N°54 - JUNE 2020



European Polysaccharide Network Of Excellence



## *"Nature makes polysaccharides, EPNOE turns them into products"*

## **Editorial**

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The Covid-19 pandemics took our lives to unprecedent experiences of being restricted at home and numerous online meetings and lectures. Suddenly we became online professors, scientists, engineers, family and friends. One of the positive sides of this situation is the realization that we are in fact social animals. We realize even more now, the joy of sharing spaces, sensations and experiences with someone else. We realize how rich our lives are and we long to have it back hopefully as we had it before.

We have been very active in EPNOE in the latest four months. In February a successful workshop about Modern analytical approaches in biopolymer characterization was organized at BOKU in Austria and since March we are working on preparations of EPNOE2021 conference to be held in Nantes, France next year. We have an excellent scientific committee with top level polysaccharide scientists of all over the world and we are inviting EPNOE members to suggest thematic sessions. EPNOE2021 will be co-organized with Cellulose Society of Japan and American Chemical Society. The restrictions applied to Covid-19 forced us to change the dates of two important EPNOE events this year. The EPNOE Junior Scientist Meeting to be held in Kortrijk, Belgium was changed from September to February next year and the EPNOE Connect workshop at XXV Tecnicelpa/ XI CIADICYP to be held in Coimbra, Portugal was changed from October to March next year. This challenge also brought opportunities. We are happy to announce that an EPNOE Junior Online Seminar will be organized on 15 September 2020 which can be followed freely and remotely. We are also currently working to continue the publication of EPNOE SpringerBrief Series and in building our new communication team. We are looking for volunteers interested in helping with edition of books and young researchers interested in communication and social media.

EPNOE Association offers numerous opportunities for networking, research and education and EPNOE members are working to create meaningful change. We would like to invite you to join us.



Pedro Fardim President of EPNOE Professor Faculty of Engineering Science Department of Chemical Engineering KU Leuven (Belgium)



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### **EPNOE** Members' News

#### FSU Jena

• Fanny Getzlaff joined the group as Bachelor student working in the field of agarose derivatives supervised by Dr. Martin Gericke and Prof. Thomas Heinze

• Gesa Pelloth joined the group as Bachelor student working in the field of pectin derivatives supervised by Dr. Hendryk Würfel and Prof. Thomas Heinze

#### <u>Łukasiewicz Research Network - Institute of Biopolymers and Chemical</u> <u>Fibers</u>

The Łukasiewicz Research Network - Institute of Biopolymers and Chemical Fibers has taken up the challenge of fighting the pandemic caused by the spreading of coronavirus COVID-19 worldwide. Together with the Łukasiewicz Research Network - the Tele- and Radio Research Institute (Warsaw) we started work on the construction of a technological line for the production of masks. It will be enable to produce FFP2 type masks with parameters that allow their use in hospitals, i.e. where they are most needed now. We will use experience in the production of nonwovens and filter materials as well as ultrasonic welding technology. Production will be carried out for the needs of the indicated medical units and will allow to produce about 2 million masks per month.

At ŁUKASIEWICZ - Institute of Biopolymers and Chemical Fibers, the work will be carried out by scientists from the Team of Keratin Composites: Krystyna Wrześniewska-Tosik; Ewa Wesołowska; Michalina Pałczyńska; Damian Walisiak, and scientists from the Experimental & Production Department of Composite Products:Tomasz Mik; Tomasz Kowalewski and Dariusz Dębiec.

#### Research Institute of Textile Chemistry/textile Physics, University of Innsbruck

The Institute is part of a consortium granted funding from the Austrian Research Promotion Agency (FFG) for a three-year project on developing a biotechnological enzyme-based modification process for natural lignocellulose stem fibres. https://projekte.ffg.at/projekt/3696174

#### **IMT Mines Ales**

Léo Bontempi started his internship at IMT Mines Alès in partnership with LIFCO industry on "3D printing of conductive natural fibre reinforced biocomposites" supervised by N. Le Moigne, A. Regazzi, S. Corn and M. Batistella.



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#### Announcements

#### EPNOE Junior Scientist Meeting in Kortrijk, Belgium postponed from 15-16 September 2020 to 3rd-4th February 2021

Due to the current circumstances, EPNOE and the local organizing committee of the EPNOE Junior Scientist Meeting have decided to postpone the International EPNOE Junior Scientist Meeting in Kortrijk, Belgium from 15-16 September 2020 to 3rd-4th February 2021. Therefore, the abstract submission deadline and registration will be postponed as well. More information on the new dates can be found on the conference website: https://www.epnoejunior2020.org/.

As the International EPNOE Junior Scientist Meeting planned for this year will be postponed to next year, we will organize an EPNOE Junior Online Seminar on 15 September 2020, which can be followed freely and remotely. More information will be available as soon as possible at https://www.epnoejunior2020.org/onlineseminar as we are still sorting out program, speakers, and practical arrangements.

Hopefully, we will see many of you online in September and in person in 2021!

EPNOE is creating an Award to recognise outstanding achievements of young scientists working with polysaccharides. The award is open to all young scientists in the field but the nominators should be EPNOE members.

#### Call for Nominations: EPNOE Young Scientist Award

EPNOE Association is inviting nominations for the EPNOE Young Scientist Award in the field of polysaccharide science and technology. The prize will be given to outstanding work of doctoral students and scientists with up to seven years of experience since completion of doctoral studies. The EPNOE Young Scientist Award is international, and all young scientists conducting research in the field of polysaccharides are eligible for nomination. The deadline for nominations is **September 30th, 2020 16:00 CET.** The process of nomination is strictly confidential and self-nomination is discouraged. Only EPNOE members can be nominators.

#### How to nominate?

- · One nomination letter describing the outstanding achievements of the candidate
- One short CV of the candidate (max 2 pages)
- · List of three most relevant publications of the candidate

Where to send the information? Combine the documents in one .pdf file and send it via email to monicaek@kth.se by September 30th 16:00 CET. No nominations will be accepted after the deadline.

**Who will judge the merits of the candidates?** A committee of seven jury members will evaluate the merits and achievements of the candidates. All members are distinguished scientists and technologists in the field of polysaccharides.

When the EPNOE Young Scientist Award will be presented? The winner of EPNOE Young Scientist Award 2020 will be announced during the EPNOE Junior Conference in Kortrijk, Belgium in February 2021. The Awardee will receive 3000 euros.

We are looking forward for your nominations. **Prof. Monica Ek, VP EPNOE Awards and Memberships** 

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#### Collaboration between The European Polysaccharide Network of Excellence (EPNOE) and the MDPI journal "Polymers and Polysaccharides"

The European Polysaccharide Network of Excellence (EPNOE) collaborates since May 2020 with MDPI Journals Polymers and Polysaccharides. MDPI is a publisher of scholarly open access journals. All journals uphold a peer-reviewed, rapid, and rigorous manuscript handling and editorial process.

Polymers (ISSN 2073-4360), https://www.mdpi.com/journal/polymers) is an international, open access journal of polymer science. It publishes research papers, communications, and review articles. Polymers provides an interdisciplinary forum for publishing papers which advance the fields of (i) polymerization methods, (ii) theory, simulation, and modelling, (iii) understanding of new physical phenomena, (iv) advances in characterization techniques, and (v) harnessing of self-assembly and biological strategies for producing complex multifunctional structures. Scientists are encouraged to publish their experimental and theoretical results in as much detail as possible. Therefore, there is no restriction on the length of the papers. The full experimental and computational details must be provided so that the results can be reproduced. The scope of Polymers includes: Polymer Synthesis Polymer Analysis Polymer Physics Polymer Theory and Simulation Polymer Processing and Performance Polymer Applications Biobased and Biodegradable Polymers.

In addition, the following two special issues are accepting contributions:

- "Polysaccharides II" - Guest Editor: Prof. Dr. Karin Stana Kleinschek, Graz University of technology. Submission deadline: 30 September 2020

- "Preparation of Bacterial Cellulose and its Biomedical Applications" – Guest Editor: Dr. Selestina Gorgieva, University of Maribor. Submission deadline: 31 January 2021



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### Virtual summer course on Lignocellulose Biorefinery/Carbohydrate Chemistry

#### Aalto University and Graz University of Technology (TUGraz)

Lecturer: Herbert Sixta

#### We offer two different course versions for doctoral and master students:

- 3 ECTS lecture attendance and preparation of a PowerPoint presentation (pitch) on a selected topic. Alternatively, they can take an oral exam via TEAMS
- 5 ECTS lecture attendance and accepted scientific report on a selected topic related to Biorefineries . Submission deadline for the report is 31 August 2020. We do not accept the course report submis sions after the 31 August 2020.

#### Course enrolment is open until June 5th 2020!

- a) Graz students register via the TUGonline system for course 661.761.
- b) Aalto students and all other participants enroll via webropol link https://link.webropolsurveys.com/S/84F4BC8C9D08586A
- c) Company participants need also to apply for non-graduate study right with us. https://into.aalto.fi/download/attachments/12356772/506\_nongraduate.pdf?ver sion=1&modificationDate=1554122921951&api=v2



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## Virtual summer course on Lignocellulose Biorefinery/Carbohydrate Chemistry

(continued)

<u>Details</u> Course T	itle Carbohydrate Chemistry/ Lignocellulose Biorefinery	
Course Nr.	TU Graz LV Nr. 661.761 Aalto University CHEM010Z-LZ	
Dates	June 16th – 18th 2020 Days are fixed, detailed lecture hours will be announced later -full day courses	
Content	At the beginning of the course the physical-chemical phenomena of ligno cellulose impregnation, typically wood, will be introduced and later deepe ned in short exercises.	
	Afterwards the most important technical pretreatment methods will be pre- sented, before the main part of the lecture will continue with the chemistry and technology of lignocellulose fractionation processes. In addition to the commercial fractionation methods of kraft and acid sulfite digestion, hydro- thermolysis and the most important organosolv fractionation methods will be presented and critically discussed.	
	In selected examples such as heat transfer during the steaming of wood chips, the kinetics of xylose formation during the diluted hydrolysis of ligno- cellulose, the diffusion of ionic species in a wood chip, the flow mechanics within a wood chip and the Donnan equilibrium are presented and calculated in detail.	
	Finally, examples for the valorization of lignocellulose-containing compo- nents such as cellulose for the production of regenerated fibres, lignin for the production of monoaromatics or polyols and hemicellulose-containing building block chemicals such as furans and the end products that can be produced from them are presented.	
	An important concern is to discuss the sustainability of new processes in comparison to conventional processes and to identify differences and potential for improvement.	
	Further information available under: https://into.aalto.fi/display/endoctoralchem/Courses+offered (Aalto University)	
	Lecture 661.761 (Graz University of Technology)	



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### Virtual summer course on Lignocellulose Biorefinery/Carbohydrate Chemistry

(continued)

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Lecturer:

Univ.-Prof. Dr. Herbert SIXTA, Head of Department of Bioproducts and Biosystems, Aalto University, Helsinki/ Espoo, Finland

Prof. Sixta has more than 35 years of experience in industrial research on pulp and cellulose chemistry. The scope was extended to lignocellulosic biorefineries after his appointment as professor at Aalto University in 2007, where he now functions as the Head of Department of Bioproducts and Biosystems comprising 24 tenured positions. His core interest comprises the use of tailored ionic liquids for the selective dissolution of different biopolymers as a novel way of biomass fractionation. In material science the focus is laid on the development of high added-value cellulose material regenerated from ionic liquid solution as well as the synthesis of building block chemicals by heterogeneously catalyzed conversion routes from polysaccharides. He has authored more than 250 peer-reviewed publications, several books and has been awarded several prizes for his work.

#### Professor Sixta's broad research interests include the following:

Pulping chemistry and technology with special emphasis on dissolving pulps Cellulose chemistry Chemistry of the fractionation of biomass, in particular lignocellulosic biomass Valorization of cellulose to regenerated cellulose fibers and cellulose derivatives Valorization of hemicelluloses as furanic compounds Isolation, characterization and valorization of lignin Chemical and mechanical purification of pulps TCF- and ECF bleaching techniques Organosolv fractionation methods with particular emphasis on GVL/water pulping of hardwood

## Treesearch and Wallenberg Wood Science Center (WWSC), Sweden, YouTube virtual conference series on Biorefinery

WWSC is a joint research center and collaboration between KTH Royal Institute of Technology, Chalmers University of Technology and Linköping University. The base is a donation from Knut and Alice Wallenberg Foundation. The forest industry is supporting WWSC via the national platform Treesearch.

https://treesearch.se/en/treesearch-wwsc-virtual-conference-series/



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#### Institute for Chemistry and Technology of Biobased Systems - IBioSys

#### New Institute at Graz University of Technology



https://www.tugraz.at/institute/ibiosys/home/

The Institute for Chemistry and Technology of Biobased Systems IBioSys is based on the amalgamation of basic research (Chemistry) and applied research (Technology) of Biobased Systems such as bioconjugates containing bioactive carbohydrate moieties, with the main goal embedding the interdisciplinary research field in combination with knowledge and technology transfer to teaching and research, industry and society on the local, regional and international level. It is a HUB of innovation for Chemistry and Technology of biobased Systems in accordance with the Fields of Expertise of Graz Technical University, most importantly Advanced Material Science and Human Biotechnology as well as with the regional cooperation NAWI Graz (University Graz) and BioTechMed (Medicinal University Graz).

The research activities of **IBioSys** implement two main research fields with a common interest **'Biobased Systems' – bioactive carbohydrate based bioconjugates**:

- Chemistry of Biobased Systems
- Technology of Biobased Systems

• **Chemistry of Biobased Systems**: The design and development of efficient and economic synthetic methods for bioactive glycoconjugates and analogues as well as glycomimetics based on renewable resources and their application as potential tools for the profiling as well as structure-activity-studies of glycoprocessing enzymes and as therapeutics for management of diseases related to the carbohydrate metabolism. Design, synthesis, modification and characterisation of biobased systems such as oligo- an polysaccharides for their application in material technologies such as 3D printing technologies, tissue engineering, coating technologies, fabrication of surface active materials, medicinal applications, applications or auxiliary materials for paper- and pulp industry.

• **Technology of Biobased Systems:** Development of complex materials, conjugates based on biomolecules of living organism origin and analogues. This implements the manufacturing, analysis and application of organic structures and inorganic/organic hybrid-systems. The focus is set on the development of biomaterials with emphasis on surface specific processes (surface functionalisation) and manufacturing of 3D structured materials and using modern technology as 3D printing or laser lithography, development of bio-inks formulations, cross linking - and self-assembly structures, for as example tissue engineering. Another research topic is the use of mono-, oligo- and polymers and derivatives for different technical applications such as packaging, paper conservation, paper process technology, textile industry, coating or cosmetics.



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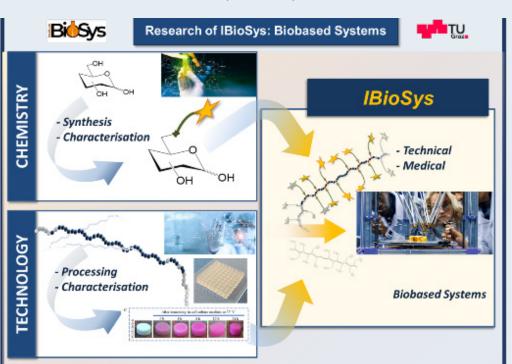
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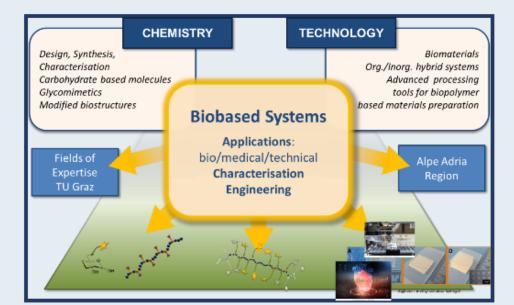


## Institute for Chemistry and Technology of Biobased Systems - IBioSys

#### New Institute at Graz University of Technology

(continued)





Prof. Dr. Karin Stana Kleinschek, IBioSys, TU Graz, Austria Prof. Dr. Tanja Wrodnigg, IBioSys, TU Graz, Austria



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Carbohydrate lectures at TU GRAZ at Institute for Chemistry and Technology of Bio based Systems



https://www.tugraz.at/institute/ibiosys/home/

At TU Graz, Austria, following lectures will be given by the EPNOE members in the frame Online Course CHE.556UF Carbohydrate Technologies course, Master studies Chemistry, organized by Prof. Dr. Tanja Wrodnigg, IBioSys (Programme attached):

• Prof. Dr. Pedro Fardim (University Leuven, president of EPNOE):

Topochemical Fabrication of Polysaccharide-based Materials for Advanced Biomedical Applications: Gels and Surfaces

• **Prof. Dr. Karin Stana Kleinschek**, TU Graz, Institute for Chemistry and Technology of biobased systems (BioSys) **Polysaccharides in Medical and Technical Application** 

• **Dr. Tiina Nypelö**, Chalmers University of Technology, Department of Chemistry and Chemical, Engineering: **Polysaccharide barrier materials** 

• Stefan Spirk, TU Graz, TU Graz Institut für Papier- Zellstoff- und Fasertechnik, TU Graz

Polysaccharide (nano)materials and analytical methods

• **Rupert Kargl**, TU Graz, Institute for Chemistry and Technology of biobased systems (BioSys)

Polysaccharide derivatization reactions in science and industry

Power point material of the lectures will be accessible for the EPNOE members on line.



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### Inorganic Post-Hybridization of 3D Printed Biopolymers and Their Biological Relevance

Selestina Gorgieva<sup>1,2</sup>, Manja Kurečič<sup>1,2</sup>, Rupert Kargl<sup>1,2</sup>, Ana Bratuša<sup>1</sup>, Roland Fisher<sup>3</sup> and Angela Hameli<sup>3</sup>

<sup>1</sup> Institute of Engineering Materials and Design, Faculty of Mechanical Engineering, University of Maribor, Maribor, Slovenia, <sup>2</sup> Institute of Automation, Faculty of Electrical Engineering and Computer Science, University of Maribor, Maribor, Slovenia,<sup>3</sup> Graz University of Technology, Institute of Inorganic Chemistry, Graz, Austria

Development of materials for bone regeneration as an immunologically safer replacement of autografts and allografts is a challenging and demanding task. Despite enormous progress in material science in term of types, modification routes and processing technologies, bone regeneration procedures still seek for material-based tools and devices that lead to more predictive outcomes. A common denominator in already successful trials is the utilization of host tissue-like materials, i.e. biological substitutes as engineering building blocks. Such approaches ultimately integrate organic and inorganic materials, similar to the type I collagen (organic) and (nano-) hydroxyapatite (inorganic) assembly in bone. The human bone is a rather complex tissue, comprised of 20–40% of organic phase (collagen and non-collagenous proteins), 10% water, and 50–70% mineral phase (hydroxyapatite and small amounts of magnesium, zinc, silicon, carbonates). Mimicking the bones' hierarchical structure in a material for regenerative medicine is expected to trigger desirable responses within the biological niche.

To contribute to fundamental know-how in this area, and introduce novelty in terms of material processing and modification, the proposed bilateral project will focus on: i) Processing and factorial optimization of a 3D printed object, based of bio medically relevant materials (single polylactic acid /PLA/ or mixture with collagenous protein) and ii) Surface modification post-printing with inorganic silica, phosphates, titanates etc. via dopamine polymerization chemistry (SLO) or (RO3)Si(CH2)3N(CH2)2NH(CH2)2NH(CH2)2NH2 polymer (AUS).

This leads to the aim of developing hybrid materials with superior properties in terms of bio-mineralization and biological response.

This project represents a continuation of research activities between both groups and a step forward from a recently established "proof of concept" on more simplified, film-like models. Very promising initial data encouraged a maturation of the idea, which can be unambiguously realized through this bilateral collaboration. Both collaborating groups will contribute complementary in material preparation and characterization techniques and expertise. This is expected to generate transfer of knowledge as well as new know-how on advance processing tools. The Slovenian principle investigator Assist. Prof. Selestina Gorgieva has over ten years of research experience with (bio) polymeric porous 3D scaffolds for medical applications, including chemical modification of biopolymers and their precursors for targeting bioactivity (mineralization). The Austrian principal investigator Dr. Angela Chemelli has experience in the application of small angle X-ray scattering and elastic and guasi-elastic light scattering methods to (bio-) polymers, nanostructured materials and nanoparticles. Besides, her research was focused on nanostructured liquid crystal and surfactant systems and their structural modification and the preparation of nanoparticles and colloids. The expertise of both teams and availability of research infrastructure supported by close collaboration with biological testing institutes will facilitate the project execution.

**Acknowledgement**: Research core program group for Textile Chemistry No. P2-0118, and project BI-AT/20-21-003, financed by the Slovenian Research Agency (ARRS).



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#### Modified Bacterial Cellulose as Artificial Biomimetic Membrane for Biological Blood Brain Barrier

Selestina Gorgieva<sup>1,2</sup> and Ivana Vinković Vrček<sup>3</sup>

<sup>1</sup> Institute of Engineering Materials and Design, Faculty of Mechanical Engineering, University of Maribor, Maribor, Slovenia

<sup>2</sup> Institute of Automation, Faculty of Electrical Engineering and Computer Science, University of Maribor, Maribor, Slovenia

<sup>3</sup> Institute for Medical Research and Occupational Health, Zagreb, Croatia

Bilateral project (BI-HR/20-21-00) presents initiation of common research activities between research teams from University of Maribor, Slovenia (Faculty of Mechanical Engineering) and Institute for Medical Research and Occupational Health, Zagreb, Croatia. Both groups will join their expertise in biotechnology and nanomedicine to develop novel and more physiologically relevant in vitro blood brain barrier (BBB) system employing bacterial cellulose (BC) as a basement membrane. To contribute to fundamental know-how in this area and introduce novelty in terms of material processing and model examination, the proposed bilateral project will focus on:

- Processing of modified BC membranes,
- Comprehensive characterization including factorial design models,
- Immobilization of specific brain microvascular endothelial cell line hCMEC/D3 on BC, and

Evaluation of quality and efficacy of novel in vitro system using relevant protocols for BBB testing.

Main expected outcome will be a detailed strategy for development of efficient in vitro BBB model that can be applied for testing of medical nanoproducts such as nano-enabled drug delivery systems. Complementary expertise and skills of Slovenian and Croatian partners will enable achievement of project objectives. The Slovenian principle investigator dr. Selestina Gorgieva has ten years of research experience with (bio) polymeric porous 3D scaffolds for medical applications (PP-gelatin implants for hernia treatment, polyphenol-chitosan hydrogels for soft tissue regeneration, mineralized gelatin-nanocellulose composites, gelatin- coated Mg monoliths for hard tissue regeneration, and GTR membranes for periodontal treatment). She is experienced in the chemical modification of biopolymers such as BC, towards targeting of bioactivity. Croatian project leader dr. Ivana Vinković Vrček has pioneered safety-oriented nanomedical research in Croatia. Her research group is a unique multidisciplinary team in the Republic of Croatia focused on nanomedicine and nanosafety with expertise in synthesis, characterization & functionalization of nanoparticles for biomedical applications, interaction of nanomaterials with biological systems, and safety assessment of nanomaterials for biomedical applications.

Project implementation (within 1.1.2020 - 31.12.2021 period) will generate both way transfer of knowledge, as well as new know-how in development of relevant in vitro BBB model. Nonetheless, the project is also a great opportunity for the involved early stage researchers to facilitate career development within an international collaborative environment, together with people from different cultural, socioeconomic and educational backgrounds.

Acknowledgement: Research core program group for Textile Chemistry No. P2-0118, and project BI-HR/20-21-00, financed by the Slovenian Research Agency (ARRS) and Ministry of Science and Education of the Republic of Croatia.



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## Recent advances in polysaccharide based nanoparticles for biomedical applications

Martin Gericke, Thomas Heinze

Friedrich Schiller University of Jena, Institute for Organic Chemistry and Macromolecular Chemistry, Center of Excellence for Polysaccharide Research, Humboldtstraße 10, D-07743 Jena, Germany

Correspondence to: martin.gericke@uni-jena.de, thomas.heinze@uni-jena.de

Nanoparticles (NP) are intensively studied in fundamental- and applied sciences due to their unique physical, chemical, and biological features. The health and environmental safety of NP is an issue of increasing concern and awareness considering their application potential in branches such as medicine, analytics, personal care, and consumer products. Polysaccharide (PS) based nanomaterials have a huge advantage in this regard because they are inherently biocompatible, sustainable, and possess a positive public perception.

PS-NP can be obtained by self-assembling of various hydrophobic PS derivatives (e.g., esters, ethers, carbamates, carbonates) with different polymer backbones (e.g., cellulose, dextran, starch, xylan, pullulan). The NP formation is induced by a controlled transition from the dissolved to the solid state (nanoprecipitation). The approach is easy to perform and broadly applicable; compounds with different hydrophobic substituents have been employed successfully. Own current research strategies focus on the development of functional PS-NP.

(I) Advanced PS chemistry. A broad variety of functionalities (e.g., bio-affinity groups, sensor molecules, drugs) has been incorporated into the nanomaterials by using advanced organic synthesis methods. Reactive PS-NP have been designed that are stable in water and enable to directly capture sensitive molecules (antibodies, enzymes) with high binding efficiency (up to 90%) and without the need for any previous activation steps.

(II) *Application potential in biomedical areas*. Biocompatible PS-NP are currently developed for the in vivo delivery of anti-inflammatory drugs with a fast and a slow release kinetics in the frame of the *Collaborative Research Center 1278 PolyTarget* (https://www.polytarget.uni-jena. de/en, Figure 1).

(III) Upscaling. The Center of Excellence for Polysaccharide Research Jena / Rudolstadt is going to address the major bottleneck of future academic and applied R&D activities; the limited production capacity of current lab-scale procedures. New technologies are explored to make functional PS-NP accessible in technical scale.

#### **References**:

1. M. Gericke, P. Schulze, T. Heinze "Nanoparticles Based on Hydrophobic Polysaccharide Derivatives - Formation Principles, Characterization Techniques, and Biomedical Applications" *Macromol. Biosci.* **2020**, *20*, 1900415

2. H. Lindemann, M. Kühne, C. Grune, P. Warncke, S. Hofmann, A. Koschella, M. Godmann, D. Fischer, T. Heinzel, T. Heinze "Polysaccharide Nanoparticles Bearing HDAC Inhibitor as Nontoxic Nanocarrier for Drug Delivery" *Macromol. Biosci.* **2020**, *doi:* 10.1002/ mabi.202000039



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## Recent advances in polysaccharide based nanoparticles for biomedical applications

(continued)

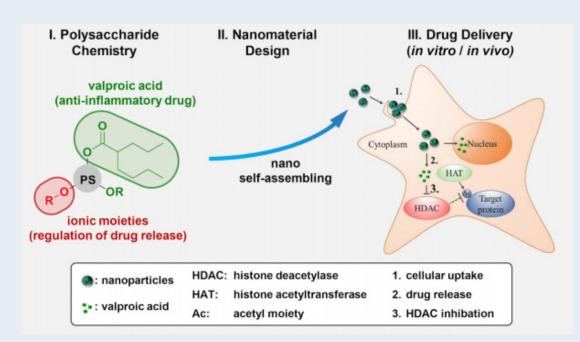


Figure 1. Schematic representation of functional polysaccharide based nanoparticles in a drug delivery application.



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# EU Single-Use Plastics Directive and its relevance to cellulosic fibres

Dr. Richard Blackburn BSc PhD CCol FSDC FRSA MRSC

#### Introduction

I have been researching, publishing, and teaching cellulose chemistry, cellulosic fibres, their application, and their sustainability for over 20 years. As such, it might be interesting to other polysaccharide researchers that I have become involved with the consultation panel for EU Directive 2019/904 on the "Reduction of the impact of certain plastic products on the environment", also known as The Single-Use Plastics (SUP) Directive.

Most people with an interest in polysaccharides would be forgiven asking why a cellulosics researcher is involved with legislation surrounding "plastic", indeed the Directive itself defines plastic as "a material consisting of a polymer... to which additives or other substances may have been added, and which can function as a main structural component of final products, with the exception of natural polymers that have not been chemically modified". The Directive further explains that "unmodified natural polymers, within the meaning of the definition of 'not chemically modified substances'... should not be covered by this Directive as they occur naturally in the environment". REACH further qualifies this by stating that a "not chemically modified substance" means "a substance whose chemical structure remains unchanged, even if it has undergone a chemical process or treatment, or a physical mineralogical transformation, for instance to remove impurities".

And yet, regenerated cellulose (cellulose II) fibres, such as viscose and lyocell, are under consideration within this draft legislation, with suggestions that they do fulfil the definition of being a "plastic" and would, accordingly, fall under the SUP Directive. Whilst this legislation is well intended, it has the potential to restrict the freedom of use of certain polymeric substances that are more sustainable, with lower environmental impact, than those it does not restrict. This requires further definition based on a more detailed knowledge of the chemistry and sustainability of cellulose-based fibres.

The purpose of my statement herein is to provide chemical rationale for why regenerated cellulose (cellulose II) fibres, such as viscose and lyocell, do not fall under the definition of "plastic" of EU Directive 2019/904, and why they meet the definition of "natural polymers that have not been chemically modified" that are exempt from the definition of "plastic".

#### **Cellulosic fibres**

Cellulose is the most abundant biopolymer on earth, a linear polymer composed of glucose units that are linked by  $\beta$ -1,4 glycosidic bonds formed between the carbon atoms C(1) and C(4) of adjacent glucose units. Cellulose is able to form highly ordered structures, which occurs as a result of extensive interaction through intra- and intermolecular hydrogen bonding of the three hydroxyl groups in each cellulose unit. The supramolecular structure of cellulosic fibres can be described by a two-phase model with regions of high orientation (crystalline) and low orientation (amorphous)., Cotton fibres are one of the purest sources of cellulose and the most used industrial natural fibre.



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# EU Single-Use Plastics Directive and its relevance to cellulosic fibres

(continued)

#### Cotton

The European Parliamentary Research Service (EPRS) highlights that the production of raw materials is responsible for a large share of the environmental impact of the textiles industry, not least from growing crops for natural fibres. The most widely used crop for producing cellulosic fibres is cotton, but the use of cotton, according to the European Clothing Action Plan (ECAP), is challenging from a sustainability perspective because it requires significant land, water, fertilisers and pesticides to grow. Indeed, the European Commission Joint Research Centre Institute for Prospective Technological Studies, the European Environment Agency, and the Ellen MacArthur Foundation all recommend a significant reduction in the use of cotton.

EPRS reports that the industry is testing less frequently used natural fibres, such as hemp, flax, linen and nettle, that require less water, fertilisers and pesticides, and working with innovative materials that are more sustainable, such as lyocell highlighting its raw material: "cellulose from eucalyptus, which grows quickly and requires no irrigation or pesticides". Clearly there is recognition from the EU that cotton is environmentally challenging, and draws attention to more sustainable alternatives, such as lyocell and viscose fibres.

#### The effect of alkali on the structure of cellulose

Cellulose as in nature is generally found in planta in the cellulose I $\beta$  crystal form, but this form may change according to various conditions that the cellulose may encounter. Alkali has a significant effect on morphological, molecular, and supramolecular properties of cellulose. In its interaction with native cellulose, aqueous sodium hydroxide above a certain concentration is able to penetrate the cellulose I $\beta$  crystalline lattice to produce a series of well-defined crystalline complexes holding a number of sodium ions and water molecules within the lattice. Okano & Sarko presented that a total of five unique alkali-celluloses (Na-celluloses) could be generated reproducibly, depending on the alkali concentration used and temperature of treatment, which are named Na-celluloses I, IIA, IIB, III, and IV (Figure 1). When treated with certain concentrations of alkali, cellulose I $\beta$  adopts a modified crystal structure, forming cellulose II, which is the stable fibre form after drying; the transformation mechanism of cellulose I to cellulose II is a chain-polarity transformation (parallel-to-antiparallel) (Figure 2).,

The most stable crystal form of cellulose I $\beta$  has intramolecular hydrogen bonds between C2 and C6 hydroxyls and C3 hydroxyl and the glucose ring oxygen and intermolecular hydrogen bonds between C2 and C6 hydroxyls of adjacent chains (Figure 3). On conversion by the action of sodium hydroxide (NaOH), the most stable crystal form of cellulose II only has intramolecular hydrogen bonds between the C3 hydroxyl and the glucose ring oxygen, and intermolecular hydrogen bonds between C2 and C6 hydroxyls of adjacent chains.



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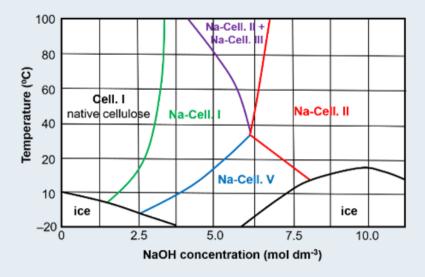
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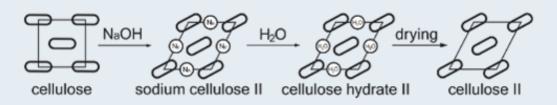


### **EU Single-Use Plastics Directive and its** relevance to cellulosic fibres

(continued)



#### Figure 1. Phase diagram of the ternary system cellulose/NaOH/water.



#### Figure 2. Cellulose crystal structure change on alkaline treatment.

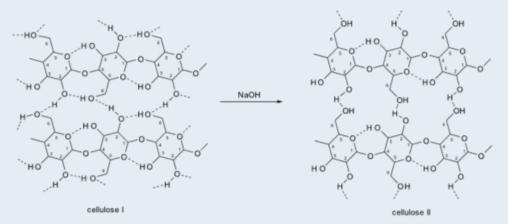


Figure 3. Cellulose Iβ crystal (showing hydrogen bond network) and conversion to cellulose II crystal (showing hydrogen bond network).



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# EU Single-Use Plastics Directive and its relevance to cellulosic fibres

(continued)

REACH states that "chemical modification includes but is not limited to hydrogenation, neutralization, oxidation, esterification, and amidation".2 Whilst this is not a finite list of chemical modification processes, what is common to these processes is that they all change chemical groups in the final product by means of covalent bond formation.

It is important to note that in the process of transformation of cellulose I to cellulose II is not a chemical change; it is a physical change in a limited number of hydrogen bonds between molecules. This is analogous to changes in hydrogen bonds when water freezes; there is no change in chemical composition of the substance, when water is liquid it has H2O molecules, when water is in solid form it has H2O molecules. When water freezes intermolecular hydrogen bonds change, but there is no change in the chemical composition of the substance, the only difference is the extent of hydrogen bonding, which is a physical change. When water molecules are in the liquid state the average number of hydrogen bonds a water molecule has is 3.4 bonds; during freezing more additional hydrogen-bonds form between water molecules, and the number of hydrogen bonds per water molecule is 4 bonds.

After treatment of cellulose I with alkali, the fibre surface appearance and the internal structure of the fibre are modified, leading to a number of changes in fibre and fabric properties, including a more circular fibre cross-section, increased lustre, increased tensile strength, increased fibre moisture regain, increased water sorption, and improved dimensional stability. In addition, mercerised cotton is more biodegradable; untreated cotton is less susceptible to enzymatic degradation than cotton treated with ammonia and/or sodium hydroxide, due to an increase in the amorphous regions in the fibre as a result of the mercerisation treatment.

#### Viscose

The viscose process was first described in the 1890s and the name originates from the viscous cellulose solution that was used as spin dope. Viscose is manufactured by soaking cellulose pulp in sodium hydroxide (NaOH) to produce alkali cellulose, followed by aging for 2 to 3 days, then mixing with liquid carbon disulphide (CS2) to produce sodium cellulose xanthogenate (Figure 4).



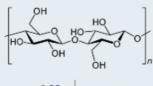
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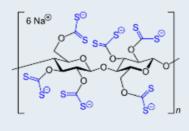


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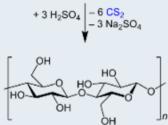


Figure 4. Viscose process for production of regenerated cellulose.

Cellulose xanthogenate is subsequently dissolved in aqueous sodium hydroxide and used in a wet-spinning process, where the cellulose solution is precipitated as pure cellulose fibres in a coagulation bath consisting of acid (mainly sulfuric acid with a broad variety of other components including salts).20, The introduction of anionic dithiocarbonate groups to the cellulose chain renders the cellulose into an alkali or a water soluble polymer, the homogeneous thick aqueous viscose solution is then passed through spinnerets in an acid bath for the regeneration to produce regenerated cellulose. The resultant regenerated cellulose fibres that are formed are cellulose II fibres, essentially the same as formed through the alkali treatment method.

There are concerns about the sustainability of viscose in relation to the way in which it is produced. However, if produced using the right processes, which is the case in Europe, viscose fibres are highly sustainable. Sustainable viscose fibres made in Europe are produced using sustainably produced wood and manufactured in the most environmentally friendly process possible. High recovery of by-products enables a closed-loop process to be achieved – wastewater and emissions are minimized, and sodium sulfate generated in fibre production is recovered (which finds high-value use in the detergent, glass and food industries).



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(continued)

#### Lyocell

Lyocell fibres are cellulosic fibres that are produced by regenerating cellulose into fibre form out of a solution in N-methylmorpholine-N-oxide (NMMO) hydrate, and have excellent environmental credentials., Solvent-spun lyocell fibres consist of crystalline cellulose II and amorphous cellulose and have been shown to have varying crystallinity content from 80%,16 through 57% and 47%,25 to 35%.3 However, it is generally observed that lyocell fibres have a higher degree of crystallinity in comparison with other regenerated cellulosic fibres, such as modal and viscose.3,16 In the crystalline regions of cellulose II polymers the layered structure is very regular, hence the length of hydrogen bonds between molecules is the same;, the structure is an antiparallel arrangement of cellulose chains with some inter-sheet hydrogen bonding, generally leading to a perfectly distributed symmetrical structure (Figure 5).

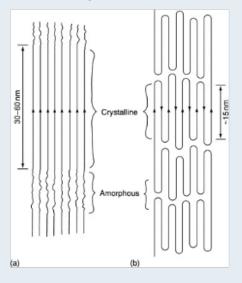


Figure 5. (a) Cellulose I parallel and (b) cellulose II antiparallel structures.

Cellulose II fibres formed through the lyocell process are essentially the same polymer as formed when cellulose I is treated with alkali. Again, it is important to note that the fibres are formed as a result of a process that courses a physical change in the structure of the cellulose through changes in the hydrogen bonding interactions. The lyocell process does not cause any chemical change in the cellulose polymer and no covenant bonds are formed.

Lyocell is arguably the most sustainable fibre available due to the highly sustainable raw materials from which it is formed (eucalyptus trees), the closed-loop process in which is it is formed using green chemistry methods, and the high biodegradability of the resultant fibre.



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(continued)

#### **Cellulose acetate**

Cellulose acetate is the acetate ester of cellulose (Figure 6). Although it is also formed from cellulose I derived wood pulp, it would be considered a chemically modified polymer, meeting the REACH definition of "chemical modification"2 as it is based on an esterification process where new covalent bonds would be formed in the final polymer. Cellulose acetate is made by reacting the cellulose with acetic acid and acetic anhydride in the presence of sulfuric acid and may be subjected to controlled, partial hydrolysis to remove the sulfate and sufficient acetate groups to give the product the desired properties. After it is formed, cellulose acetate is dissolved in a suitable solvent (typically acetone) and extruded through spinnerets into fibres.

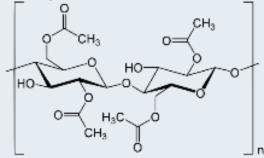


Figure 6. Chemistry structure of cellulose acetate.

#### Biodegradability of cellulosic fibres

In reducing the impact of "plastic" products on the environment, it is important that the biodegradability of polymers in that environment is considered. The 2019 European Chemicals Agency (ECHA) restriction report on intentionally added microplastics specifically states that their proposed restriction shall not apply to "polymers that occur in nature that have not been chemically modified" and "polymers that are (bio)degradable". The report goes on to state, under the heading "Biodegradability", that "natural polymers are inherently biodegradable and therefore, not included in the scope".

Cellulosic fibres of all forms are biodegradable as determined through many research studies; indeed, cellulose II fibres have often been found to be more rapidly degraded than their cellulose I counterparts. Park et al. evaluated the biodegradability of different cellulose fabrics by use of a soil burial test, an activated sewage sludge test, and an enzyme hydrolysis test. They demonstrated that the most biodegradable fibre was viscose (rayon), followed by cotton, with cellulose acetate being the least biodegradable; linen showed inconsistent behaviour. It was argued that viscose fibres (cellulose II) were the most degradable because of their low crystal-linity and low degree of orientation, relative to cotton and linen (cellulose I). Despite cellulose acetate also having low crystallinity, the presence of hydrophobic acetate groups in its structure confers very low biodegradability.



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### EU Single-Use Plastics Directive and its relevance to cellulosic fibres

(continued)

Sang et al. studied the biodegradability of viscose and lyocell fibres (both cellulose II) using soil burial test, activated sludge test and enzymatic hydrolysis. They observed that viscose fibres exhibited higher biodegradation in comparison with lyocell fibres, indicating that lower crystallinity in viscose favoured the biodegradation; however, both fibres were readily biodegradable. An examination of variation within lyocell fibres showed that fibres with lower crystallinity and higher moisture regain were the most biodegradable. Both studies concluded that biodegradability of cellulose fibres was closely related to the moisture regain of the fibres, which is related to hydrophilicity and internal structure of the fibers.31,32

*Warnock et al.* studied the biodegradation rates of viscose, cotton, and lyocell woven fabrics in soil. Half-life values (the time required for 50% of the fabric to decompose) were 22 days for viscose, 40 days for cotton, and 94 days for lyocell. Research by the USDA Agricultural Research Service into the biodegradation of nonwoven fabrics showed that in aerobic, moist, and warm soil cotton and viscose were highly degradable with half-lives of 12.6 and 7.6 days, respectively (viscose was more biodegradable than cotton). Tests on nonwovens made from the synthetic fibres polypropylene and polylactic acid showed that they were not biodegradable under the same test.

There is limited information available pertaining to the marine degradation of fibres. A study by the US EPA Mote Marine Laboratory reported "decomposition rates" for a range of household and other items of marine debris;, decomposition was estimated as the time it takes for the item to be no longer visible. A cotton shirt was found to have completely disappeared in 2-5 months, which would suggest that the marine degradation of cellulose II fibres will be within the same range, based on the soil biodegradation trends described above and the knowledge that cellulose II fibres are highly susceptible to enzymatic degradation.

#### Conclusions

The purpose of this paper is to demonstrate that highly sustainable cellulose II fibres, such as lyocell and viscose should not fall under the definition of "plastic" under EU Directive 2019/904 as they are based on natural biopolymers where their production process results in only a physical change in comparison with their form in planta. This paper also demonstrates that cellulose II fibres (regenerated cellulose fibres) are as biodegradable as cellulose I fibres in both terrestrial and marine environments.

This paper highlights the fact that if cellulose II fibres are in any way restricted or prejudiced in their use in products that the result is likely to be for manufacturers to move exclusively to cellulose I fibres, primarily cotton, for these applications and products, which would lead to a much less sustainable life cycle for such products due to the sustainability challenges of cotton.

This is all my scientific opinion, but I hope it provides the opportunity for scientific debate amongst other polysaccharide researchers within the scope of this proposed Directive, and more widely within the context of sustainability and circular economy.

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## **EPNOE Member's Scientific Publications**

At Jena University, Germany:

• Recent progress on cellulose-based ionic compounds for biomaterials Y. Yang, Y.-T. Lu, K. Zeng, Th. Heinze, Th. Groth, K. Zhang Advanced Materials (2020) DOI: 10.1002/adma.202000717

• Layer-by-layer coating of aminocellulose and quorum quenching acylase on silver nanoparticles synergistically eradicate bacteria and their biofilms A. Ivanova, K. Ivanova, A. Tied, Th. Heinze, T. Tzanov Advanced Functional Materials (2020) DOI: 10.1002/ adfm.202001284

• Polysaccharide nanoparticles bearing HDAC inhibitor as non-toxic nanocarrier for drug delivery H. Lindemann, M. Kühne, Ch. Grune, P. Warncke, S. Hofmann, A. Koschella, M. Godmann, D. Fischer, Th. Heinzel, Th. Heinze Macromolecular Bioscience (2020) DOI: 10.1002/mabi.202000039

• Synthesis and characterization of novel water-soluble 6-deoxy-6-(2-amino-2-(hydroxymethyl)propane-1,3-diol)cellulose derivatives A. Pfeifer, M. Gericke, Th. Heinze Advanced Industrial and Engineering Polymer Research (2020) DOI: 10.1016/j. aiepr.2020.02.001

• α-1,3-Glucan benzoate – A novel polysaccharide derivative M. Gericke, A. Tied, C. Lenges, Th. Heinze Advanced Industrial and Engineering Polymer Research (2020) DOI: 10.1016/j. aiepr.2020.01.003

• Nanoparticles based on hydrophobic polysaccharide derivatives - Formation principles, characterization techniques, and biomedical applications M. Gericke, P. Schulze, Th. Heinze Macromolecular Bioscience (2020) 1900415

#### **At IMT Mines Ales**

• Al Hokayem, K., El Hage, R., Svecova, L., Otazaghine, B., Le Moigne, N., & Sonnier, R. (2020). Flame Retardant-Functionalized Cotton Cellulose Using Phosphonate-Based Ionic Liquids. Molecules, 25(7), 1629.

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• Hajj, R., El Hage, R., Sonnier, R., Otazaghine, B., Rouif, S., Nakhl, M., & Lopez-Cuesta, J. M. (2020). Influence of lignocellulosic substrate and phosphorus flame retardant type on grafting yield and flame retardancy. Reactive and Functional Polymers, 104612.

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