

Novel nanostructured polymeric materials for food packaging and beyond

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International Workshop

Book of Abstracts

Edited by
Mika Vähä-Nissi

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Under the auspices of **COST Action FA0904:**
Eco-sustainable Food Packaging
Based on Polymer Nanomaterials



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Preface

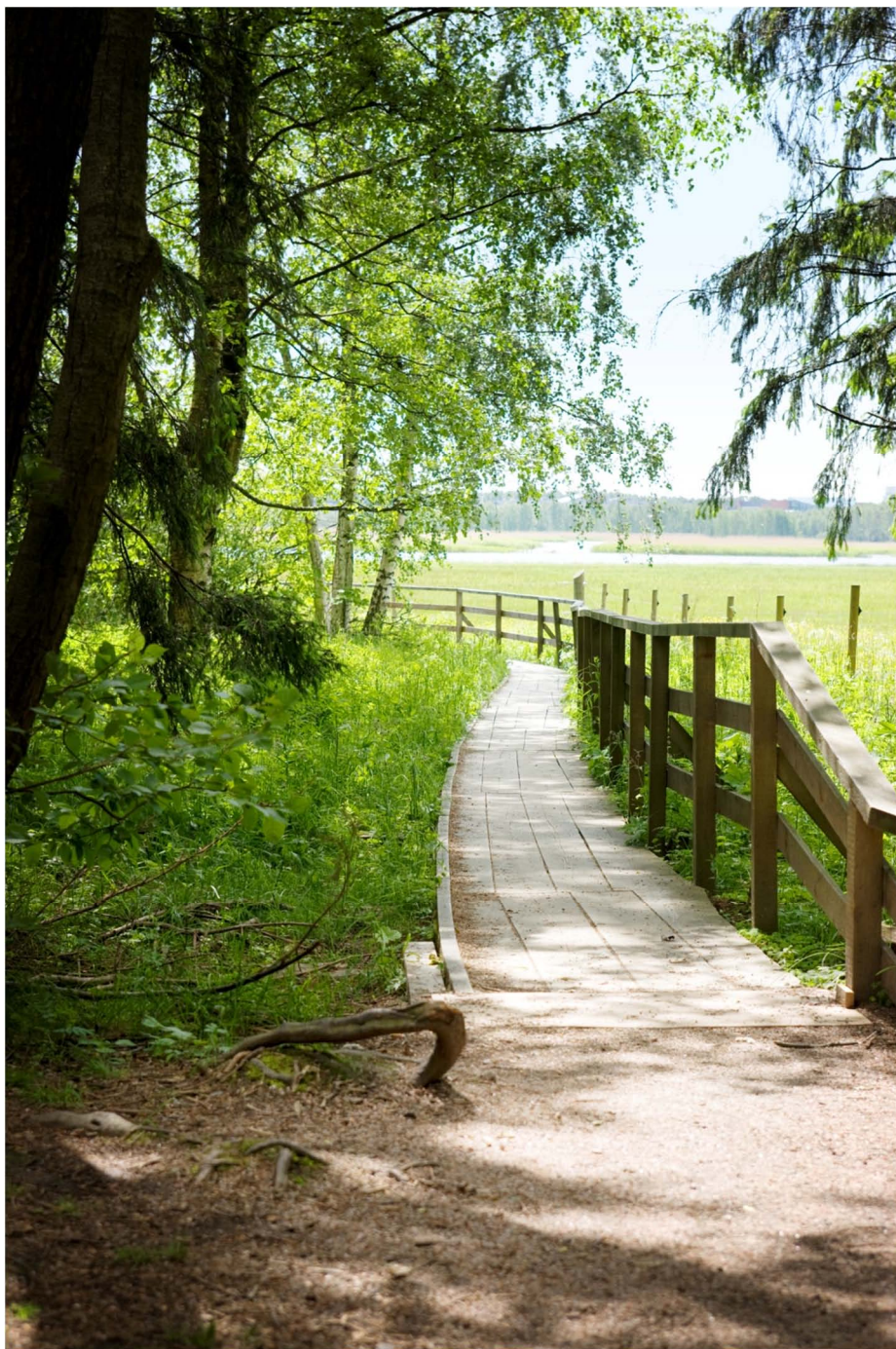
As part of COST Action FA0904 “Eco-sustainable food packaging based on polymer nanomaterials” an **International Workshop “Novel nanostructured polymeric materials for food packaging and beyond”** is organized related to Working Group 1 of this Action. The main goal of WG 1 (Development of new safe PNFP) is to consider new polymer nanomaterials for food packaging – also for other applications – and breakthroughs and knowledge leading to new features gained through nanotechnology. The presentations cover the following topics:

- Selection, synthesis, production of matrix, nanoparticles and -devices
- Surface functionalization and chemical treatments of nanoparticles
- Link between nanostructures and macroscopic properties
- Characterization and measurement tools.

COST Action FA0904 is comprised of an international scientific and technology network focused on issues related to Eco-sustainable Polymer Nanomaterials for Food Packaging (PNFP), for the preservation, conservation and distribution of high quality and safe food. The Action aims at exploiting the potentiality of polymer nanotechnology in the area of food packaging treating in a complete way the demanding needs of the users, such as health, environment, taste, cost and the specific requirements of the food industry. The envisioned direction is to look at the complete life cycle of the PNFP by the combined efforts of leading research and industrial groups. The Action will identify the barriers (in research and technology, safety, standardization, trained workforce and technology transfer) that prevent the successful development of PNFP and will indicate the strategies to proceed further. In this Action there are 25 COST countries and 2 non COST institutions involved. The action is organized into four working groups.

Further information:

www.cost.esf.org, www.ictp.cnr.it
Dr. Clara Silvestre, ICTP-CNR, Chair of FA0904
Dr. Mika Vähä-Nissi, VTT, Head of WG 1



Nature trail in Espoo

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Abstracts of Presentations September 15

Welcome and FA0904 in short

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ABSTRACT

Nanotechnology can give impetus to industries no longer competitive, as well as cultivate totally new industries. In spite of the potential, most examples of nanotechnology in packaging in specific can be found outside of Europe. To enhance Europe's competitiveness, top level research and development work is needed. We have to overcome various barriers by providing and generating information on nanotechnology, and by verifying that various claims are justified. Public trust and acceptance are the factors determining the success of nanotechnology in packaging. Our goal is to promote awareness among the public and authorities on the safe use of nanomaterials for food packaging. European Commission published January 2011 a new regulation on food contact plastic materials. New technologies that engineer substances in particle size that exhibit properties differing from those at a larger scale should be assessed on a case-by-case basis as regards their risk until more is known.

This Action aims at exploiting the potential of polymer nanotechnology in the area of food packaging treating (PNFP) in a complete way the needs of the users, such as health, environment, taste, cost and the specific requirements of the industry. The idea is to look at the life cycle of the PNFP by the combined efforts of research and industrial groups. The Action will identify the barriers that prevent the successful development of PNFP and will indicate the strategies to proceed further. The goal of WG 1 is to consider new polymer nanomaterials for food packaging, breakthroughs and knowledge likely leading to success. This workshop helps indicating the areas of nanotechnology with significant activities.

This Action requires also an interdisciplinary approach. Therefore, the Otaniemi Campus with a community of over 32.000 people and featuring a mix of research organizations, academic institutions and companies from start-ups to multinational corporations is an appropriate location for this workshop. With this, also on behalf of VTT, I would like to welcome You all to this workshop.

Keynote Speech – Novel nanomaterials promise to food packaging

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ABSTRACT

Packaging valuable part of food value chain. Continuous development in populations require us focus on food availability and its quality. World population has recently increased to level of 7.3 billion and is growing. Simultaneously demographic changes and life-styles in the industrial countries are creating new customer needs. The growing consumption markets have not reduced importance of the food sector, but when global economics and logistics enables both widening the offering and balancing the availability anywhere, we need efficient and sustainable packaging solutions.

Efficiency has been always the major requirements for packaging, which comes often to issues on minimum use of material. Beyond that, we should see packaging as a part of delivery chain, where it should support agile and energy efficient logistics. Even further it is vitally important to reduce losses in the delivery chain like in transportation or homes, and provide light handling robust solutions and packaging sizes fitting together with modern family structures.

Sustainability in packaging is typically seen nearly equivalent to waste management problems from littering to filling the dumping sites. Reduction of waste combustion has been understandable, but more recently energy recovery has opened the discussion again. Biodegradability has been seen solution especially for oceanization and earth plastic waste loading, but simultaneously is promoted disposables. Generally recyclability has become a markedly pronounced and requirements to enable recyclability with natural and bio-based materials.

Increasing society and consumer requirement of sustainable packaging has been understood among retailers and brand-owners. The limitations of natural and bio-based materials are typically in their barrier properties and weight, and they fail in several applications competition with fossil materials, as an example flexible and liquid packaging.

Emerging nanotechnologies combined with polymeric materials in fiber and bio-plastic packaging is promising several answers linking nanostructures and

macroscopic properties. Nanotechnologies based on synthesis, production of matrix, nanoparticles and –devices as well surface functionalization and chemical treatments of nanoparticles provide especially but not limiting on lighter constructions and higher barriers.

Clearly can be seen, that enabling nano- and polymer technologies in packaging provide marked potential to improve both efficiency and sustainability, where also nano-safety is focused while developing the novel materials. With the novel technologies we are answering the challenges of food value chain on 21st century.

Sustainable packaging; role of nanotechnology

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ABSTRACT

Packaging is an absolute necessity especially in the modern world. It is a short cycle product used in combination with other products, aiming at delivering protection, safety, convenience, efficiency, identification, as well as improved marketing.

With the outlook on potential growth of packaging materials market around the world, packaging materials, methods to obtain packaging, as well as processes, require an evolution. This evolution should involve new strategies and approaches. The objective is to improve further in terms of functionality, minimize the negative impact on environment and energy consumption, while the positive impact on society security of supply and economy is maximized. This evolution will also bring higher added value and new products along. The focus will also be very much on renewable raw materials. Despite some drawbacks that are associated with these materials such as limitations in properties (barrier, mechanical), costs, these materials are expected to grow in packaging applications along with new technologies for improvement.

Using nanotechnology is potentially a very interesting approach to obtain new packaging materials especially based on renewable raw materials with improved mechanical and barrier properties. Furthermore functionalities such as antimicrobial properties, delivery of beneficial compounds and nano-sensors for traceability and monitoring can be made possible. Surely the implementation of the solutions including nanotechnology will depend on the elimination of possible risks and maximized achievable benefits.

This lecture will discuss the possible added value of nanotechnology in sustainable packaging approaches. Examples are discussed in terms of nanotechnology based products in the packaging market as well as relevant forecasts.

Novel polymer nanomaterials for food packaging based on modified cellulose nanofibres

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ABSTRACT

Nanocomposite materials based on polymers and various types of nanoparticles or -fibres have gained much interest lately as packaging materials, coatings, wound healing bandages, etc. Microfibrillated cellulose (MFC) with a thickness of 1–50 nm and a length of several μm (regarded as a nanomaterial) has been surface modified using its large number of hydroxyl groups. A wide variety of new functionalities have been introduced, both reactive groups as epoxy, amines, succinic and maleic acid for covalent introduction to matrix materials and hydrophobic compounds such as octadecylamine and poly(styrene-co-maleic anhydride) for enhanced compatibility with the matrix. These modified MFC may be added to various different commonly used polymers in food packaging, e.g. PE, PP, PS, or paper, both to bulk materials and films to introduce new and improved properties. The strength of base paper increased significantly when coated with a layer of less than 10 % of MFC, whether pure or surface modified, and the air permeability decreased dramatically.

Special focus will be given to antimicrobial MFC materials for use in food packaging. The challenge with antimicrobial packaging materials is often possible leakage to the food and/or fast wash-out with only temporary effect. Quaternary C18 ammonium ions were covalently attached to MFC. These functional groups disrupt the cell membrane of the bacterial cells through physical and ionic phenomena and will kill bacteria attached to the packaging material. These modified MFC are promising candidates as fillers for novel food packaging nanocomposite materials, combining antimicrobial activity with increased strength and decreased permeability due to fibre reinforcement.

Health and environmental safety aspects of nanofibrillated cellulose

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ABSTRACT

Nanomaterials will improve the performance of many products in future but at the same time they exhibit novel properties and may expose humans and environment to new risks. One of the most abundant, sustainable and promising nanomaterial for forest sector is nanofibrillated cellulose (NFC). NFC is produced by grinding the cellulose fibres under high compression and shear forces. NFC forms an opaque and stable, well-dispersed, aqueous suspension consisting of long and curly fibrils with a high aspect ratio.

Wood pulp and powdered celluloses are generally recognized as safe and can be used for example as a raw material for food contact materials or even as food additives. However, as with other nanomaterials, the biological effects of nanocelluloses cannot be predicted solely from the chemical nature of cellulose. The size, shape, aggregation properties, degree of branching and specific surface properties, among others, still poorly understood factors, may affect the interactions of cellulose nanofibers with cells and living organisms.

In this study we evaluated the health, environmental and occupational safety aspects of nanofibrillated cellulose. Processing of NFC with either a friction grinder or a spray dryer did not cause significant exposure to particles during normal operation. Grinding generated small amount of particles, which were mostly removed by fume hood. Spray dryer leaked particles when duct valve was closed, but when correctly operated the exposure to particles was low or nonexistent. The toxicological tests with several human and mouse cell cultures did not indicate any cyto- or genotoxic properties. Studied NFC samples were not acutely ecotoxic in kinetic luminescent bacteria test (*Vibrio fischeri*) with studied concentration up to 0.5 % NCF.

New approach to classification of cellulose fibrils and suitable methods for their characterization

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ABSTRACT

Nowadays the manufacturing of cellulose micro- and nanofibrils has been significantly developed and products with tailored properties can now be introduced. Different fibrillation methods and chemical treatments generate cellulose fibrils with varied dimensions, branching degree and chemical properties. It would be beneficial to classify the different kind of cellulose fibrils according to their properties with consensual agreement. We present here a classification based on the morphological and chemical properties of cellulose fibrils. We discuss the applicability of classification and different characterization methods for certain grades (from nanowhiskers and nanofibres to nano fibrillated materials with branched or ribbon-like structures) and for different applications.

Characterization of cellulose fibrils is challenging not only because of the wide size distribution and high aspect ratio but also due to the wide variety of morphological properties and the complex structure of single fibrils. This paper introduces a set of simple methods which gives the basic characteristics of nanocelluloses.

Five commercially available cellulose nano/microfibrils and three self-made samples produced by and birch pulp ground to various levels using supermass-colloider equipment were analysed in detail. Different rheo- and viscometers were tested and a viscosity measurement sensitive towards high aspect ratio fibrils is introduced. The transmittance measurement gives an estimation of the amount of large particles and the optical microscopy effectively detects the amount of unfibrillated material. SEM measurements can be used to analyze the fine structure nanofibers. The aspect ratio, degree of fibrillation, fibril size and the amount of unfibrillated material can be evaluated with a combination of these indirect measurements, which provide a basic set for quality assurance.

Solid board as a base material for novel sustainable packaging applications

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ABSTRACT

Solid board is a typical Dutch paper and board industry product. In 2009, 25 percent (649.000 tons) of all paper and board produced in The Netherlands was solid board. It is a versatile material as it combines properties of both folding cartons and corrugated packaging and thus offers possibilities to be used in a wide variety of, often demanding, applications. Some of those applications are for instance, book covers, board games such as puzzles, luxury boxes, displays, and food/retail packaging (even for fresh products and long term deep frozen applications). Solid board is also often used in packaging applications where it needs to have waterproof or fat repellent properties. This is done by coating the solid board with materials such as PE or PP, or by application of specialty surface papers.

This opens up opportunities for making novel combinations with solid board as base material and various newly developed materials in forms of coating, laminates or filler being applied for achieving improved or new properties. These could be nanomaterials or biobased polymers as well, which makes it very interesting to look into new potential combinations for development of sustainable packaging, both for food and other packaging applications. As solid board is biobased, renewable and reusable, our objective is to use this paper based material in combination with recent developments in surface treatments. This will enhance the (barrier) properties and makes it competitive to PE or PP coated substrates which are not biobased or reusable.

Starch-clay paper coatings for sustainable, flexible packaging

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ABSTRACT

Clays have been used in traditional paper coating technologies for many years but the focus has been on non-swelling clays because these exert the least effect on the rheological properties of a formulated coating. However, work in the SUSTAINPACK and FLEXPARENEW projects has demonstrated that combining swelling clays, particularly smectites, with starch and plasticizers, to form a sustainable coating on paper, results in water vapour transmission rates (WVTR) that are competitive with oil-derived barrier coatings. The starch-clay-plasticizer matrix offers the barrier properties, whereas the paper provides the mechanical strength. Typically, WVTR values of $10 \text{ g (m}^2 \text{ day)}^{-1}$ are obtained when exposed to 50 % relative humidity at 23 °C.

Achieving the low WVTR value required strongly depends on the type of clay and plasticizer used in the formulation and thus the need to understand their interactions when combined with starch in the final coating is important. The plasticizer is also a critical ingredient needed to overcome the inherent brittleness of the starch which becomes a major factor when the coated paper is bent or creased.

Characterisation of samples, prepared from an extended component/concentration matrix, using x-ray diffraction has revealed a relationship between a successful coating and how the expansion of a specific clay is influenced by the type and concentration of plasticizer and/or starch.

Nanostructured materials based on electrospinning of interest in food biopackaging applications

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ABSTRACT

Looking genuinely at nature, nanofibers often serve as a basic platform where either organic or inorganic components are built upon. For instance, cellulose nanofibers would represent the building block in plants while collagen nanofibers in the animal body. The fiber structure exhibits, from a structural point of view, the certain ability to transmit forces along its length and, thus, reducing the amount of materials required. While strong enough for their designed purpose, nanofibers have the added advantage of giving high porosity to the natural supports which allows faster diffusion of chemicals to the inner structure. To follow this extraordinary nature's design, a process that is able to fabricate fiber nanostructures from a variety of materials and mixtures is an indispensable prerequisite. Control of the nanofibers arrangement is also necessary to optimize such structural requirements.

Electrospinning is a physical process used for the formation of ultrathin fibers by subjecting a polymer solution to high electric fields. At a critical high voltage (5–25 kV), the polymer solution droplet at the tip of the needle distorts and forms a Taylor cone to be ejected as a charged polymer jet. This stretches and is accelerated by the electrical field towards a grounded and oppositely-charged collector. As the electrospun jet travels through the electrical field, the solvent completely evaporates while the entanglements of the polymer chains prevent it from breaking up. This results in the ultrathin polymer fibers deposition on a metallic collector to habitually assemble the fibers as non-woven mats.

Since the electrospinning is a continuous process, fibers when winded can be as long as several metres or even kilometres. The formed fibers are not only ultrathin and relatively large in length but also fully inter-connected to form a three-dimensional network.

The current paper will highlight some recent advances carried out within our research group in which various nanostructured materials based on biopolymers

and biopolymeric blends will be reviewed (1–10). These include examples in which new antimicrobial nanostructured fiber mats with strong biocide efficiency were successfully developed, as well as nanostructured systems to reinforcing the barrier properties of biopolyesters and also novel nanoencapsulation of active and bioactive food ingredients for food bioactive packaging applications.

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Changes in water vapour uptake ability of xylan derivatives monitored by QCM-D humidity module

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ABSTRACT

In surface science, model surfaces of small amounts of chemically defined compound are deposited on a flat surface by means of e.g. spincoating technique. The model surface approach enables the investigations of the direct surface interactions on the molecular level since it enables efficient and well-established use of the surface analytical techniques such as quartz crystal microbalance with dissipation monitoring (QCM-D) and atomic force microscopy (AFM).

The resistance to humidity, swelling behaviour and the ability to uptake water vapour are features which are strongly related to the behaviour wood and agro-derived polymers in different applications such as in film and membranes. These features have a direct link also to the mechanical and thermal properties and therefore they need to be fully understood and controlled when the aim is to prepare biobased materials which are effectively functioning also at higher humidities.

In this work the changes in the water vapour uptake and swelling behaviour of the xylan derivatives were studied using the model film approach. Model films were prepared using the different crosslinking xylan derivatives. The water vapour uptake of the thin xylan model films was monitored using QCM-D humidity module. With the QCM-D, the mass uptake due to the water vapour binding was defined. Simultaneously the changes in the viscoelastic properties of the crosslinking xylan when subjected to different relative humidity conditions were determined. Clear changes in the water uptake ability were taking place before and after crosslinking of the films.

Abstracts of Posters September 15

The technology of antibacterial food packaging materials production

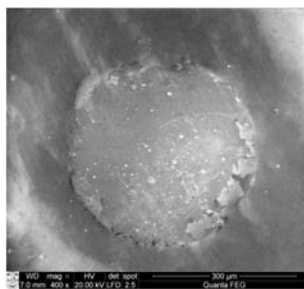
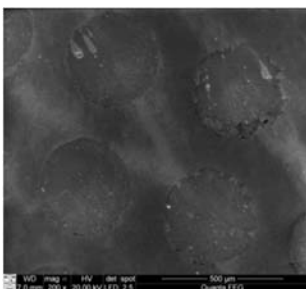
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ABSTRACT

The main objective of this work was to explore the ways to obtain new active environmentally friendly nanomaterials, their compositions and convenient technological approaches for food packaging materials production.

The production technology developed at the Biopolymer research laboratory (KTU) and implemented at the company “Panoden” is based on applying of antibacterial coating film on flexible packaging material by using flexographic printing. The coating composition can be comprised from inorganic or organic antibacterial agent and food contact approved film-forming material. The different amount of the aqueous coating composition was applied on the packaging films (OPP and thermo-stretch films) by varying the density and coverage area of the coating. The antibacterial properties of the produced packaging materials were assessed. Prepared samples possessed an excellent antibacterial activity against the tested organisms. The developed flexible packaging materials could be used for protection of food against bacterial contamination and growth. The subsequent investigation is concerned with the replacement of inorganic particles by biopolymer e.g. starch nanoparticles which can be modified by attaching the active agents and different properties to the food packaging materials can be imparted.



SEM images of antibacterial coating on OPP film. The flexographic printing dots observed in the micrographs: left – magnification $\times 200$; right – magnification $\times 400$.

On the structure and exploitation properties of biodegradable and synthetic polymers based nanocomposites with layered silicate nanofillers

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ABSTRACT

Layered silicates are perspective modifiers of polymer based nanocomposites in respect to their elastic, thermal and barrier properties. It is well known that addition of nanolevel modifiers allows increase these exploitation properties more efficiently in comparison to traditional microcomposites. Increment of exploitation properties of polymer based nanocomposites largely depends on the type of silicate modifier used, compliance of polymer matrix and particularly compatibility between the ingredients of the nanocomposite. Choice of ingredients of the nanocomposite, designed for packaging applications, from the viewpoint of sustainable development is largely determined also by its environmental friendliness and safety aspects.

Considering the above mentioned in the current report the effects of nanolevel clay modifiers on the structure and properties of polymer matrix nanocomposites are summarized. Two types of popular, but principally diverse, packaging materials – polyethylene (PE) and modified starch (MS) – are compared as matrices for nanocomposites. Three types of clays – bentonite (B), self-modified bentonite (SMB) and commercial organically modified clays (COMC) – are used as nanofillers. Content of the nanofiller in the chosen polymer matrix is varied in the interval from 2 to 12 wt. %.

Most important results of the investigation testify that modification of the bentonite is successfully carried out as testified by the shift of 2θ angles to smaller values. It is observed that nanocomposites with higher compatibility between clay reinforcement and polymer matrix possess higher elastic properties, improved barrier characteristics and higher thermal resistance. Besides it is observed that the effect of SMB on the properties of polymer nanocomposites is comparable to that of COMC.

Abstracts of Presentations September 16

Antimicrobial surface modification of polyethylene

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ABSTRACT

Polyethylene films were surface modified by corona discharge. LDPE films pretreated in corona and further impregnated/coated/grafted with chitosan were analyzed by polyelectrolyte titration, potentiometric titration, zeta potential, ATR-FTIR spectroscopy and XPS and compared with untreated films.

The potentiometric titration was used for direct determination of the amino groups onto the LDPE surface. The potentiometric titration results for the LDPE reference show zero charge of the sample, the corona pretreated LDPE and further covered with chitosan present an increase of the positive charge on the surface because of the immobilization of the chitosan onto the surface, results revealed even by ATR-FTIR spectroscopy and XPS studies. In the case when the corona treated LDPE surface was activated with a solution of EDC and NHS the amount of the chitosan adsorbed onto the surface was less but the ATR-IR spectra revealed the formation of the new amide bond between chitosan and LDPE functionalized surface by corona discharge plasma.

Surfaces were tested for chitosan retention and release, as well as antimicrobial activity against microorganisms relevant to food quality and food safety, including *Escherichia coli*, *Listeria monocytogenes* and *Salmonella*.

The modified films exhibited antimicrobial activity and reduced microbial growth suggesting the potential for biocidal action. Such antimicrobial surfaces could supplement existing cleaning and sanitation programs in food processing environments to reduce the adhesion, growth, and subsequent cross-contamination of food pathogens, as well as food spoilage organisms.

Atmospheric plasma technology for surface modification of nanoparticles

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ABSTRACT

VITO has a broad experience in dry surface modification and coating of different types of materials by means of low temperature atmospheric plasma treatment. Dedicated equipment is available for process and product development from lab to pilot-scale. So far, developments have been mainly oriented towards roll-to-roll treatment of foils and fibers and in-line treatment of 3D parts and components. However, recently two dedicated prototype systems for atmospheric pressure plasma functionalisation and coating of micron- and nano-sized particles have been established. In addition, specified equipment for analysis and evaluation of treatments has been installed. Some examples of powder treatments will be discussed as well as ongoing developments for continuous nanopowder treatment equipment.

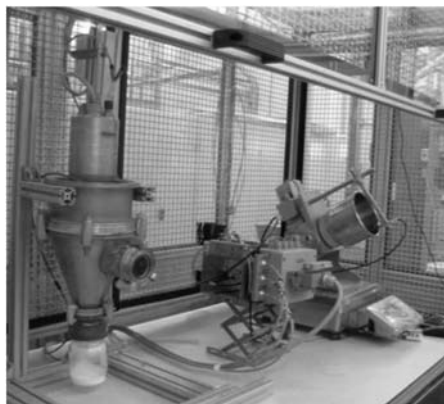


Figure: VITO lab setup for plasma treatment of nanoparticles.

The plasma technology team at VITO is well experienced in the coordination and participation in both industrial and EU research projects related to innovative surface engineering e.g. integrated projects FP6-Kristal, FP6-Foremost, FP7-Nano2Production, FP7-PlasmaNice.

Nanoscale surface processing of extrusion coated substrates and plastic films with atmospheric plasma activation and deposition

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ABSTRACT

PlasmaNice – Atmospheric Plasmas for Nanoscale Industrial Surface Processing – is a four-year project funded by the European Union 7th RTD Framework Programme in the context of the NMP – Nanosciences, Nanotechnologies, Materials and new Production Technologies. The project focuses on studying the utilisation of atmospheric plasma techniques for packaging applications. The main objective is to develop sustainable packaging materials and equipment for on-line atmospheric plasma deposition of functional nanoscale coatings on various fiber- and polymer-based substrates. The project aims to combine and integrate state-of-the-art in atmospheric plasma technology, sol-gel chemistry and extrusion coating of bio-based and/or biodegradable materials.

Atmospheric plasma techniques as processing methods have a number of advantages which include for example a possibility to tailor the surface chemistry at the nanometer level. As such, the plasma treatments are energy efficient, reproducible and environmentally clean. In surface modification of materials, plasmas can be used e.g. to activation, cleaning, etching and coating. In plasma activation functional chemical groups are created to the surface of the treated material. For packaging materials plasma activation can be used to enhance adhesion properties, wettability and printability of surfaces. In plasma deposition, a completely new surface is created which enables the possibility to create barrier coatings from precursor such as e.g. sol-gels chemistry.

In the PlasmaNice project, non-thermal atmospheric pressure plasmas will be used for altering the surface properties of fiber- and polymer-based substrates. The atmospheric plasma devices used in the project are based on the dielectric barrier discharge (DBD) reactors, which enable low temperature plasma treatment which is suitable even for heat-sensitive materials such as polymer films. Dielectric barrier can also be scaled up to larger dimensions for on-line processes

such as paper converting at low costs compared to vacuum plasma processes. This project aims to transfer atmospheric plasma processing technology from the laboratory scale to industrial level in the packaging industry. Special attention will go out to the very promising combination with sol-gel technology. A method and equipment for in-line plasma deposition of high-barrier bio-based coatings to be applied in conjunction with extrusion coating at industrial line speeds will be developed. Furthermore, an important part of PlasmaNice project is also to study the health and safety aspects of nanotechnology in plasma-assisted processes. LCA (Life Cycle Assessment) and RA (Risk Assessment) will be used in the project to evaluate the safety aspects and environmental performance of the new products and processes.

To achieve these objectives, several leading European institutes and universities in atmospheric plasma deposition technology (VITO and TUE), sol-gel development (FhG-ISC and VTT), extrusion coating (TUT), analytics development (JSI) and life cycle and safety analysis (DTU and 2B) together with a range of industrial participants are incorporated in the project.

Atomic layer deposition in food packaging and barrier coatings

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ABSTRACT

Atomic layer deposition is coating method for preparation of thin films down to nanometer scale. It is based on sequentially used self-terminating solid-gas reactions. Control of coating properties and good conformality can be obtained, since substrate is first subjected to one reactant, followed by purge and then same with another reactant. Thus, uncontrolled reactions between reactants in gas phase can be prevented. In certain cases second reactant can be replaced by activation treatment of surface, when only one reactant is used. Typical coating materials are ceramics. Metal oxides, for example, can be prepared using H_2O , O_2 , O_3 , alcohols or oxygen plasma as oxygen source and nitrides using typically ammonia NH_3 or N_2/NH_3 plasma as nitrogen source. Requirements for reactants include volatility (RT or elevated), they should not decompose at ALD processing temperatures, and their reactions should self-terminate. ALD temperatures vary from around 50 °C to 500 °C. Low temperature ALD enables coating of temperature sensitive materials such as polymers.

ALD coating layers are typically well organized and pin hole free. They can be used as barrier materials when applied on substrates including synthetic polymer films and paper boards. Properties of the initial layer depend on the surface properties, chemistry and roughness. Sometimes interface between polymer and ceramic coating can be challenging. For example, lack of functional groups of the functional groups of polymer surface may cause poor bonding. Typically ALD barrier layers are thin, in a range few tens of nm. Thicker layers are in risk of cracking and thus lowered barrier properties.

In this presentation use of ALD in food packaging and barrier coatings are reviewed. Production issues and properties of such ALD based materials are discussed, and compared with other barrier materials.

Characterization of nanoparticles in polymers and their impact on the morphology for films for food packing

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ABSTRACT

Polymer films containing nanoparticles have considerable potential for use in safe food packaging either in terms of controlling the gas permeability or providing additional functionality such as anti-microbial properties. In general the properties of a polymer film such as polypropylene are highly dependent on the morphology of the final product. This in turn will depend on both the polymer matrix and the nature of the nano-particulates and on the processing used to fabricate the film. In this contribution we provide a quantitative view of isotactic polypropylene containing either inorganic nano-platelets to increase the tortuosity of the gas paths or spherical particles which provide anti-microbial properties. In each case we have designed and implemented special experimental apparatus which allows us to evaluate in-situ the morphology which develops during a representative processing cycle. In these in-situ experiments we are able to reproduce the complexities of the material interactions. We have exploited time-resolving small angle and wide-angle x-ray scattering techniques which allow us to follow both the nanoparticles and the structure of the polymer matrix. The use of quantitative scattering techniques has been complemented through the use of scanning and transmission electron microscopy and where appropriate scanning probe microscopy. We reveal that in certain combinations of processing parameters and nanoparticles, the presence of the nanoparticles has a strong impact on the semi-crystalline morphology of the polymer matrix. Equally in certain situations the nanoparticles become oriented in particular directions as a consequence of the shape and interaction of the nanoparticulate with the polymer and processing field. We have explored this latter effect to provide a three dimensional view of the nanoparticles and the polymer morphology. Using these studies we have identified the minimum of nanoparticles required to influence the polymer matrix and the range of processing conditions. Part of the response of the nanoparticles is independent of their shape and part of the response can be directly attributed to whether they are plate-like or spheroidal.

Mechanical, thermal and morphological properties of Polylactide/Polyhydroxybutyrate/ nanocomposite blends

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ABSTRACT

Polylactide (PLA) and Polyhydroxybutyrate (PHB) degradable composite blends were prepared via melt blending on a Brabender Plastograph. Blends with various weight percentages of PLA and PHB were prepared with the addition of nanoclays with various surfactant structures and the resulting systems were analysed for morphological, thermal and mechanical properties.

The blends were characterized by Differential Scanning Calorimetry (DSC), Thermo gravimetric Analysis (TGA), Dynamic Mechanical Analysis (DMA), Tensile properties and Scanning Electron Microscopy (SEM).

Various blend and nanoclay concentrations resulted in improved mechanical and thermal properties.

Properties of PLA based micro- and nanocomposites

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ABSTRACT

Being aware of growing environmental consciousness and environment protection standards becoming more and more demanding as well, it is clear that polymer industry has to accelerate on their way to biocompatible materials manufacturing. Hence, polyolefin based systems are increasingly substituted with so called 'green composites'. But there is still some work to be done to improve their performance. According to particular application, these might be Young modulus, strain, elongation at break, gas permeability, density, surface properties and many other. Manufacturing costs (end-user price), components availability are also of great importance.

In our study we took under consideration a composite made of semi crystalline polylactic acid (PLA) matrix and crystalline cellulose as a filler, with a filler load estimated to 5 wt.% and 30 wt.%. Three different forms of cellulose was used: 60 μm (CC60), 700 μm (CC700) and 900 μm (CC900). The results were referred to a composite filled with 5 wt.% of montmorillonite (MMT) and precipitated calcium carbonate (PCC). All materials were prepared by a melt mixing process.

Additionally, the PLA plate was covered with PTFE layer to increase its barrier properties. The permeability of PLA-PTFE system was then compared to 5 wt.% MMT and PCC composites.

To provide a multi-field characterization, the following tests were undertaken: physical (density, oxygen permeability), mechanical (bending/tensile test, resilience), thermal (DSC, DMA, heat conduction coefficient, MFR, HDT). It was verified that the fillers of different dimensions (aspect ratio) modify the composite's properties in a different way. The cellulose filler with the lowest size (CC60) was supposed to decrease the tensile strength, making the materials brittle. Contrary to CC700 and CC900, the CC60 aspect ratio is much lower, thus 60 μm filler acts in a way like particulate ones do.

Also, the tensometry is presented as a technique allowing to measure the wetting properties of loose materials generally used as a polymer fillers (fibers, granulates, powders).

Antibacterial composites based on iPP for food packaging application

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ABSTRACT

The purpose of the present work is that to investigate the effect of zinc oxide (ZnO) onto barrier, mechanical properties and antibacterial activity of isotactic polypropylene (iPP) based film for applications in food packaging sector.

ZnO powder was mixed with iPP by using a twin screw extruder by varying the composition. In order to increase the compatibility between the organic matrix and the inorganic oxide polypropylene grafted with maleic anhydride (PPgMA) was used as compatibilizer. The pure components and the samples have been characterized by DSC, TGA, WAXD and SEM.

The results indicate that the ZnO and PPgMA do not affect the thermal properties and the crystal structure of iPP. Thermogravimetric analysis shows that the presence of ZnO increases the thermal stability of iPP which increases with the content of ZnO and that it is not affected by the presence of PPgMA. SEM micrographs show a relatively good dispersion of ZnO in iPP and enhancement of the interaction between ZnO and polymer matrix with the presence of PPgMA.

Films have been obtained by compression moulding and have been exposed to UV radiation to study the effect of zinc oxide on the photodegradation of iPP. During this treatment the samples have been analyzed by FTIR. Molecular degradation of iPP has been characterized by carbonyl index (C_i). For neat iPP C_i increases much faster than that of mixtures containing ZnO. This result indicates that the zinc oxide plays an important role in the stabilization of the molecules of iPP exposed to UV irradiation.

Tensile tests have shown that the modulus increases with ZnO and the elongation and stress at rupture decrease. No variation in the yield parameters is observed.

Antibacterial activity of iPP/ZnO composites—were studied according the ASTM E 2149-01. The microorganisms used to test the antimicrobial efficiency were *Escherichia coli* DSM498. All the organisms were maintained according to good microbiological practice and checked for purity. The results show that the antibacterial activity increases by increasing the ZnO content and it is delayed by PPgMA.

In the presentation some preliminary results of ZnO migration in food simulants obtained in cooperation with the National Food Institute of Denmark (DTU) will be shown.

The work described was supported by the bilateral project CNR/CNRS (Italy-France) “Antibacterial nanocomposites based on polyolephins for food packaging”.

The effects of processing on natural fibres in starch acetate composites

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ABSTRACT

Composite compounds of triethyl citrate plasticised starch acetate and natural fibres (hemp and flax) were prepared by melt processing in kg scale. Plasticiser contents from 20 to 35 wt% and fibre contents of 10 and 40 wt% were used. The material was injection moulded to tensile test specimens. The effect of process steps, melt viscosity and fibre type on the fibre length was investigated. The lengths of fully processed fibres were determined by dissolving the starch acetate matrix and measuring the length of the remaining fibres by optical microscopy and image analysis. A clear reductive effect of the pelletising and melt processing on the fibre length was noticed. Also a reduction of fibre length along the increasing fibre content and the decreasing plasticiser content was detected. This reduction was originated from the increasing shear forces during compounding, which again depended on the increased viscosity of the material. Comparison of the fully processed hemp and flax fibres showed that hemp fibres remained longer and fibrillated more than flax fibres, leading to higher aspect ratio. Thus, the reinforcement efficiency of hemp fibres by the processing was improved, on the contrary to the reduced reinforcement efficiency of flax fibres. In addition, the analysis of fibre dispersion and orientation showed a good dispersion of fibres in the matrix, and a predominant orientation in the melt flow direction.

Appendix 1: Programme

September 15

11.00–12.00 Registration and Coffee

INTRODUCTION – Chaired by Dr. Mika Vähä-Nissi, VTT

12.00–12.15 Welcome and FA0904 in short; Dr. Mika Vähä-Nissi, VTT, FI

12.15–12.45 Keynote speech; Prof. Ali Harlin, VTT, FI

12.45–13.15 Sustainable packaging; role of nanotechnology;
Dr. Gulden Yilmaz – Jongboom, Wageningen UR, NL

13.15–13.30 Break

NANOCELLULOSE – Chaired by Dr. Tekla Tammelin, VTT

13.30–14.00 Novel polymer nanomaterials for food packaging based on modified cellulose nanofibres;

Dr. Ruth Schmid & Per Stenstad, SINTEF, NO

Kristin Syverud, PFI, NO

Martin Andresen, Borregaard, NO

14.00–14.30 Health & environmental safety aspects of nanofibrillated cellulose;

Marja Pitkänen, Jari Vartiainen & Anu Kapanen, VTT, FI

14.30–15.00 New approach to classification of cellulose fibrils and suitable methods for their characterization;

Asko Sneek, Marja Pitkänen, Heli Kangas, Tekla Tammelin & Erkki Hellén, VTT, FI

15.00–15.30 Coffee Break

SURFACES I – Chaired by Dr. Annaleena Kokko, VTT

15.30–16.00 Solid board as a base material for novel sustainable packaging applications;

Sanne Tiekstra, Bumaga BV, NL

Arie Hooimeijer & Maja Stanic, Kenniscentrum Papier en Karton, NL

16.00–16.30 Starch-clay paper coatings for sustainable, flexible packaging;

Dr. Francis Clegg & Prof. Chris Breen, Sheffield Hallam Univ., UK

16.30–17.00 Nanostructured materials based on electrospinning of interest in food biopackaging applications;

Dr. José-M. Lagaron, IATA-CSIC, ES

17.00–17.30 Changes in water vapor uptake ability of xylan derivatives monitored by QCM-D humidity module;

Dr. Tekla Tammelin, Kari Kammiovirta, Harri Setälä, VTT, FI

**18.00 Salad Buffet Dinner (Sponsored by VTT) & Posters:
The technology of antibacterial food packaging materials**

Paulius P. Danilovas, Dr. Ramune Rutkaite,

Algirdas Zemaitytis, Kaunas Univ. of Tech., LT

On the structure and exploitation properties of biodegradable and synthetic polymers based nanocomposites with layered silicate nanofillers;

Remo Merijs Meri, Janis Zicans, Velta Tupureina, Anita Kaulina, Madara Bartule, Juris Bitenieks, Valentina Musteata, Visvaldis Svinka, Riga Tech. Univ., LV
Cornelia Vasile, “P. Poni” Inst. Macromolecular Chemistry, RO

September 16

SURFACES II – Chaired by *Dr. Kalle Nättinen, VTT*

- 8.45–9.15** **Antimicrobial surface modification of polyethylene;**
Elena Paslaru & Cornelia Vasile, “P. Poni” Inst. Macromolecular Chemistry, RO
Lidija Fraszemljic, Univ. of Maribor, SI
Dona Constantinescu, SC ECEFS-Savinești, RO
Gina Procope, Food Safety Department, RO
- 9.15–9.45** **Atmospheric plasma technology for surface modification of nanoparticles;** Dirk Vangeneugden, Annick Vanhulsel & Sofie Put, VITO, BE
- 9.45–10.15** **Nanoscale surface processing of extrusion coated substrates and plastic films with atmospheric plasma activation and deposition;**
Dr. Johanna Lahti & Juho Lavonen, Tampere Univ. of Tech., FI
- 10.15–10.45** **Atomic layer deposition in food packaging and barrier coatings;** Dr. Pirjo Heikkilä, VTT, FI
- 10.45–11.30** **Lunch break**

COMPOSITES – Chaired by *Dr. Pirjo Heikkilä, VTT*

- 11.30–12.00** **Characterization of nanoparticles in polymers and their impact on the morphology for films for food packing;**
Prof. Geoffrey R. Mitchell, Inst. Polytechnic Leiria, PT
Donatella Duraccio, Marilena Pezzuto & Clara Silvestre, CNR ICTP, IT
- 12.00–12.30** **Mechanical, thermal and morphological properties of Polylactide/Polyhydroxybutyrate/Nanocomposite blends;**
Trevor W Woods, Werner J Blau, Kevin O’ Connor & Ramesh P Babu, Trinity College, IE
- 12.30–13.00** **Properties of PLA based micro- and nanocomposites;**
Dr. Karol Leluk & Marek A. Kozłowski, Wrocław Univ. of Tech., PL
- 13.00–13.30** **Coffee Break**
- 13.30–14.00** **Antibacterial composites based on iPP for food packaging application;**
Dr. Sossio Cimmino, Dr. Donatella Duraccio, Marilena Pezzuto, Clara Silvestre, CNR ICTP, IT
Antonella Marra, Veronica Ambrogio, Università degli Studi di Napoli, IT
Ida Romano, CNR ICB, IT
- 14.00–14.30** **Effect of processing on natural fibres in starch acetate composites;**
Dr. Kalle Nättinen, Heidi Peltola, VTT, FI
Bo Madsen, Tech. Univ. of Denmark, Risø, DK
Roberts Joffe, Luleå University of Technology, SE



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