maintained appropriate morphology and expanded up to approximately 80% confluence by day 7. Among the formulations tested, gelatin-functionalized CNF supported cell growth comparable to Matrigel®.

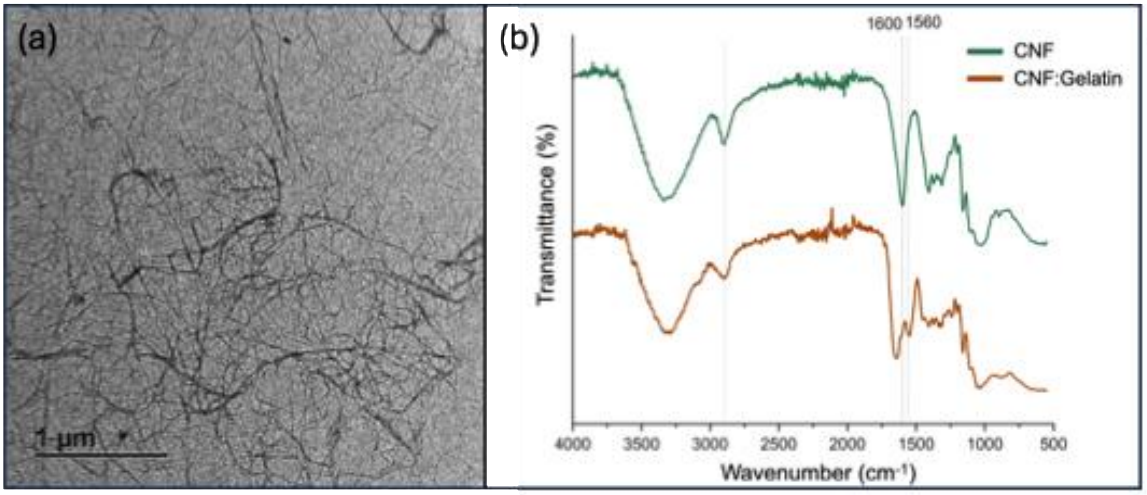

Figure 1. Characterization of CNF hydrogels. **(a)** TEM image of TEMPO-oxidized CNFs at 3000× magnification. **(b)** FTIR-ATR spectra of CNF before and after gelatin immobilization.

These results demonstrate that TEMPO-oxidized CNF hydrogels are transparent, biocompatible and biofunctional polysaccharide materials that can be used for cell culture applications. This platform combines renewable cellulose chemistry, optical compatibility and tunable bioadhesion, offering a sustainable route towards more defined matrices for retinal progenitor cell expansion.

KEYWORDS

Cell-culture matrix, functionalized cellulose nanofibrils, Polysaccharide hydrogels, Tissue engineering, Retinal progenitor cells

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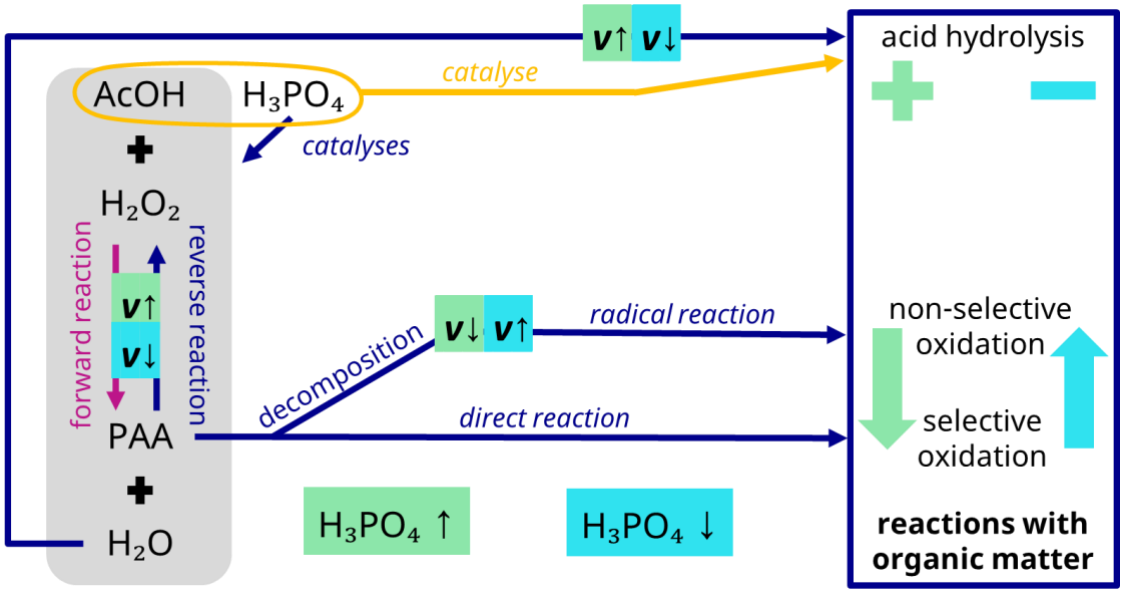
UNVEILING THE 'BLACK BOX': KINETIC INSIGHTS INTO CARBOHYDRATE DEGRADATION DURING *IN SITU* PERACETIC ACID PULPING

Max Emanuel Winkler

ABSTRACT

Pulping with peracetic acid (PAA) facilitates highly selective carbohydrate recovery. In recent years, biomass processing via *in situ* generated PAA has increasingly been under investigation. However, fully exploiting these systems requires a fundamental understanding of the interplay between internal reaction kinetics and the resulting pulp properties. Whilst the equilibrium kinetics of the H_2O_2/PAA system are well-characterised, the dynamic evolution of the liquor composition during the digestion itself has largely remained a 'black box'.

This study investigates the impact of the initial composition of the pulping liquor on the evolution of the peroxide fraction during the pulping of soybean straw. The initial objective was to identify reaction groups relevant to the properties of the pulp's carbohydrate fraction by investigating the kinetic relationships within the actual pulping system. Subsequently, the process-property relationships between the initial liquor composition, the rates of these relevant reaction groups, and the resulting pulp properties were examined. To this end, 18 digestions were performed using an aqueous system of acetic acid, hydrogen peroxide, and phosphoric acid. The reaction progress was monitored via continuous titration of the peroxide species. The resulting carbohydrate properties were characterised through alkali resistance (R_{18}), viscometric degree of polymerisation, carbohydrate composition (HPLC), and molecular weight distribution (GPC) of the pulps. Furthermore, distinct molecular weight distributions for the hexose and pentose fractions were calculated.



The results demonstrate that three primary reaction groups exert a direct influence on the pulp's carbohydrate fraction: acid hydrolysis, and two distinct groups of peracetic acid turnover—direct reactions with the organic matter and radical reactions resulting from PAA decomposition. Whilst the direct reaction of PAA with the organic matter is highly selective in terms of delignification and degrades the holocellulose primarily from the end-groups, both radical reactions and acid hydrolysis induce random chain scission within the accessible regions of the carbohydrate polymers.

Phosphoric acid serves as a pivotal kinetic lever within this system. It accelerates PAA formation whilst simultaneously inhibiting its decomposition, thereby shifting the irreversible PAA turnover towards the carbohydrate-preserving pathway. Consequently, the PAA consumption proceeds more rapidly yet remains more selective. Simultaneously, however, the higher mineral acid content catalyses the acid hydrolysis. In terms of carbohydrate degradation, the phosphoric acid concentration thus exerts two opposing effects. This mechanistic insight allows for the tailoring of pulp properties according to specific objectives, such as maximum holocellulose yield, targeted pentose fraction compositions, or desired molecular weight distributions of the cellulose.

KEYWORDS

peracetic acid pulping, process-property relationships, carbohydrate degradation, non-wood lignocellulosics

DEVELOPMENT OF SUSTAINABLE CORK AGGLOMERATES USING MARINE POLYSACCHARIDES

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ABSTRACT

Cork is a distinctive, renewable material harvested from the bark of the cork oak (*Quercus suber L.*), a slow-growing evergreen tree native to the western Mediterranean [1]. This unique material is mostly well known for its use in the winery industry as a bottle stopper. However, its use has been explored across a wider range of applications, for example, in the building, automotive, and aerospace industries, as well as on the design and apparel sectors, taking advantage of the distinctive properties of this natural material, such as excellent elasticity, low permeability to liquids and gases, acoustic and thermal insulation, and low density [2].

The goal of this study is to produce sustainable cork agglomerates using more eco-friendly binder alternatives. For this, the use of marine polysaccharides is investigated as a direct replacement of the conventional PU resin (up to 30%) or for the design of a fully biobased binder. This leads to an increase in the total biobased content of the cork-based materials to up to 86% (for the partial replacement of the PU binder) and 100% (for the fully biobased solution). For all formulations, the cork agglomerates remained cohesive and uniform. Structural, morphological, and spectroscopic analyses confirmed successful curing of both resins and the maintenance of the cork's natural cellular structure. Thermal and mechanical analyses indicated that all cork agglomerates produced following both approaches maintained adequate thermal stability and mechanical performance. In addition, all materials withstood 3 h of immersion in boiling water, satisfying a key requirement for this type of application. Additionally, the incorporation of marine polysaccharides may also introduce new functional properties to these thermoset materials, expanding potential applications while valorizing abundant, renewable feedstocks derived from waste.

Acknowledgments: This study was funded by the PRR – Plano de Recuperação e Resiliência and by the NextGenerationEU funds at Universidade de Aveiro, through the scope of the Agenda for Business Innovation "Pacto da Bioeconomia Azul" (Project no. 16 with the application no. C644915664-00000026) and the project CICECO–Aveiro Institute of Materials, UID/50011/2025 (DOI 10.54499/UID/50011/2025) & LA/P/0006/2020 (DOI 10.54499/LA/P/0006/2020), financed by national funds through the FCT/MCTES (PIDDAC). FCT is also acknowledged for the research contract under the Scientific Employment Stimulus to C.S.R.F. (CEECIND/00464/2017).

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KEYWORDS

Cork, Marine polysaccharides, Molding, Sustainability, Thermosets

SELECTIVE DISASSEMBLY OF BREWER'S SPENT GRAIN FOR THE RECOVERY OF POLYMERIC XYLAN USING MILD CHEMICAL AND ENZYMATIC TREATMENTS

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ABSTRACT

The growing accumulation of petroleum-derived plastic waste has intensified the search for renewable, bio-based polymers derived from lignocellulosic biomass. Among these, xylans are the most abundant hemicelluloses in nature and represent a highly attractive, yet underutilized, class of biopolymers with potential applications ranging from functional food ingredients to sustainable materials. Agro-industrial residues constitute particularly promising feedstocks for xylan recovery from both a circular economy and sustainability perspective.

Brewers' spent grain (BSG), which accounts for approximately 85% of the solid by-products generated during beer production, is rich in hemicelluloses and therefore represents an appealing, renewable source of xylan. However, current valorization strategies for BSG are predominantly focused on saccharification or oligosaccharide production, often at the expense of polymer integrity. While enzyme-assisted treatments are widely applied to enhance biomass hydrolysis, their potential for selectively disrupting the plant cell wall to enable the recovery of polymeric hemicelluloses has remained largely unexplored, particularly in secondary agro-industrial residues such as brewer's spent grain.

In this context, the present work aims to optimize the extraction of polymeric xylan from BSG by combining mild acidic or alkaline pre-treatments with the targeted use of accessory enzymes. In a first step, the effect of different chemical pre-treatments on hemicellulose solubilization was assessed, with special emphasis on maximizing xylan yield while preserving molecular weight and the native arabinose/xylose (A/X) ratio. Subsequently, the synergistic action of cellulases, lytic polysaccharide monooxygenases (LPMOs), proteases and laccase was explored as a strategy to selectively loosen the lignocellulosic matrix without directly cleaving the xylan backbone.

The extracted fractions were characterized by high-performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD) and high-performance size-exclusion chromatography (HPSEC) to determine xylan yield, A/X ratio and molecular weight distribution. The results demonstrate that the combined application of mild chemical treatments and carefully selected accessory enzymes significantly enhances xylan recovery from BSG, while enabling a controlled balance between extraction yield and preservation of polymeric xylan structure. This approach provides a promising basis for the tailored isolation of xylan from brewery residues for high-value applications.

KEYWORDS

Xylans; brewer's spent grain; lignocellulosic biomass; enzyme-assisted extraction; polymeric hemicelluloses

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SELECTING PECTIN: A DECISION GUIDE FROM MOLECULAR STRUCTURE TO END-USE PERFORMANCE

Chloé Larrue, Gino Mangiante

ABSTRACT

Pectin (E440i) is a food additive industrially extracted from citrus peels and apple pomace[1]. It is widely used as texturizer to change the physico-chemical properties of food matrices. The term “pectin” encompasses a broad range of materials with distinct structural characteristics and functional properties. From a commercial point of view, the pectin is segmented into 3 main families according to their degree of esterification: high-methoxyl pectins (HM), low-methoxyl pectins (LM), and amidated low-methoxyl pectins (LMA, E440ii).

Pectin intrinsic parameters (degree of methoxylation, amidation, molar mass, etc.)[2], formulation parameters (pH, sugar content, etc.), and processing conditions affect gel strength. Accordingly, careful selection of pectin is essential and should be guided by the intended texture and specific end-use requirements (reduced-sugar jam, fruit preparations, dairy desserts, etc.). This choice will be driven by the gelation mechanisms, ranging from sugar-acid gelation for HM pectins to calcium-mediated gelation for LM and LMA pectins[3].

This presentation will provide the audience with the necessary tools to select the appropriate pectin through a synthetic view of the specificities of HM, LM and LMA pectins and the link between molecular structure, functionality, and end-product performance.

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KEYWORDS

Pectin, Gelation mechanisms, Degree of methoxylation, Food formulation

LARGE SCALE SOLVENT-FREE PRODUCTION OF CELLULOSE NANOCRYSTALS USING MECHANOCHEMISTRY AND AGING

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ABSTRACT

Cellulose nanocrystals (CNCs) are high aspect ratio nanoparticles extracted through hydrolysis of native cellulose sources through high-temperature acid or oxidation-based conditions. Yet, these solution-based approaches result in high amounts of acid waste and water consumption during the synthesis process, which is detrimental for the production and utilization of CNCs for advanced downstream applications. To solve this, ongoing efforts have been realized by a variety of groups to study solvent-free conditions to isolate CNCs through mechanochemistry, yet much of the literature often shows very low scale (e.g., 0.5 g and 1.5 g) or low CNC purity. Here, we demonstrate that by exploiting High-Humidity Shaker Aging (HHSA), we can fabricate CNCs at 100 g-scale. Moreover, we demonstrated that the obtained CNCs have comparable quality to solution-based methods, both in terms of morphological and structural features, but also in terms of liquid crystalline properties. Finally, life-cycle assessments validate the efficacy of the process in comparison to solution-based pathways.

KEYWORDS

cellulose nanocrystals, oxidation, solvent-free, large scale, self-assembly

OPTIMIZING BIODEGRADABLE FILAMENT FORMULATIONS FOR ENHANCED EFFICIENCY FERTILIZERS: FROM MICROPARTICLES TO EXTRUSION

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ABSTRACT

Sustainable agriculture demands innovative strategies to deliver essential nutrients while minimizing environmental impacts. Enhanced efficiency fertilizers (EEFs) have emerged as a promising alternative by enabling slow nutrient release and reducing losses associated with conventional fertilizers. In this context, lignocellulosic materials (LCMs), such as lignin and cellulose, combined with biodegradable polymer matrices, offer a sustainable platform for developing advanced fertilizer systems. This study aims to optimize the production of filaments based on LCMs containing monoammonium phosphate (MAP), potassium nitrate (KNO_3), and a starch (St)/poly(vinyl alcohol) (PVA) matrix. Initially, LCMs were combined with MAP by spray-drying to produce nutrient-loaded microparticles. These microparticles were incorporated into a starch St/PVA matrix via melt mixing to ensure uniform dispersion and subsequently extruded to form filaments. The base formulations, previously selected based on biodegradability and nutrient release performance, were modified by adding KNO_3 during melt mixing to obtain a material containing the three primary macronutrients required for plant growth (NPK). The filament optimization focused on microparticle-to-nutrient ratio, presence or absence of cellulose, and plasticizer content in the PVA matrix. The filaments were characterized in terms of morphological, physical, thermal, and mechanical properties, aiming at their use as EEFs (after pelletization) or as feedstock for additive manufacturing. Preliminary results indicate that a total nutrient content of 10 wt.% yields homogeneous filaments resistant to thermomechanical stresses during extrusion. The presence of cellulose improved filament diameter uniformity, while pre-processing enhanced morphology. Nutrient incorporation and higher plasticizer content reduced both elastic modulus and elongation at break. These findings highlight the potential of optimized formulation and processing to produce biodegradable NPK filaments for sustainable EEFs in agriculture.

KEYWORDS

Enhanced efficiency fertilizers, biopolymers, NPK filaments, extrusion processing, additive manufacturing

BIODEGRADABLE MATRICES BASED ON PVA/TPS FOR ENHANCED EFFICIENCY FERTILIZERS

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ABSTRACT

The use of conventional fertilizers has led to significant environmental impacts, including nutrient losses through leaching, volatilization, and runoff. Enhanced-efficiency fertilizers (EEFs) have emerged as a promising alternative to mitigate these issues because they can control nutrient availability to crops, thereby increasing nutrient use efficiency. However, currently marketed EEFs are composed of non-biodegradable coatings that accumulate in the soil. In this context, the development of biodegradable matrices capable of controlling nutrient release is essential for sustainable agricultural practices. This study aimed to develop and evaluate a biodegradable system based on a 50/50 (w/w) poly(vinyl alcohol) (PVA)/thermoplastic starch (TPS) blend, incorporating potassium nitrate (KNO_3) as source of macronutrients. The materials prepared via thermomechanical processing in an internal mixing chamber were subjected to a biodegradation test in soil, monitored by the Bartha-Pramer respirometry technique over 100 days. Additionally, materials were characterized in terms of their structural (FTIR), morphological (SEM), and thermal (DSC and TGA) properties before and after the biodegradation test. Nutrient release was investigated in aqueous medium by conductivity. The results indicated that the incorporation of KNO_3 influenced the matrix organization and, consequently, water absorption, thereby affecting both nutrient release and biodegradation behavior. EEF exhibited a prolonged release profile compared to pure KNO_3, confirming the polymeric matrix's role as a diffusion barrier. Biodegradation analysis demonstrated that mineralization occurs in fractions, preferentially by the phase attributed to TPS, which appears to favor PVA mineralization, and that macronutrients prolong microbial respiration. These findings highlight the potential of PVA/TPS-based systems containing KNO_3 as biodegradable supports for more efficient applications, contributing to the development of environmentally friendly practices.

KEYWORDS

starch; biodegradation; controlled release.

UNLOCKING THE POTENTIAL OF MISCANTHUS X GIGANTEUS: A COMPARATIVE BENCHMARK OF SUSTAINABLE PULPING TECHNOLOGIES FOR NEXT-GENERATION MAN-MADE CELLULOSE FIBRES

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ABSTRACT

The global pulp market is a highly dynamic sector, with a production capacity of approximately 200 million tons (2024) and a projected value of 182 billion USD by 2025, driven by an expected compound annual growth rate (CAGR) of 3.74%. Although wood currently accounts for 95% of the primary raw material supply, the projected 5–6% CAGR for the non-wood sector highlights a significant shift toward alternative fibre resources. This development is primarily driven by rising demand in emerging economies across Asia, Africa, and Latin America, where limited industrial availability of native wood necessitates a strategic diversification of the feedstock base. India serves as a prime example, meeting nearly 50% of its pulp requirements through non-wood plants.

The market dynamics correlates with the growing global demand for Man-Made Cellulose Fibres (MMCF) as ecological alternatives to petroleum-based synthetics and resource-intensive cotton. As the Lyocell process has established itself as a leading sustainable technology, the industry increasingly relies on cellulose sources beyond traditional forestry. In the textile hubs of India, Pakistan, and Bangladesh, tapping into such alternatives is essential to meet regional demand and reduce import dependencies. In this context, *Miscanthus x giganteus* has emerged as a promising source. As a fast-growing, low-input crop, Miscanthus provides high biomass yields with a minimal ecological footprint and a favourable cellulose-to-lignin ratio for dissolving pulp production.

The suitability of *Miscanthus x giganteus* was evaluated by producing dissolving pulp and processing it into Lyocell fibres. To assess the potential of this non-wood plant, a systematic comparison was conducted between established wood pulping and alternative methods tailored to non-wood resources. Conventional processes—namely Kraft, Sulphite (wood-based references), and Soda (non-wood standard)—were compared against three eco-friendly alternatives: the oxidative NBP process (Acetosolv), Natural pulping and an ionic liquid-based method. While conventional routes serve as benchmarks, this study focuses on how effectively alternative processes leverage the specific morphology and lower lignin content of Miscanthus. Evaluation was based on technical effort, yields, and resulting fibre properties. The findings highlight the advantages of sulphur-free concepts and

demonstrate how Organosolv and ionic liquid processes enable efficient, high-quality dissolving pulp production from agricultural biomass.

KEYWORDS

Man-made cellulose fibres, Dissolving pulp, Ionic liquids, Miscanthus, Organosolv

PROGRAMMABLE INTERFACIAL ASSEMBLY OF CELLULOSE NANOCOMPOSITES FOR STABILIZING MULTIPHASE SYSTEMS

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ABSTRACT

Cellulose nanocrystals (CNCs), as charged rod-like nanoparticles, have attracted significant attention as eco-friendly stabilizers for Pickering emulsions. However, their inherent hydrophilicity often requires surface modification to achieve optimal interfacial performance. In this study, we propose a biomimetic strategy to fabricate programmable cellulose nanocomposites through the integration of CNCs with phase-transitioned bovine serum albumin (PTB). Specifically, reduction of disulfide bonds in bovine serum albumin induces amyloid-like aggregation, enabling rapid self-assembly of PTB oligomers onto CNC surfaces. The resulting PTB/CNC nanocomposites synergistically combine the enhanced interfacial activity of the hybrid particles with bioadhesive properties arising from protein structural reorganization. The hybrid nanocomposites exhibit excellent interfacial stabilization capabilities and spontaneously adsorb at fluid interfaces, enabling effective stabilization of both oil-in-water and water-in-water emulsions (Fig. 1). Moreover, the interfacial behavior and stabilization performance can be precisely tuned by adjusting the PTB-to-CNC ratio, allowing programmable control of the nanocomposite architecture and emulsion properties. These nanocomposite-stabilized emulsions demonstrate strong potential for sustainable agricultural applications, such as precision pesticide delivery. More broadly, the programmable interfacial assembly strategy provides a versatile platform for developing functional soft materials with controlled droplet deposition and surface adhesion, with potential applications in functional coatings, cosmetic formulations, and targeted drug delivery systems.

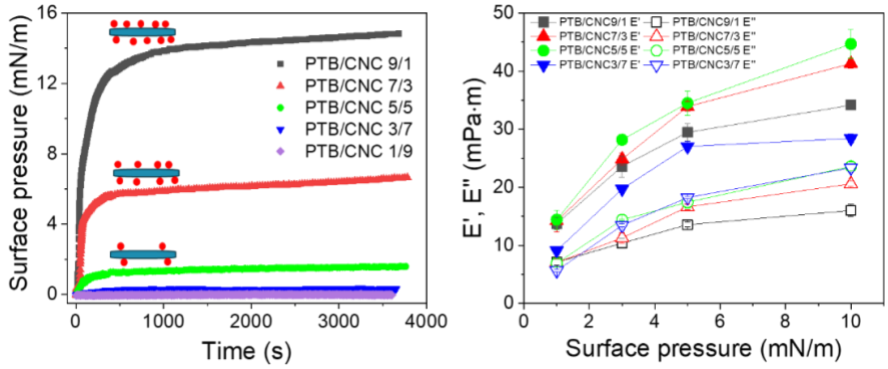

Figure 1. Behaviour of the PTB/CNC nanocomposites at air-water interface

KEYWORDS

CNC, Interfacial Assembly, Multiphase Systems

ANIONIC CELLULOSE DERIVATIVES AS SUSTAINABLE SEQUESTERING AGENTS

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ABSTRACT

Industrial water treatment relies heavily on synthetic additives such as phosphonates and petroleum derived polymers as sequestering agents to control scaling and prevent mineral deposition. While these compounds are highly effective, they are often associated with limited biodegradability, persistence in aquatic environments and concerns regarding their long-term sustainability and regulatory acceptance.

In this context, anionic cellulose derivatives have been proposed as promising bio-based and eco-friendly alternatives. They offer inherent biodegradability, low toxicity, and structural tunability which renders them attractive as candidates for sustainable sequestering agents.

In the current study, various synthetic strategies were exploited to prepare diverse anionic cellulose derivatives. Cellulose ethers were obtained by direct etherification of cellulose under homogeneous or heterogeneous reaction conditions. Additionally, cellulose was pre-oxidized to dialdehyde cellulose and further derivatized by reductive amination.

The obtained materials were characterized using solid and liquid state NMR, FTIR and UV/Vis spectroscopy, as well as elemental analysis to confirm successful functionalization and to assess their structural features.

Subsequently, the inhibitory effect of the synthesized anionic cellulose derivatives against calcium carbonate scale formation was evaluated according to standardized turbidity-based precipitation assays. The results demonstrate that the obtained cellulose-based sequestering agents exhibit significant inhibition of crystal nucleation and growth, effectively reducing scale formation under tested conditions.

Overall, the findings highlight the strong potential of anionic cellulose derivatives as sustainable and biodegradable alternatives to conventional scale inhibitors, combining high performance with improved environmental compatibility.

KEYWORDS

Anionic cellulose derivatives, dialdehyde cellulose, cellulose ethers, industrial water treatment

AMINO-FUNCTIONALIZED DEXTRAN PALMITATE FILMS FOR THERMAL NANOIMPRINT LITHOGRAPHY AND TOPOGRAPHY-GUIDED CELL ORGANIZATION

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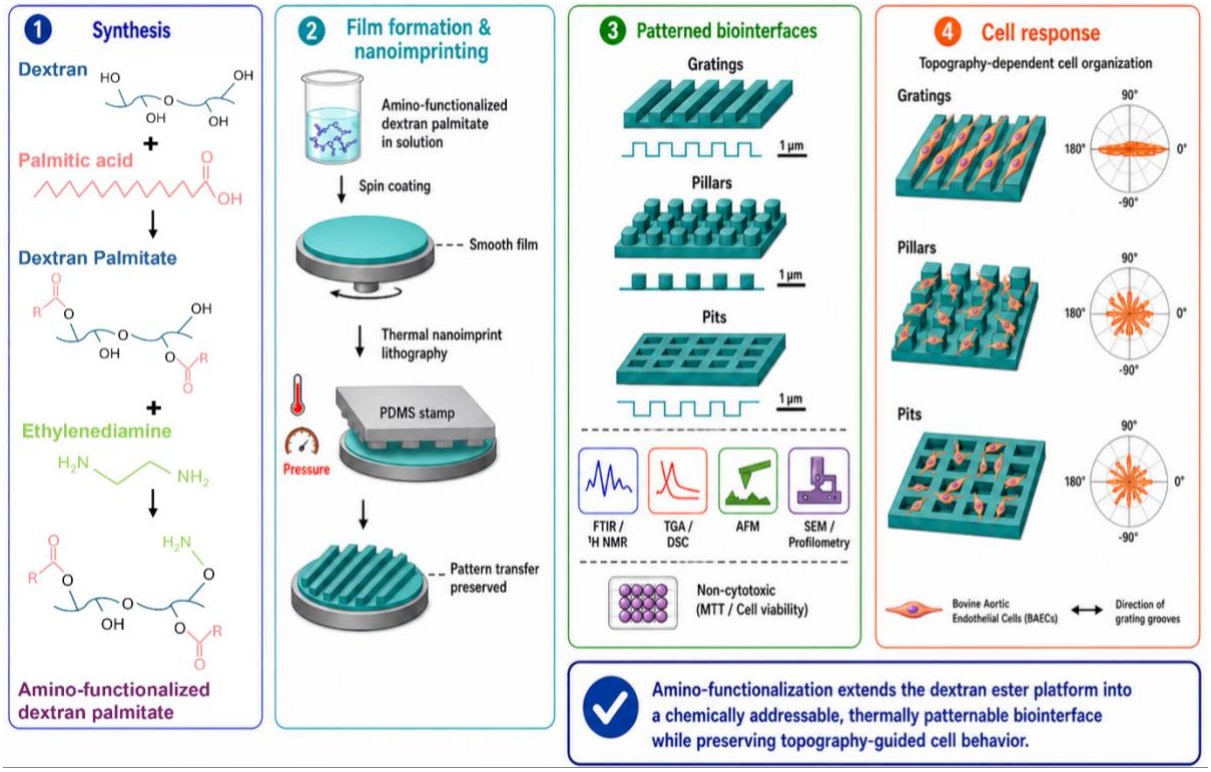
ABSTRACT



Objectives: This study aimed to develop amino-functionalized dextran palmitate films as chemically addressable and thermally patternable biointerfaces for investigating topography-guided cell organization. The objective was to introduce amino functionality into a dextran ester nanoimprint platform while preserving film-forming ability, thermal processability, pattern transfer, and cellular response to surface topography.

Methods: Dextran was first esterified with palmitic acid and subsequently aminated through a CDI-mediated reaction using ethylenediamine. The resulting derivatives were characterized by FTIR, ^{1}H

NMR, elemental analysis, TGA, and DSC. Thin films were prepared by spin coating and evaluated for wettability and nanoscale roughness using water contact angle measurements and AFM. Representative grating, pillar, and pit topographies were fabricated by thermal nanoimprint lithography and characterized by SEM and 3D optical profilometry. Indirect cytotoxicity was assessed using an extract-based MTT assay with NIH3T3 cells, while cell morphology, alignment, and elongation on patterned films were evaluated using Bovine Aortic Endothelial Cells (BAECs) by optical microscopy and image analysis.

Key Results: Spectroscopic and elemental analyses confirmed successful esterification and amino-functionalization of dextran palmitate while retaining the modified polysaccharide framework. The spin-coated films showed composition-dependent wettability and low nanoscale roughness suitable for patterning. Grating, pillar, and pit structures were successfully transferred onto the films, demonstrating that amino-functionalization did not compromise thermal nanoimprint processability. Extract-based MTT assays indicated that the films were non-cytotoxic under the tested conditions. Among the patterned surfaces, grating topographies induced the strongest BAEC alignment and elongation, whereas pillar and pit structures showed weaker directional guidance.

Conclusion: Amino-functionalized dextran palmitate films were successfully developed as thermally patternable, non-cytotoxic biointerfaces. The platform preserves topography-dependent regulation of cell morphology while introducing additional chemical functionality, supporting its potential use as a tunable model system for studying combined chemical and physical cues at biomaterial interfaces.

KEYWORDS

Dextran palmitate, Amino-functionalization, Patterned biointerface, Thermal nanoimprint lithography, Cell alignment

SURFACE DISPLAY SYSTEM IN A BIOMATERIAL-PRODUCING MICROBE FOR PROGRAMMABLE PROTEIN ASSEMBLY

Shuang Zhang[1], Ruxia Fan[2], Sesilja Aranko[*], Rahul Mangayil[*]

ABSTRACT

Bacterial cellulose (BC) produced by *Komagataeibacter* is an attractive matrix for composite materials, yet controlled, in situ integration of functional proteins into the growing network remains difficult. A central limitation is the lack of modular tools that allow stepwise assembly of biomaterials under process-relevant conditions.

Here, we develop a modular surface-display platform in *Komagataeibacter* to enable situ protein assembly during BC formation. An orthogonal SpyTag–SpyCatcher pair was first used as a validation module, using externally added SpyCatcher–mScarlet protein that binds specifically to the displayed SpyTag, providing a background-free fluorescence readout. The platform was then extended to a two-component assembly scheme using a SilkTag–SilkCatcher interface. Fusion proteins assemble in solution via SpyTag–SpyCatcher interaction and are subsequently immobilized on the cell surface through SilkTag–SilkCatcher binding. This enables in situ retention of functional proteins within the forming BC matrix.

Fluorescence imaging shows that recruitment occurs only when all components are present, indicating specific and orthogonal interactions. To assess process compatibility for in situ composite formation, BC growth was evaluated in the presence of lysis-like buffers that mimic protein- or lysate-containing environments. Pellicle formation was maintained in a ~0–20% lysis buffer, indicating partial tolerance to the tested conditions.

This approach enables in situ incorporation of functional proteins into BC during growth, linking cell-surface programming with material assembly and providing a route toward engineered cellulose-based composites.

KEYWORDS

Bacterial cellulose; in situ assembly; protein incorporation; composite materials

RICE STARCH-BASED ADHESIVES FOR WET-STRENGTH PAPER APPLICATIONS

Urška Klenovšek, Urška Vrabič Brodnjak

ABSTRACT

Bio-based adhesives are gaining increasing attention as sustainable alternatives to synthetic petroleum-based systems in packaging applications. In this study, rice starch-based adhesives were developed and evaluated for bonding wet-strength packaging papers intended for glass packaging materials such as jars and bottles. Four adhesive formulations were prepared: L1 (rice starch, alginate, chitosan, and CO_3 for pH regulation), L2 (rice starch and alginate), L3 (rice starch, chitosan, and CO_3), and L4 (rice starch only).

Three wet-strength papers with different grammages were investigated: V1 (65 g/m^2), V2 (80 g/m^2), and V3 (75 g/m^2). Paper samples were cut in machine direction (MD) and cross direction (CD) to evaluate the influence of fiber orientation on adhesive performance. Mechanical characterization was performed using T-tests and S-tests on bonded paper specimens. Additionally, the durability of bonded labels was evaluated after washing in a commercial washing machine at 55 °C for 1 h using washing tablets.

The results demonstrated that both adhesive composition and paper orientation significantly influenced bonding behavior. In T-tests, L1 exhibited the highest overall tensile/peel strength, while formulations containing alginate or chitosan showed increased elongation and flexibility compared to the starch-only adhesive. S-tests revealed generally higher shear resistance than peel resistance for all formulations, indicating good suitability for packaging applications exposed to mechanical loading. Differences between MD and CD directions were consistently observed, confirming the strong influence of fiber orientation on mechanical performance. Among the tested systems, formulations containing biopolymer additives (alginate and/or chitosan) showed improved bonding characteristics compared to pure rice starch adhesive. The washing stability test further demonstrated the potential applicability of the developed adhesives under realistic packaging use conditions.

Overall, the study demonstrates the potential of rice starch-based bio-adhesives as environmentally friendly alternatives for wet-strength paper packaging applications. The findings further highlight the importance of paper fiber orientation and adhesive formulation in optimizing bonding performance for sustainable packaging systems.

KEYWORDS

Rice starch adhesive, bio-based adhesives, wet-strength paper, sustainable packaging

STRENGTHENING THE INTERPHASE IN SELF-REINFORCED POLY(LACTIC ACID) COMPOSITES VIA LOCAL DEPOSITION OF CELLULOSE NANOCRYSTALS

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ABSTRACT

Self-reinforced composites (SRCs) are polymer composites in which both the matrix and reinforcement are made from the same polymer but with different morphologies, allowing the production of monomaterial structures that simplify recycling at end of life. In these systems, pre-aligned high-strength poly(lactic acid) (PLA) fibers are sandwiched in PLA matrix and subsequently consolidated by hot pressing under controlled temperature and time conditions. Although this approach offers clear sustainability advantages, polymer chain interdiffusion at the fiber-matrix interface is inherently slow [1-3], which often results in long processing times and limited interfacial cohesion.

To tackle these limitations, this study proposed a localized interfacial modification strategy based on the deposition of unmodified cellulose nanocrystals (CNCs) directly onto the highly crystalline PLA fibres surface prior to consolidation. Because the nanoparticles are introduced only at the fiber surface, low CNC loadings should be sufficient, largely preserving the monomaterial character of the composite while potentially enhancing mechanical performance [4]. Processing parameters and CNC contents are systematically varied to establish structure-property relationships and to evaluate their influence on fiber-matrix interface behaviour, providing insight into how nanoscale interfacial engineering can overcome diffusion-limited bonding in self-reinforced composites [5-6]. The initial transverse tensile stress value of the materials exhibited differently when CNCs were included at the fiber-matrix interface. This strategy offers a promising route towards stronger, more efficient, and fully recyclable PLA-based composites with reduced processing time and improved material cohesion.

KEYWORDS

Cellulose nanocrystals, interface, self-reinforced PLA

PROGRAMMABLE INTERFACIAL ASSEMBLY OF CELLULOSE NANOCOMPOSITES FOR STABILIZING MULTIPHASE SYSTEMS

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ABSTRACT

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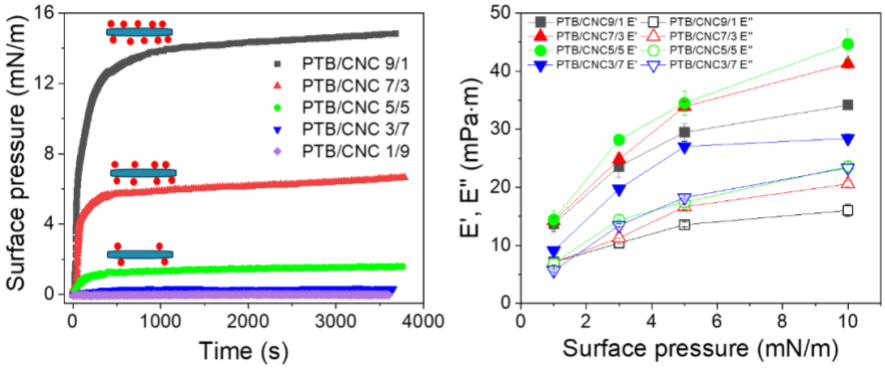

Figure 1. Behaviour of the PTB/CNC nanocomposites at air-water interface

KEYWORDS

CNC, Interfacial Assembly, Multiphase Systems

ANIONIC CELLULOSE DERIVATIVES AS SUSTAINABLE SEQUESTERING AGENTS

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[2] Zschimmer & Schwarz Mohsdorf GmbH & Co.KG, Burgstädt, Germany

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