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European Polysaccharide Network Of Excellence



"Nature makes polysaccharides, EPNOE turns them into products"

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editorial

Dear readers,

Two EPNOE scientists received major international awards this year, each on different continents.

Hans-Peter Fink, director of the Fraunhofer Institut Angewandte Polymerforschung in Gölm, Germany, received the **Anselme Payan award** from the American Chemical Society for his life time achievements in cellulose science. The ceremony will take place in New Orleans in April 2013.

Antje Potthast, assistant professor at BOKU university Vienna, Austria, received the **Hayashi Jisuke International cellulose award** from the Japanese Cellulose society in Sapporo in October 2012 for her work on polysaccharide aging applied to conservation science.

The EPNOE society is very proud of them and we all congratulate them for these remarkable achievements.

This issue is dedicated to the presentation of some of the works performed within EPNOE academic partners around the production and use of nano-objects. Lignocellulosics and polysaccharides are well suited for such a production owing to the complexity of molecular structures found in nature, opening ways to tailor the morphology of these nano-objects.

My final words are about the **EPNOE 2013 conference**. It will take place in Nice, France. Beware that dates have been changed. EPNOE 2013 will take place **21-24 October 2013**. Please keep this date free to join us in Nice.

With my best wishes,

Dr. Patrick Navard
Coordinator of EPNOE
Armines/Mines ParisTech/CNRS
CEMEF - Centre for Material Forming
Sophia-Antipolis
(France)

news

Forthcoming events

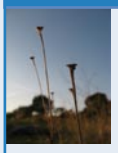
Course on Food Analysis

The course "Advanced Food Analysis" is organized by Graduate School VLAG in cooperation with Food Chemistry (Wageningen University), Organic Chemistry (Wageningen University) and the FP7 EC project QSAFFE and is held in Wageningen, the Netherlands from 28 January until 1 February 2013. More information is available via the link: http://www.vlag-graduateschool.nl/courses/food-analys.htm

BFF 2013

The symposium Biorefinery for Food, Fuel and Materials 2013 (BFF2013) will be held on 7-10 April 2013 at Wageningen University (The Netherlands). It will address technological innovations, implementation in the production chain and a public debate for the sustainable development of a Biobased Economy, with special focus on the synergy of food and non-food applications. More information on http://www.bff2013.wur.nl/UK/

Members' info



Awards

- On the occasion of the Zellcheming Annual Meeting 2012, Prof. Dr. Thomas Heinze from the University of Jena, Germany, was awarded the **Dr. Edmund Thiele votive medal** for excellent scientific achievements in the field of cellulose chemistry.

- Holger Wondraczek, from the University of Jena, Germany, was awarded the **ALCERU-Prize 2012** in the category «young academics» for his excellent work in the field of photoactive polysaccharide derivatives.

New PhD student

In the frame of the Industrial Chair in Bioplastics, Ahmed Abdennadher started his PhD at Cemef, Sophia-Antipolis, France, on the topic of «Structure of injected natural fiber reinforced composites», under the supervision of Tatiana Budtova and Michel Vincent.

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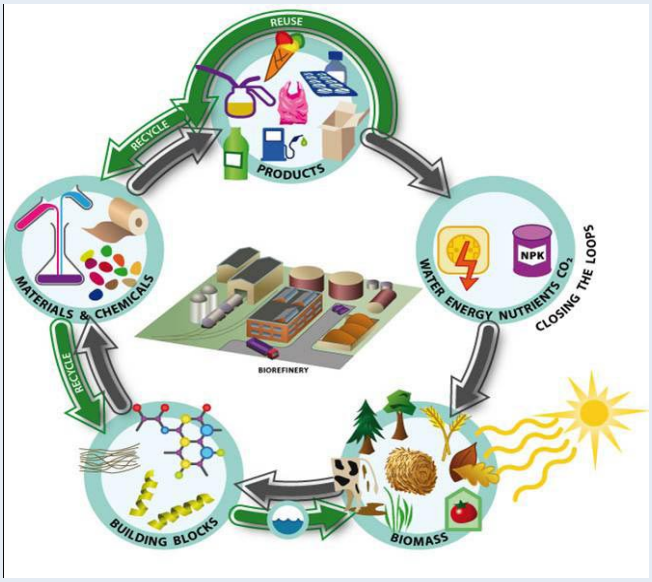
A European initiative

Biobased Industries Public Private Partnership: Biobased for Growth

At the beginning of 2012, several businesses, farmers and forests organisations started building the Public Private Partnership Bio-based Industries. This partnership cooperates in research and innovation towards demonstration of new biobased value chains and products, performing together activities that would not succeed when industries and sectors would act individually.

This partnership will operate within the European Union framework, financially supporting research and innovation, Horizon 2020. The partners represent this new value chain and include farmers, forestry, logistics, businesses in the agro-food and paper industries and biotechnology companies whose ambition is to create an economy based on renewable, recyclable and upcyclable raw materials. They recognise the enormous potential of making more efficient use of what is growing on the fields, one of the greatest challenges facing the 21st century.



You are invited to take part in the public consultation

The European Commission has launched the public consultation on "Bio-based industries towards a public-private partnership under Horizon 2020?". This public consultation is intended to provide opportunities for inputs from all those directly or indirectly connected with the bio-based industry value chain, e.g. scientists. The survey seeks input on the creation of a public-private partnership to promote research and innovation under Horizon 2020, the next EU Research Framework Programme.

You are encouraged to complete the questionnaire online until December 14:

http://ec.europa.eu/research/consultations/bio_based_h2020/consultation_en.htm

To learn more about the Biobased Industries PPP please, contact:

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Brussels
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bio.economy@cepi.org

This article was proposed and written by Margaretha Söderqvist Lindblad from Södra Innovation

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Special focus on nano objects

Preparation of spherical particles of nano-cellulose

Novel spherical nanoscaled cellulose particles have been prepared by high-pressure homogenizing of different pre-treated cellulose samples with Microfluidizer ™ processor (MF) in aqueous media.

One possibility of pre-treatment is a decrystallization step realized by dissolving and regenerating cellulose from a melt NMMO*H2O solvent system. Nanocellulose was obtained by a subsequent high-pressure mechanical treatment of the precipitate in aqueous dispersion. Decrystallization was also realized by grinding cellulose in a planetary mill. Ground cellulose was subsequently dispersed with high-speed stirrer Ultra-Turrax™ (UT) and high-pressure homogenizer. The amorphous intermediates were characterized by means of WAXS, Raman spectroscopy and DP determination.

By another way the preparation of nanoscaled cellulose was conducted by hydrolysis and following mechanical treatment of hydrolyzed cellulose with Ultra-Turrax™ and Microfluidizer™. A further alternative was given by the mechanical treatment of aqueous dispersions of low substituted cellulose derivatives. For example methyl cellulose, carboxymethyl cellulose and oxidized cellulose gave nanoscaled materials with interesting properties.

All cellulose nanosuspensions feature an increased viscosity and are stable without sedimentation or phase separation. Depending on the starting material and the pre-treatment step the dispersions are transparent (CMC, oxidized cellulose) or opaque (hydrolyzed cellulose, ground cellulose MCC). In contrast to cellulosic nanofibrils and cellulose nanowhiskers which are extensively discussed in literature, the novel cellulosic nanoparticles possess elemental particles with a spherical shape and diameters in the range from 50 to 500 nm. These particles are partly agglomerated, either forming string like, laminated, scaly or fluffy structure.

In order to obtain information about cellulose particle sizes, UT and MF treated dispersions were characterized by means of static and dynamic light scattering (DLS), ultra-centrifugation and scanning electron microscopy (SEM). Rheological measurements revealed the viscoelastic properties and gel-like structure of the materials as well as time- and shear-dependent effects like thixotropy and pseudoplasticity (structural viscosity).

In conjunction with potential applications, film forming properties and temperature dependent behaviour (e.g. viscosity) of the materials were investigated.
Selected samples of nanocellulosic dispersions were dried via lyophilizsation, via spray drying, and solvent exchange. The dried products were characterized in terms of porosity (mercury porosimetry) and particle morphology (SEM). Re-dispersed samples were compared with starting dispersions by means of SEM, DLS and rheometry.

Kay Hettrich[1], Manfred Pinnow[1], Bert Volkert[1], Lars Passauer[2], Steffen Fischer[2], Hans-Peter-Fink[1]

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[2] Technische Universität Dresden, Department of Forest Sciences, Institute of Plant and Wood Chemistry, Pienner Str. 19, 01737 Tharandt, Germany

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Special focus on nano objects

Design of polysaccharide- based nano-particles

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The nanoprecipitation by solvent displacement was introduced as simple and fast method for the preparation of well-defined spherical nanoparticles from different polysaccharide derivatives. Commercial cellulose esters including cellulose acetate and cellulose acetate propionate were studied for the encapsulation of pharmacologically and cosmetically active substances. Moreover, we focus on the design of new highly engineered polysaccharide derivatives and nanoparticles thereof. Thus, sensor-functionalities were covalently attached to polysaccharides and the nanoparticles obtained were successfully applied to explore the micro-environment in living cells (Fig. 1).

The particles obtained can be termed as passive systems, since their properties are governed by the intrinsic polymer characteristics and the preparation conditions. The specific conditions (e.g. pH value) for a drug release can be predetermined by the molecular structure of the polysaccharide derivative, but neither the exact location nor the timing of the release of the pharmacologically active compound can be controlled. Hence, the delivery cannot be adjusted to particular needs. Therefore, we start to develop responsive nanostructures with properties actively controlled by an external stimulus, e.g., light. For this purpose polyfunctional polysaccharide derivatives decorated, e.g., with photo-crosslinkable coumarine based chromophores were prepared applying sophisticated polysaccharide chemistry. The unique properties of polysaccharides are the basis for products that provide a controlled environment, spatial distribution, and mutual orientation of the chromophores. Such tailored structures result in very specific photochemical or photophysical effects (e.g. amplification of photomechanical effects). On the other, non-linear effects can be implemented in the molecular structure to extend the spectral range for triggering photoinduced changes from the UV to the physiologically harmless NIR, which presents a prerequisite for future in vitro and in vivo applications.

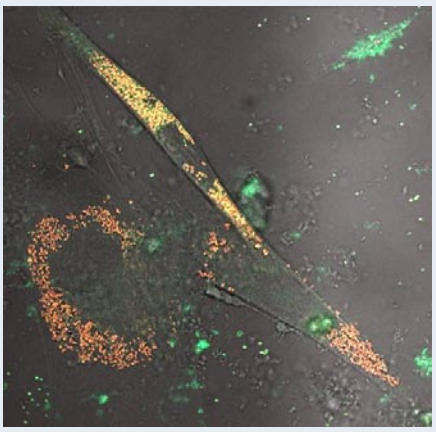

Fig. 1: Confocal micrograph of human fibroblasts incubated with Fluorescein-/Sulforhodamin dextran propionate nanoparticles; orange: acidic-, green: basic environment

Beside the application of light as a stimulus for the control of the properties of nano-scaled assemblies, techniques of optical spectroscopy give insight into the morphology of such structures. The fluorescence lifetime analysis of nano-scaled particles prepared from the coumarine decorated polysaccharides reveal that the particles consist of two domains. One very polar domain can be assigned to the charged surface of the particles. The other domain display not a discrete fluorescence lifetime and thus no exactly specified polarity but a lifetime distribution, which indicates the penetration of water in to the core of the particles. On the basis of this finding the simple hydrophobic core / polar surface model of the particles need to be redrawn (Fig. 2).

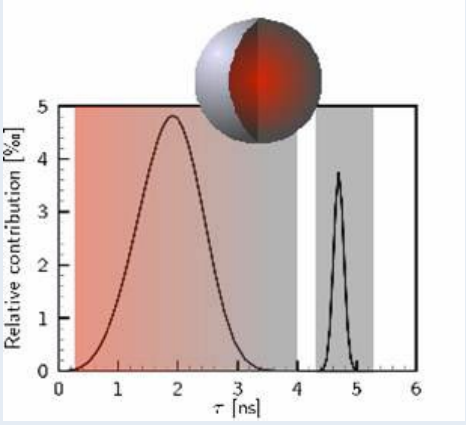

Fig. 2: Results of fluorescence lifetime distribution analysis and tentative picture of particle mophology; gray: polar-red unpolar micro environment

Holger Wondraczek, Thomas Heinze

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

European Polysaccharide Network Of Excellence

Special focus on nano objects

Plant- based nanoparticles

Thermoplastic starch is an interesting material that is now widely used in biodegradable polymer blends. However, the mechanical properties of thermoplastic starch are not very good and nanocomposites have been used successfully for improving them. Nanofillers like cellulose whiskers, montmorillonite, hectorite, etc… have been tried. Another synthetic class, called layered double hydroxide (LDH), has also been tested. LDH is a class of layered materials made of positively charged layers and charge counter-balancing anions located between the layers. The advantage is the huge variety of anions that can be intercalated which leads to many different materials.

Together with the laboratory of Dr F. Leroux in Clermont-Ferrand, LDH having lignosulfonates as interlayer anionic charge carriers were successfully prepared and used as a filler in thermoplastic starch. Synthesis is performed at room temperature. Lignosulfonate (LS) accommodates the interlayer space adopting a bilayer molecular arrangement resulting in a basal spacing of 2.54 nm.

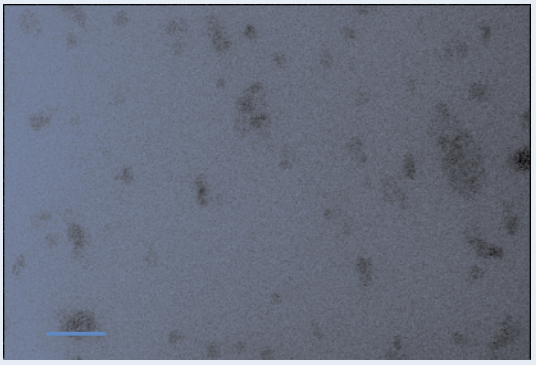

Fig. 1: LDH/LS dispersed in starch at 1% concentration seen by transmission electron microscopy. The white bar represents 100 nm.

However the crystallinity of the resulting bio-organoclay is weak, probably due to the difficulty of the inorganic sheets to be built on amorphous polymer chain, the latter inducing low structural ordering. Organoclays of composition Zn2Al/LS were then successfully incorporated and dispersed down to nanometer size in thermoplastic starch with an internal mixer. LDH/LS contents as low as 1% show a remarkable increase of mechanical properties.
This work is part of the PhD of Edwige Privas.

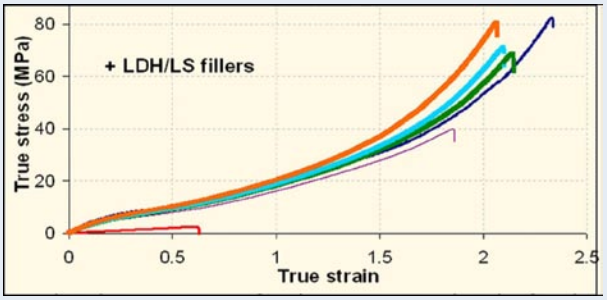

Fig. 2: Stress-strain curve for LDH/LS filled thermoplastic starch concentrated at 20% in polyethylene (PE). Blue: pure PE. Red: pure starch, pink: unfilled starch-PE. Other curves LDH/LS based blends with content of LDH/LS of1, 2, and 4%.

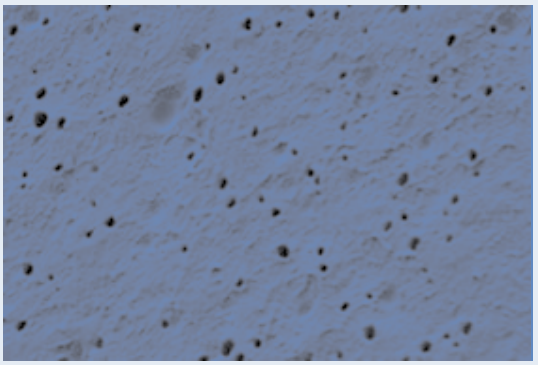

Fig. 2: HDL-LS 1%-reinforced starch particles (0.5µm diameter) dispersed in polyethylene

Patrick Navard[1], Fabrice Leroux[2]
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[2] Université de Clermont-Ferrand, France

EPNOE Member's award

Outstanding cellulose research

International ACS Award for Prof. Hans-Peter Fink, Director of Fraunhofer IAP

This year's Anselme Payen Award of the American Chemical Society, Cellulose and Renewable Materials Division, was bestowed on Prof. Hans-Peter Fink, Head of the Fraunhofer Institute for Applied Polymer Research, Potsdam, Germany. This award is given annually to honor and encourage outstanding professional contributions to the science and chemical technology of cellulose and its allied products. It is the most prestigious and internationally recognized award in the field of cellulose research. Prof. Fink is the first recipient of this award coming from any of the Fraunhofer Institutes. These Institutes, mostly located in Germany constitute the largest applied research organization in Europe.

Cellulose is a fascinating biopolymer and the most abundant renewable resource - not only in the wooded Germany, but also on the entire earth. Due to its structure, this sustainable chemical raw material is not meltable and insoluble in common solvents. Compared to meltable petroleum-based commodity polymers, the chemical-technical processing of cellulose into fibers, films and plastics is a special scientific and industrial challenge that Prof. Hans-Peter Fink has been meeting for over 20 years.

Early in his research career, Hans-Peter Fink examined structural changes and structure-property relationships of cellulose in chemical engineering processes. Later, together with his colleagues at Fraunhofer IAP he developed new processing methods and cellulose products up to pilot plant scale. These include processing principles for the production of fibers, blown films and meltblown nonwovens which were developed in close cooperation with industry. Starting from the basic idea of a melt-like processing of cellulose, Fink and his colleagues succeeded last to develop novel and environmentally friendly methods for producing high-strength industrial cellulose fibers.

At the ACS Spring Meeting in New Orleans on April 7 to 11, 2013 Professor Fink be recognized with the Award. In addition an award symposium „Chemical processing of cellulose - from raw materials to novel applications" will be organized to highlight and honor the contributions of Prof. Fink to this important field of cellulose research.

Prof. Dr. Hans-Peter Fink, Director of Fraunhofer Institute for Applied Polymer Research IAP in Potsdam-Golm, © Fraunhofer IAP, Foto: Manuela Zydor

EPNOE Member's award

Antje Potthast was awarded with the 2012 Hayashi Jisuke International Cellulose Award

During the 3rd International Cellulose Conference in Sapporo, Japan, Antje Potthast received the Hayashi Jisuke International Cellulose Award for her «contribution to cellulose analytics, in particular profiling of oxidized groups in polysaccharides, and its applications to conservation science.»

The awardee is Assoc.Prof. at BOKU University Vienna, Department of Chemistry. At the Division of Chemistry of Renewables, she is heading the «Biopolymer analytics» research group, and is co-director of the Christian Doppler laboratory «Advanced cellulose chemistry and analytics».

Besides the Anselme Payen Award of the American Chemical Society, the Hayashi Yisuke International Cellulose Award is the most prestigious scientific prize in the field of cellulose science. It is awarded every five years by the Japanese Cellulose Society on recommendation of an international scientific committee. The award ceremony was part of the 3rd International Cellulose Conference" (ICC2012) in Sapporo, Japan, attended by more than 200 international experts in the field of cellulose, wood and renewables´ science. The award was won for the second time by a scientist from BOKU Vienna, Division of Chemistry of Renewables (2007, T. Rosenau).

The 2012 Hayashi Jisuke award recognizes Antje Potthast´s fundamental research in the field of polysaccharide analytics, in particular that of celluloses and pulps, as well as the application of these methods in conservational practice. In her studies, multi-detector gel permeation chromatography (GPC) is combined with group-selective fluorescence labeling. The «CCOA method» for carbonyl detection and the corresponding «FDAM method» for carboxyl detection have been introduced several years ago and have been extensively validated and used in diverse studies. Nowadays, the two analytical methods are commonly recognized as standards of in-depth cellulose characterization. In contrast to conventional sum parameters, the two approaches provide the carbonyl/carboxyl content relative to the molecular weight distribution, i.e. profiles of the functional groups, so that dynamic changes in carbonyls/carboxyls in response to almost any chemical alteration, such as chemical treatments, processing steps, bleaching, aging etc., can be monitored in detail at the molecular level. They are particularly valuable with regard to damage assessment of historic cellulosic objects, and to evaluate resulting conservation treatment options.

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EPNOE Member's event

Fraunhofer IAP inaugurated Application Center and celebrates 20th anniversary

On June 12, 2012, the Fraunhofer Institute for Applied Polymer Research IAP celebrated its 20th anniversary and inaugurated the "Application center for Innovative Polymer Technologies" where new materials and technologies will be transferred from laboratory to the near-industrial scale. High-tech polymers with special electrical and optical properties, biocompatible materials and renewable plant raw materials are the focus of the research activities.

The Application Center before its inauguration.

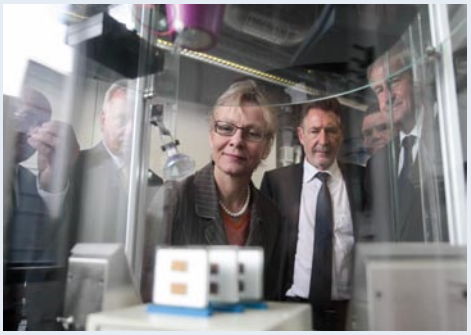
"The step from laboratory to industrial production is great," said director Professor Hans-Peter Fink at the opening. Hence, the Application Center constitutes an evolutionary leap for the institute, Prof. Fink emphasized. He thanked for the support, notably EU, the Fraunhofer-Gesellschaft, the BMBF and the state of Brandenburg.

Opening ceremony with H.-P. Hiepe, Prof. H.-P. Fink, Prof. S. Kunst, Prof. U. Buller, Ch. Nagel-Hirschauer, J. Jakobs (from left).

"The Fraunhofer IAP is celebrating not only 20 years of polymer research, but also 20 years enthusiastic and hard-working employees" said Professor Buller, who headed the institute from 1997 to 2006 and is since 2006 the Fraunhofer board for research planning. "A hallmark of the Fraunhofer-Gesellschaft is the applied and industrial research. We cooperate with partners all over the world, and increasingly in Brandenburg", Fink supplemented.

Following the speeches and the opening ceremony with Minister Kunst, the guests were able to gain an impression of the facilities and research topics of the application center. Division Head Dr. Armin Wedel presented a pilot plant for the production of organic light-emitting diodes and organic solar cells, including flexible systems. Department Head Dr. Joachim Storsberg develops biocompatible materials for implants. In particular, artificial corneas of the second generation are the focus of his work. On another ground, researchers will make by-products of agriculture - such as oat hulls or wheat bran - usable for the manufacture of plastics. „This could make the plastics industry more independent from crude oil," said scientist David Dietz.

A lecture meeting in the afternoon completed the festivities. Renowned Fraunhofer researchers spoke about the highlights from 20 years of polymer research, after Prof. Gerhard Kossmehl which was Deputy Chairman of Trustees for many years, reviewed the story of the Fraunhofer IAP.



VIP tour through the Application Center: Minister Prof. Kunst inspects organic light diodes.

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EPNOE Member's event

60 years of scientific and research activity of the Institute of Biopolymers and Chemical Fibres 1952-2012

On 28th of September 2012, the Institute of Biopolymers and Chemical Fibres of Lodz celebrated its 60th anniversary of scientific and research activity. Numerous guests participated in the ceremony including present and retired employees of the Institute.

Director Danuta Ciechanka and Dr Dieter Hofman from Fraunhofer institute, Germany.

On the stage of the "New Theatre" in Lodz, in which the ceremony took place, outfits that had been made of fabrics produced in the Institute within research projects financed from Innovative Economy Operational Programme (2007-2013) were exhibited. These clothes were the symbols of the success of the following projects:

- "Biodegradable fibre products" – BIOGRATEX,
- "Technology of preparation of biodegradable polyesters from renewable resources" – BIOPOL,
- "Biomass application for production of environmentally friendly polymer materials" – BIOMASA,
- "Modern technologies for textile industry. Chance for Poland" – FORESIGHT.

Patrick Navard delivering a speech in the honour of the Institute, on behalf of EPNOE

The ceremony of the 60th anniversary of the Institute of Biopolymers and Chemical Fibres was hosted by PhD Eng. Danuta Ciechanska, the Director of the Institute and the local television presenter Przemyslaw Lisiecki who welcomed all guests on behalf of the organizers: the Director of the Institute – PhD Eng. Danuta Ciechanska, and the Head of the Scientific Council of the Institute – Professor Eng. Izabella Krucinska.

Afterwards, the Director of the Institute welcomed all invited guests, representatives of the Ministry of Economy, authorities of the city of Lodz, authorities of universities and Polish Academy of Sciences, representatives of scientific and industry units, and also foreign guests with whom the Institute is bound together not only professionally but also by cooperation and friendship.

The Director of the Institute gave then a presentation on the last 60 years of scientific activity of the Institute of Biopolymers and Chemical Fibres before presentations given by invited guests.

At the end of the ceremony, all guests and employees of the Institute were offered a piece of anniversary cake to celebrate this important event for our institute.

A large audience attended the ceremony.