Executive summary

The objective of the INCOM project was to develop techno-economically viable solutions and production methods for lightweight structures based on advanced sustainable materials for use in packaging, vehicles, sporting goods and aeronautical applications.

The main modules of the INCOM project were:

- nanocellulose fibre (NFC) production and modification process
- composites processing and sandwich structure manufacturing
- mechanical testing and verification in counterbalance with modelling and LCA.

Two approaches were used for manufacturing, modification and processing of nanocellulose fibre: The first approach was fibrillation of cellulose readily in a pre-polymer by basket bead milling. The second approach was fibrillation of bioresidue-based cellulose in aqueous media by grinding and development of a quality assessment tool for optimization of the process.

The NFC production using (TORUSMILL®) basket bead mill was developed for milling cellulose fibres in monomers (e.g. epoxy resin), sol-gel dispersion or solvents. In the Masuko grinding in water, the mechanical, morphological, rheological, and optical scattering behaviour was characterized to assess and optimize the degree of fibrillation. A relationship between viscosity and strength of the network (in dry state) was established. The relation from online viscosity measurements can be used for prediction of the energy necessary to reach the maximum network strength. Pilot implementation of both NFC production routes as well as upscaling evaluation was performed with industrial partners.

NFC reinforced resins were used to manufacture composite and lightweight sandwich structures. For sandwich structures, three types of cores were developed: expanded NFC reinforced bio-based PU foam core, bio-based thermoplastic foam core and thermoplastic honeycomb core. Industrially viable production methods thereof were developed. NFC reinforced bio-PU foams were found to perform in a level of commercial PU foams and addition of NFCs further improved the compressive strength and modulus of the foams. Regarding PLA foam composition and extrusion process were optimized and promising properties were achieved with small and even cell size. Novel industrially viable solutions for thermoplastic honeycomb core were studied and developed.

NFC-modified Inorganic-organic hybrid coatings were used to modify the surface properties of honeycomb and web structures. The combination of comparably high film thicknesses (20 µm) with high NFC contents (>20 %w/w) as well as impregnation of cellulose-based webs were used. Increment of mechanical properties by coatings proved to be quite challenging but potential solutions were developed especially with thermally curable hybrid coatings.

Novel composite structures and techno-economically viable processing techniques with high commercialization potential were developed and up-scaled during the project. An ecodesign approach was used and evaluated by LCA studies right from the early stages. This ensured a path of reduced environmental impact for these new industrial processes for cellulose reinforced nanocomposites.

The work done opens a completely new exploitation possibility for strong environmentally friendly composites and replacement of traditional state-of-the-art solutions leading to lighter high performance composite structures.

Summary description of the project context and the main objectives

The main objective of the INCOM project was to develop techno-economically viable solutions and production methods for manufacturing of lightweight structures based on advanced sustainable materials for use in vehicles, aeronautical applications and sporting goods. Special attention was dedicated to upscaling of production processes, pilot scale trials and industrial implementation.

In other words, lightweight, sustainable and durable composite sandwich structures were developed and manufactured for different applications. These structures are reinforced with nanocellulose fibre (NFC), a nano-scale material derived from cellulose found in biomass and bio-waste.

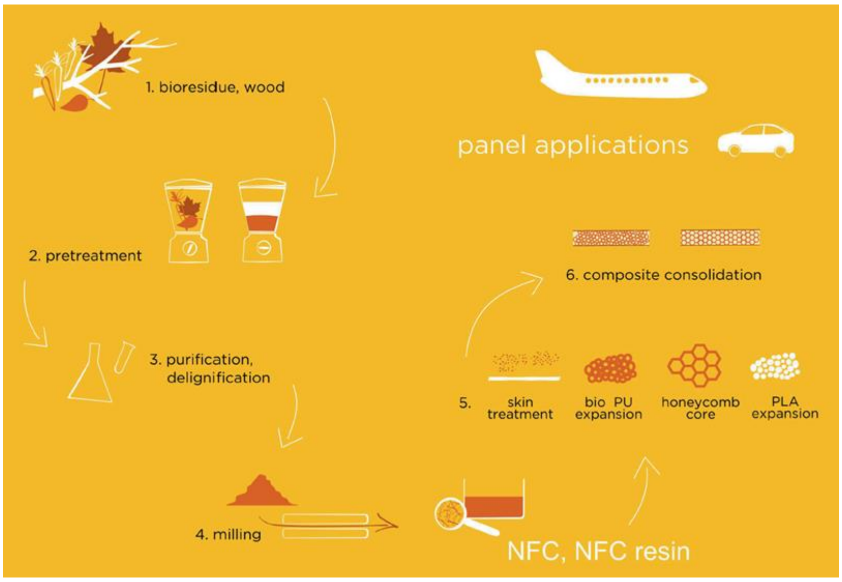
The main modules of the INCOM project were:

- Nanocellulose fibre (NFC) production and modification
- Composites processing and sandwich structure manufacturing
- Mechanical testing, verification and modelling
- Life cycle assessment

In the INCOM project, two approaches were used for the manufacturing and modification of NFC. The first approach was the nanofibrillation of cellulose in a pre-polymer (monomer or oligomer) medium. This avoids problems related to incompatibility of aqueous medium and pre-polymers and creates an optimal, homogenous fibril dispersion in the resin to be used in composite structures. Resins and sol-gel coatings reinforced with NFC or functionalised NFC were used in cores and skins of structural sandwich composites, to enhance mechanical properties. The project also aimed at adding value to bio-waste materials to be used for NFC production, focusing on the optimisation of energy consumption of the NFC grinding process. Therefore, the second approach was the development of nanofibrillation mainly in aqueous media and the optimisation of the nanofibrillation of bioresidues (e.g. carrot bioresidues).

Sandwich composite materials can replace monolithic structures in many applications, thus saving by reduction of input materials and overall weight of the structure. Moreover, the reduced use of materials in sandwich composites, compared to monolithic structures, can reduce their environmental impact. Three types of cores were developed for composite sandwich structures to meet different technical requirements: expanded NFC reinforced bio-based PU foam, bio-based PLA thermoplastic foam and thermoplastic honeycomb cores.

Demonstrators, such as automotive components, airplane cabin parts and sporting goods were produced during the project.



The quality and properties of the nanoreinforced materials were constantly tested, and numerical models were used to simulate mechanical behaviour and facilitate industrial upscaling. During the development process, LCA (Life Cycle Assessment) and LCC (Life Cycle Costing) were used to provide ecodesign feedback to project partners. Health and safety aspects were also taken into account, through a constant monitoring of existing national/international standards and health and safety related studies on nanocomposites.

The consortium of the INCOM project was composed of industrial participants (eight SMEs and one large company) together with leading European institutes and universities specialized in biocomposites, processing technologies and sol-gel development from seven European countries. The value chain represented by the expertise of the R&D partners was coupled with the selection of the industrial partners' line of business. The whole production chain from bio-based raw materials processing to different fields of applications was included.

Technical Research Centre of Finland – VTT (Finland)

Luleå University of Technology – LTU (Sweden)

Fraunhofer Institute for Silicate Research - Fh-ISC (Germany)

Technical University of Denmark – DTU (Denmark)

2B Srl - (Italy)

Diehl Aircabin GmbH (Germany)

Axon Automotive Ltd (United Kingdom)

Millidyne Oy (Finland)

VMA-Getzmann Gmbh (Germany)

SurA Chemicals (Germany)

Bergius Trading AB (Sweden)

Composite Solutions and Innovations Ltd, CSI (Finland)

EconCore N.V. (Belgium)

Description of the main S & T results/foregrounds

Nanocellulose fibre production

The work concerning nanocellulose fibre production focused on efficient NFC production using upscalable approaches and different milling mediums. The aim was to develop and scale-up process methods for the economic production of NFC with low energy demand at high consistency, repeatability and process stability.

Two different methods for the manufacturing of the nanocellulose were used: Basket bead milling at VTT and VMA-Getzmann and Masuko grinding at LTU and Bergius. The basket bead milling focused on screening the boundaries of the system using new milling mediums: monomers and hybrid sol-gel solutions. The Masuko grinder is an established technique for the isolation of nanocellulose thus the focus here was on upscaling and the production of NFC of uniform quality at low energy consumption.

Masuko grinding (LTU, Bergius)

In the Masuko grinding, different industrial residues: Carrot residue and Brewers spent grain (BSG) were evaluated. For comparison, these residues were compared to birch kraft pulp (see Figure 1).

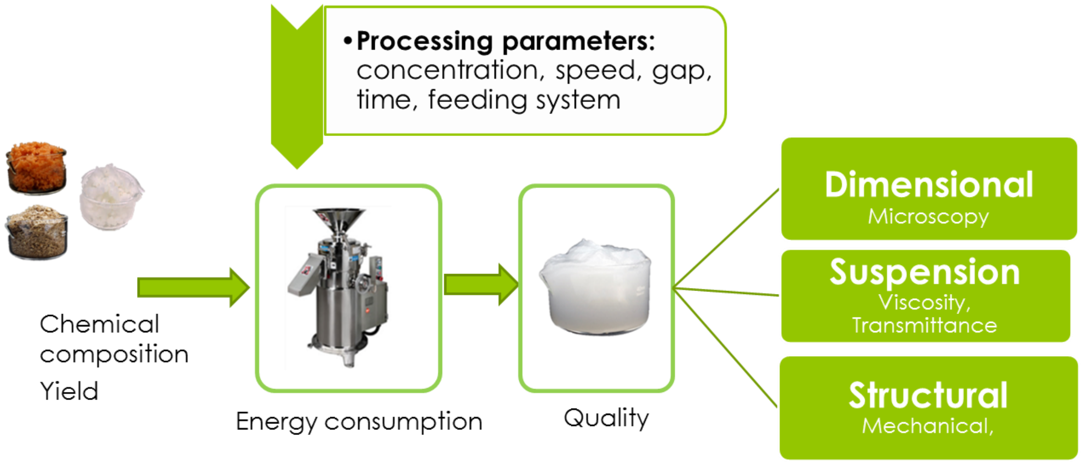


Figure 1. Processing optimization parameters and characterization using Masuko ultrafine grinding

The mechanical, morphological, rheological, and optical scattering behavior was characterized to assess the degree of fibrillation. For online assessment of the fibrillation process; a relationship between viscosity and strength of the network in dry state was established for several different sources of raw material. Figure 2 shows the linear relationship between the energy demand taken at the maximum measured viscosity and the energy demand at the maximum strength. This linear relationship was close to being directly proportional, which would mean that the ability of network formation measured in wet state, represented by the maximum viscosity value, was strongly correlated to the strength of the network in dry state. Thus the equation of the linear least squares fit could be used for prediction of the energy necessary to reach the maximum network strength from online viscosity measurements of nanofibre suspensions, using ultrafine grinding and at the given processing parameters. For pilot implementation and upscaling evaluation, the development continued with carrot residue, the most promising industrial residue. The production of NFC from carrot residue was achieved at low energy consumption using less than 2 kWh/kg, hence well meeting the targeted energy demand for the process. In a larger perspective both the use of this type of suitable industrial cellulose residue and the tool for predicting energy requirement for strong networks based on viscosity enable processing with significantly less energy demand. This presents a more environmentally friendly option for the processing routes, promoting the industrial production of nanofibres and their subsequent use as reinforcement in nanocomposites where the composite depends on a strong network formation.

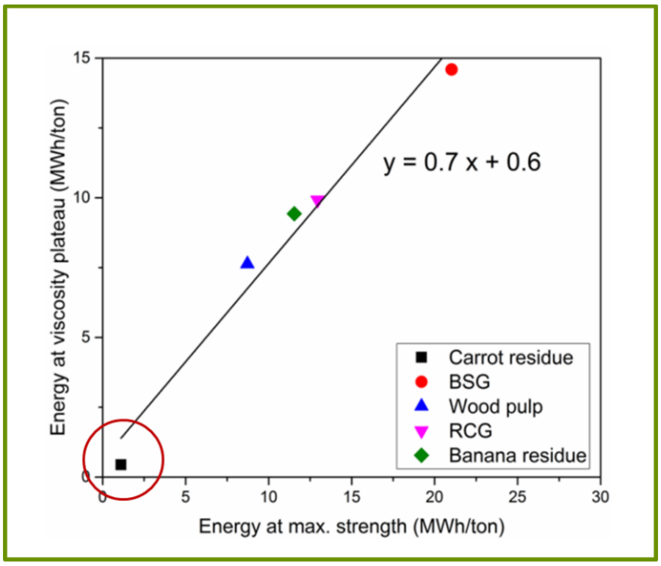


Figure 2. Correlation for the grinding process energy needed for maximum strength raw materials: carrot residue, Brewer´s spent grain (BSG), Wood pulp, reed canary grass (RCG), and banana residue.

Carrot-based nanofibres produced by LTU and Bergius were further utilised e.g. in Diehl's demonstrator, reinforcements in PU-foams and organic coatings as well as nanopapers surface treated by SurA, Figure 3.

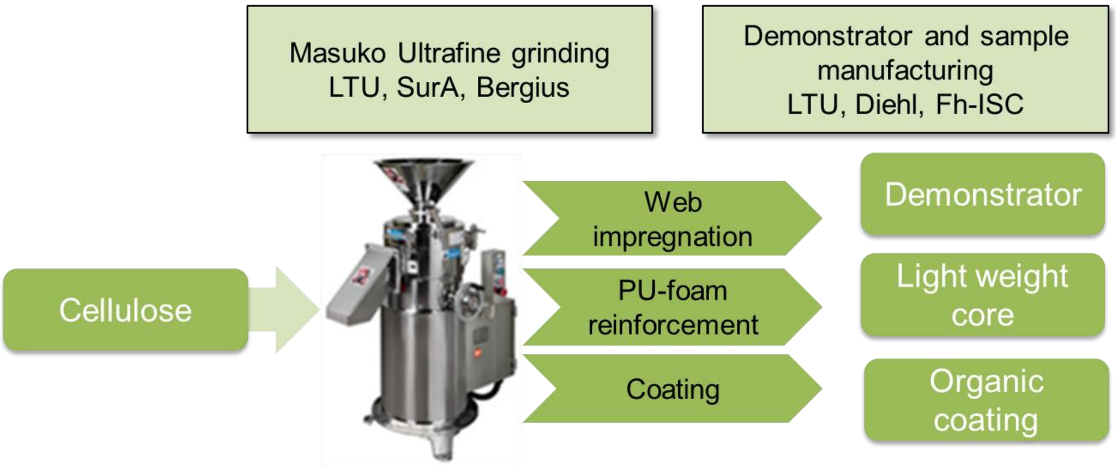


Figure 3. Schematic of demonstrator and sample manufacturing via ultrafine grinding

Basket bead milling (VTT, VMA-G)

Basket bead milling (Figure 4) of cellulose fibres hasn't earlier been done in monomer dispersions and hybrid sol-gel's thus one important objective of the project was the boundary assessment for bead milling in those medias. Based on the results even never dried hardwood can be bead milled to micro/nanoscale in epoxy resin and sol-gel pre-solutions with highly volatile solvents are processable and yield ultrafine dispersions, which are very challenging to reach with any other technique.

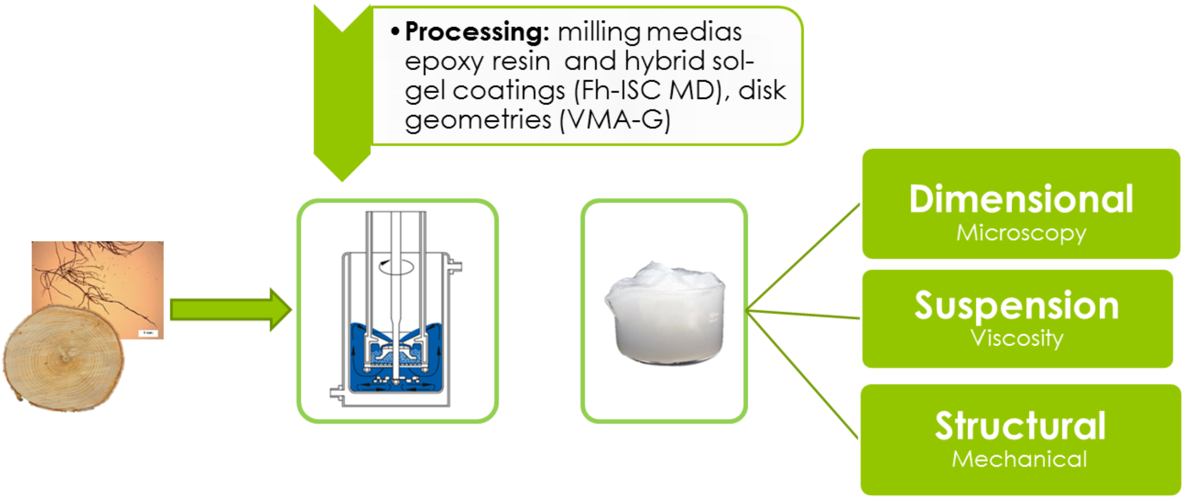


Figure 4. Principle of basket bead milling process

Wood cellulose-based fibres were fibrillated and dispersed in epoxy resins and sol-gel pre-solutions by VTT and VMA-Getzmann. The cellulose fibres in the resin or sol-gel solutions were further utilised e.g. in skin and monolithic structures as well as in Axon Automotive's and CSI Composite's demonstrators and in Millidyne's and Fh-ISC's hybrid coatings for honeycomb structures, Figure 5.

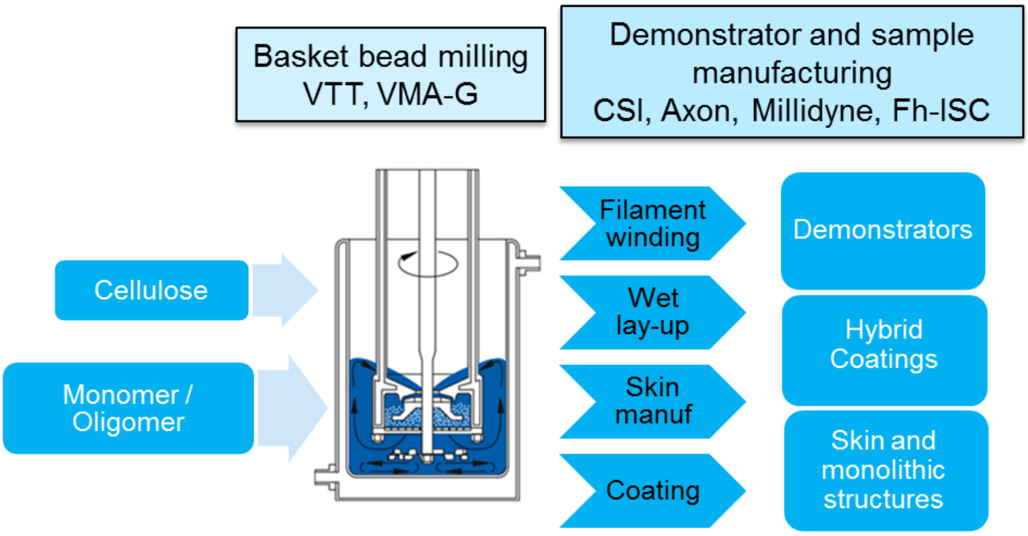


Figure 5. Schematic of demonstrator manufacturing via basket bead milling

Cellulose fibrillation in monomer (VTT, VMA-G)

The main target was the milling of NFC in monomers in order to reach better mechanical properties for further processed thermoset structures. Commercial epoxy resin chosen by end user partners was used as milling media. Cellulose types used in bead milling were mainly woodbased fibres of three groups: Never dried cellulose fibres, dried microcellulose and readily in nanoscale processed aqueous fibrils. Water from never dried and NFC solutions was removed either before milling by solvent change, by freeze drying or after/during milling by evaporation. These actions enabled a moisture free compound when the curing agent was finally incorporated in the composite consolidation phase. VTT in co-operation with VMA-G was responsible for the laboratory scale millings while VMA-G managed the up-scaling of the millings. Both partners delivered milled batches to partners CSI and Axon for sample and demonstrator manufacturing.

Basket bead millings in epoxy resin were successfully done for micro- and nanoscale fibres. With never dried fibres, there was an issue with blocking of the screen. In addition, a basket bead mill screen with new construction was developed and evaluated. With better understanding of milling and flow phenomena, never dried hardwood fibres could be milled but in minor concentration compared e.g. to microscale cellulose. Due to length of the softwood fibres and their tendency to form dense networks in basket bead milling they were not feasible to mill with lab scale basket bead mill. Instead, they were demonstrated to be millable by vertical milling configuration.

Due to case-specific viscosity demands of sample and demonstrator manufacturing processes, the viscosities of bead milled batches were measured as function of temperature by VTT. An example is given in Figure 6.

°C	mPas
63.1	114.0
60.0	152.0
55.0	212.0
50.0	317.0
47.5	368.0
45.0	447.0
43.6	500.0
40.0	680.0
35.8	1000.0
35.0	1080.0
30.0	1800.0
29.0	2000.0
25.4	3000.0
25.0	3140.0
23.9	3570
22.1	4400.0

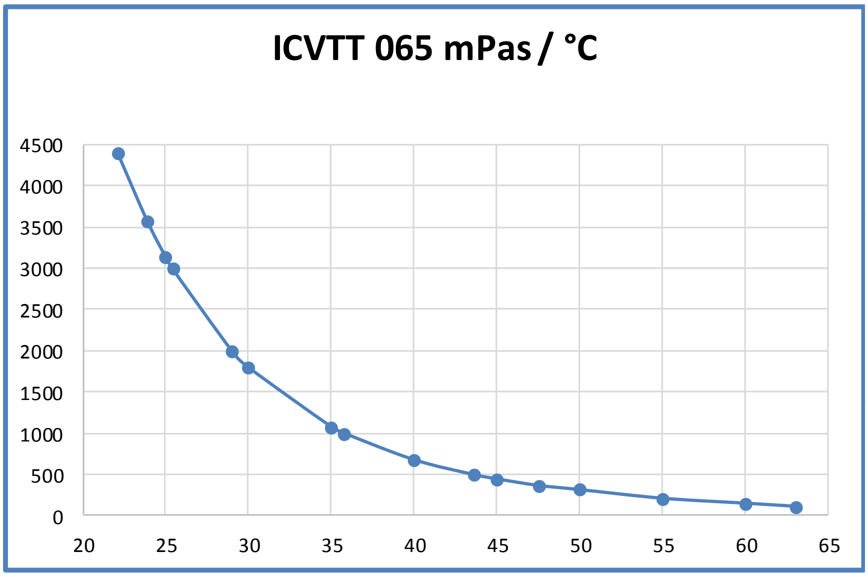


Figure 6. Example of viscosity vs. temperature dependence of NFC-resin

In addition to supplying partners NFC-modified epoxy resin, VTT also fabricated universal ‘dog-bone’ test samples with resin transfer moulding (Figure 7) for mechanical tests.

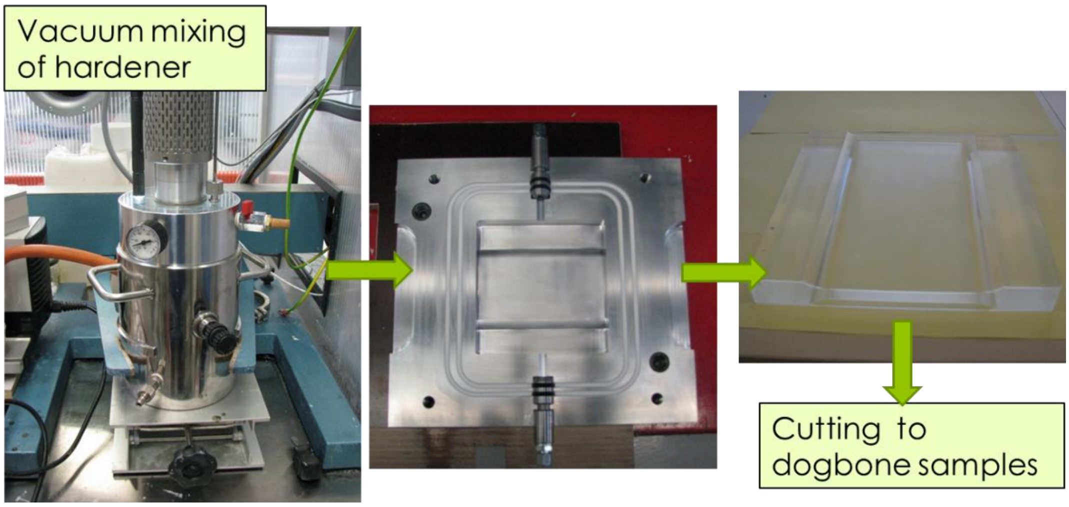


Figure 7. RTM sample preparation of test samples from bead milled NFC-resin.

Based on needs evolved during the project, VMA-G developed new milling disc geometries for more efficient milling .In addition, a new type of screen with tangential direction slots was developed in co-operation between VTT and VMA-G. VMA-G also used a simulation software to improve bead movement and circulation in order to get a better fibrillation effect for the cellulose (**Error! Reference source not found.**).

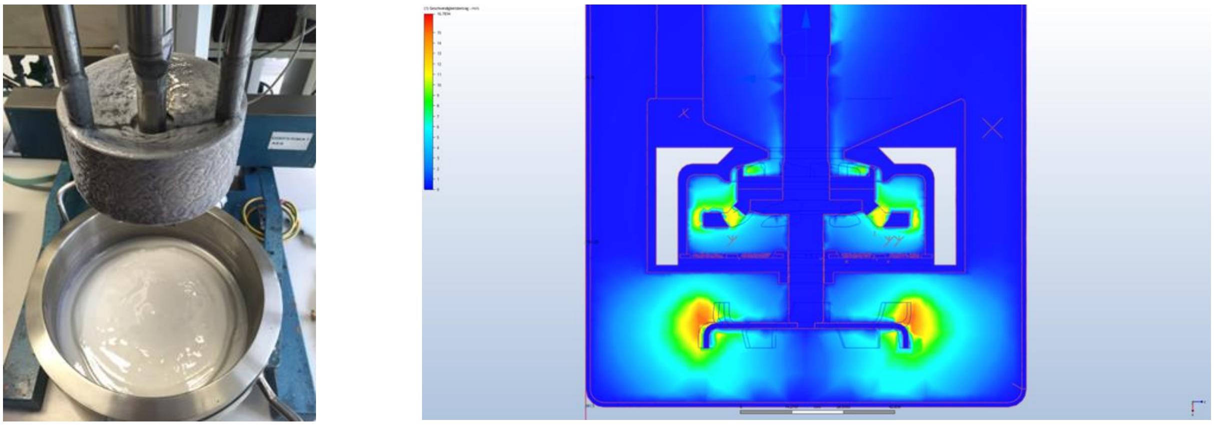


Figure 8. Simulation software for developing new milling disc geometries and testing method.

Cellulose fibrillation in hybrid sol-gel (VTT, VMA-G, Fh-ISC, MD)

The main target of the task was the fibrillation and fine dispersing of NFC during milling to hybrid sols. This was done by utilizing silane coupling agents enabling covalent bonding of NFC to sol-gel matrix through the reaction between hydroxyl groups of cellulose and functionalized siloxanes (e.g. methacrylate, epoxy or amino). The millings were done by VTT with TORUSMILL® TML10 basket bead mill. Various silylated and non-silylated wood-based celluloses from VTT were milled in several sol-gel systems from Fh-ISC and Millidyne who also further processed the solutions to coatings.

Cellulose types used in bead milling were mainly wood-based fibres of three groups: Never dried cellulose fibres, dried microcellulose and readily in nanoscale processed aqueous fibrils. The cellulose type, amount, milling accessories and parameters were chosen application-based by VTT, Fh-ISC and Millidyne. In addition, the effect and boundaries of solvents used in sol-gels were taken into account and fine dispersions of sprayable NFC-modified coatings were milled successfully.

Nanocellulose fibre treatments (LTU, DTU)

Collaboration work between DTU and LTU has been performed to study the effect of plasma treatments on nanocellulose. It has been found that helium plasma treatment improved the wetting of nanocellulose by deionised water and glycerol and increased the contents of oxygen, carbonyl group, and carboxyl group on the nanocellulose surfaces. Ultrasonic irradiation during the plasma treatment further enhanced the wetting and oxidation. In the perspective of composite materials, these changes to the nanocellulose can potentially improve their processability when they are to be impregnated with a polymeric matrix.

Scanning electron microscopic observations showed skeleton-like features on the plasma-treated surface, indicating preferential etching of weaker domains, such as low-molecular weight domains and amorphous phases. Ultrasonic irradiation improved the uniformity of the treatment. Increase in surface area by roughening and/or etching can improve adhesion with a matrix due to the increase in the contact area.

Altogether, it is demonstrated that plasma treatment is a promising technique to modify the nanocellulose before composite processing.

Up scaling of milling processes_(Bergius, VMA-G)

Both Masuko and basket bead milling (TORUSMILL®) were found to be well up-scalable to targeted volume (> 100 kg) and rate (> 1000 kg/day). With Masuko ultrafine grinding of carrot residue to nanofibrils in water energy consumption below 2 kWh/kg was successfully met. The available equipment for Masuko ultrafine grinding is shown in Figure 9 and available basket bead mill systems in the Figure 10. With basket bead milling, various wood-based celluloses were successfully milled in monomer resins and hybrid inorganic-organic sol-gel solutions.

Figure 9. Upscaling possibilities with Masuko ultrafine grinding equipment, going from lab scale to production

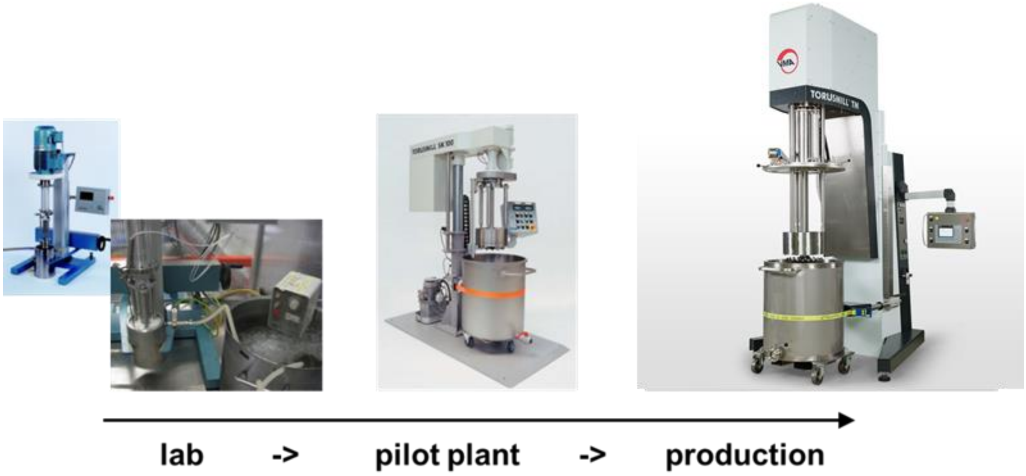


Figure 10. The available up-scaling equipment of basket mill system; from lab to industrial production.

Coating development

The core of the coating work in the INCOM project was the use of NFCs, preferably in inorganic-organic hybrid coating matrices, to coat the surface of honeycomb or web structures with the aim to increase the stiffness of the polymer-based structure. The NFCs used in this part were designed and manufactured in nanocellulose fibre production section. The reason for the use of inorganic-organic hybrid coating materials as a matrix for the coating/NFC composite is their high degree of variability, which facilitates the application on different substrate polymer films or paper, from which honeycomb structures are produced. The compatibility of coating materials with the substrates coated and with the nanocellulose incorporated as well as overall processability are essential requirements and therefore were intensively studied and optimized during the project.

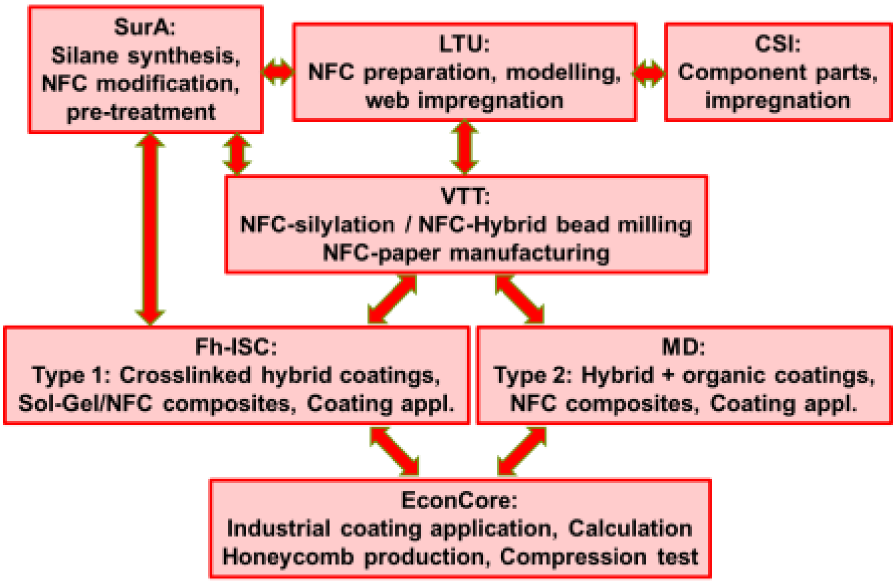


Figure 11. Diagram showing the co/operation in coating development and utilisation in INCOM/project

Fraunhofer ISC and Millidyne were the main partners in the project working on the sol-gel technology to manufacture hybrid coating materials.

Fraunhofer ISC's materials were based on special organically functionalized siloxanes bearing reactive groups, which have to be activated to form a solid film by a polymerization process (type 1, Figure 12). Millidyne used, in general, in-organically crosslinked, physically drying materials based on the progress of sol-gel condensation reactions followed by evaporation of solvent as well as acrylic and PU modified compositions (type 2). Both types were basket bead milled with NFC incorporations at VTT and coatings thereof done by/for industrial partners and characterization.

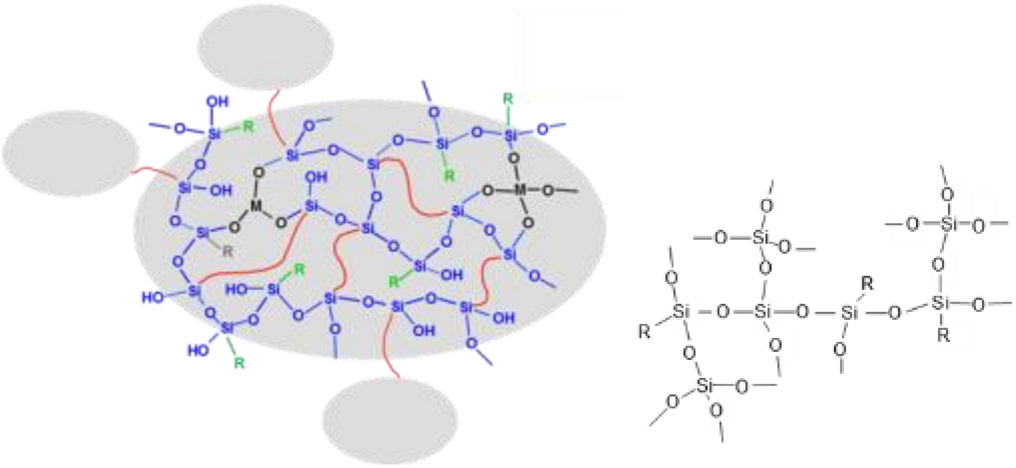


Figure 12. Sol-gel coating types with crosslinked clusters and oxidic network developed in the project, type 1 = left, type 2 = right.

A vast variety of coating matrices, their compatibility with different NFC:s and performance were studied by Fh-ISC and Millidyne. VTT and SurA provided NFCs with various surface modifications (e.g silylations) for better compatibility and performance with coating matrices. As one example, sol-gel matrix with reactive components from Fh-ISC and surface modified hardwood based NFC from VTT (Figure 13).

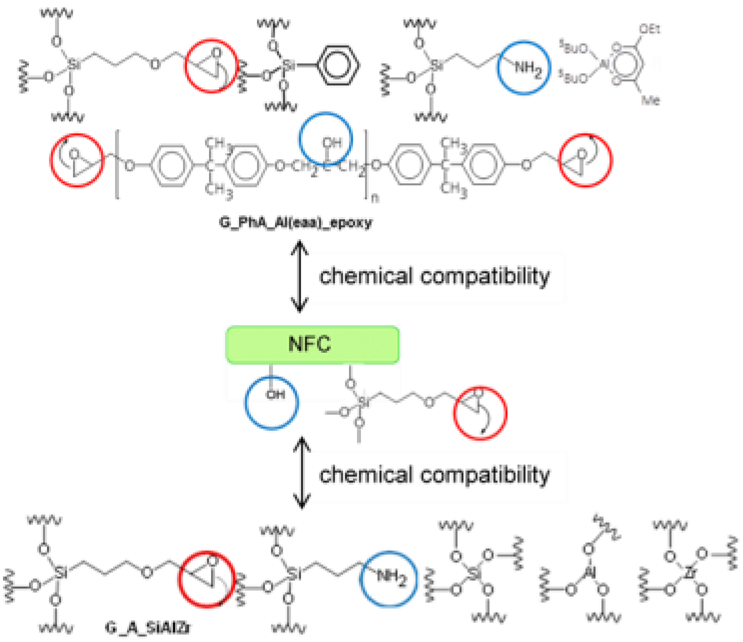


Figure 13. Thermally curable hybrid materials with groups chemically compatible to modified NFCs (see red and blue circles)

Regarding mechanical properties of coated honeycomb structures up to 30% increase in compression strength could be obtained with Fh-ISC's inorganic-organic thermally curable hybrid coatings.

NFC modified organic coatings (e.g. epoxy and polyurethane based) were developed mainly by Millidyne and SurA. Coatings studied in the project had different structures and chemical properties as well as different affinity and compatibility to NFCs.

SurA focused mainly on surface treatments (e.g. CCVD, Combustion Chemical Vapor Deposition) enhancing coatability (e.g. wettability, adhesion) of substrates as well as functionalizing the NFC's provided by LTU and hydrophobizing NFC-papers provided by VTT, Figure 14.

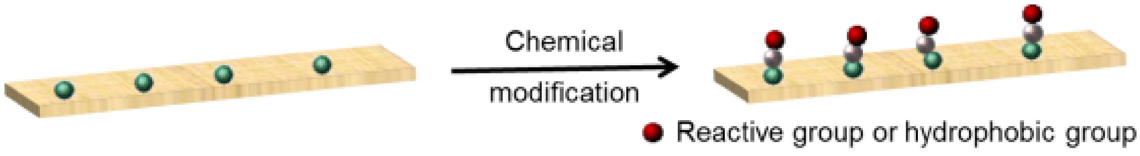


Figure 14. Chemical modification of surfaces

In addition to stiffness, other mechanical properties were also enhanced in the project. In work done by SurA scratch resistance of waterborne PU-resins could be increased up to 200% with less than 2% NFC addition while the coating still remained transparent, picture below.

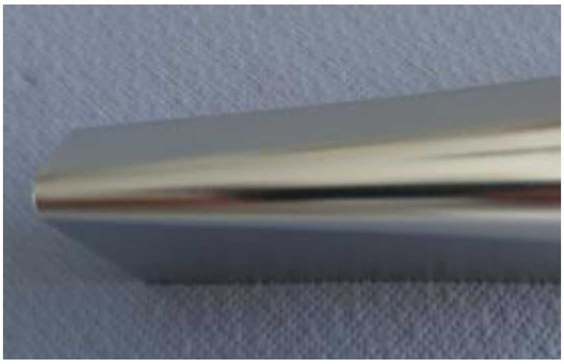


Figure 15. Waterborne PU resin with 2% NFC on aluminium

Nanocellulose based films and webs

NFC-based papers were also manufactured for coating tests. LTU manufactured carrot-based nanopaper via Masuko-milling and VTT hardwood based NFC-paper where the NFC slurry was first done with fluidizer and translucent NFC-film sheets fabricated with R-2-R Surface treatment concept (SUTCO). The NFC-papers were further surface treated and coated by partners Fh-ISC, Millidyne and SurA targeting to barrier properties and water repellence. As an example of hydphobising effect, some treatments done by SurA are presented in Figure 16. All three coating partners were able to successfully hydrophobise the NFC-papers. In addition, reduction of the water vapour and oxygen transmission was gained.

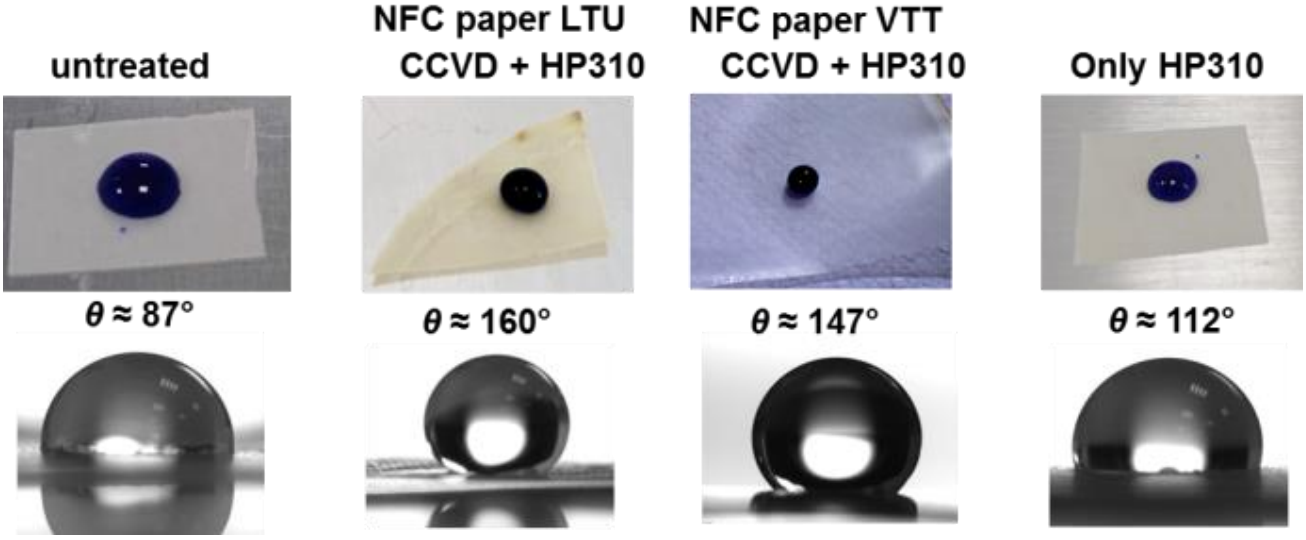


Figure 16. Hydrophobisation of NFC-papers.

LTU studied and developed impregnated nanocellulose based webs. In this task, NFC was used as a web or skin on a sandwich structure and therefore it was impregnated with a suitable resin. Typically, NFCs form dense networks with high mechanical properties but are difficult to impregnate.

The key to produce the composite was the use of a novel method of impregnation, whereby NFC is filtered but not allowed to dry (maintain in a hydrogel state), then submersed in a resin before being consolidated and finally the resin is cured to a B-stage (see Figure 17). This produces an impregnated sheet (a pre-preg) that can then be consolidated and cured with other sheets to produce a thin laminate. The pre-preg can also be inserted in other (glass) pre-pregs to produce hybrid composites.

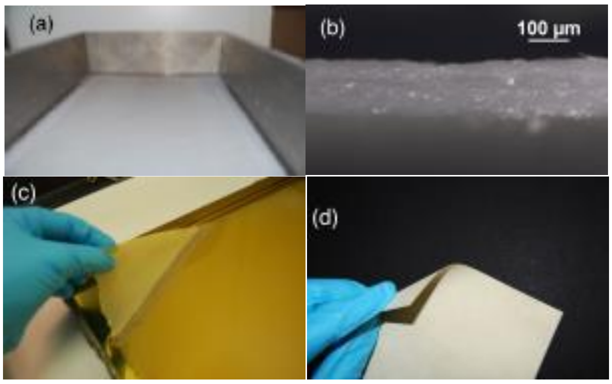


Figure 17. (a) Scaled filter system for nanopaper manufacture, (b) stereomicroscope image of the nanopaper in a hydrogel state prior to impregnation (c) impregnation stage with the nanopaper in resin bath, and (d) resulting NFC/Bakelite pre-pregs.

The work done here comprised a close collaboration between LTU and Diehl. NFC from carrot was bleached by SurA and ground with an ultra-fine grinder, and the nanofibres were subsequently used for preparation of the nanopapers.

In summary, the result was a stiff, relatively strong NFC/Bakelite composite with high nanocellulose content (approximately 50%). The laminates passed the fire resistance test, required by Diehl, despite the high cellulose content and resulted in lower heat release and heat evolution rate values compared to that of the glass-fibre counterpart. The results of the mechanical test are shown in Figure 18 where the NFC composite is compared to glass fibre laminates tested by Diehl.

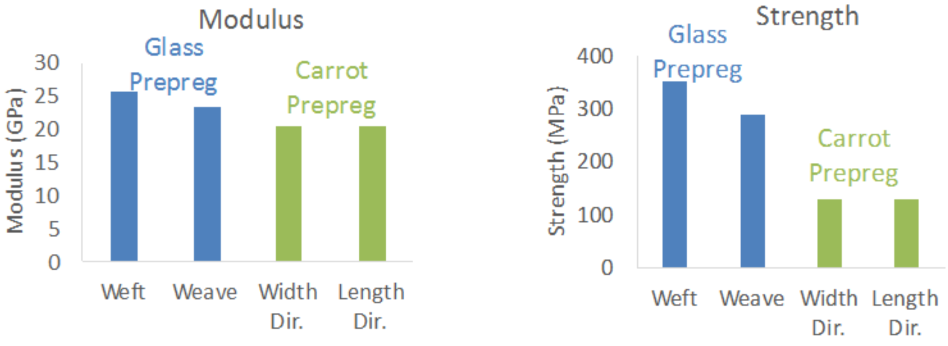


Figure 18. (a) Modulus (to the left) and (b) strength (to the right) of the NFC/phenolic composites and glass fibre/phenolic composites.

The results show that the modulus is close to that of the glass-fibre composite manufactured with the same resin, but the strength of the NFC composite is lower than that of the glass-fibre composite.

Coating work was done in close co-operation between coating developers and end application manufacturers. For example, Millidyne did on-site coating tests in Econcores production facilities.

Light weight core development

Sandwich composites materials can replace monolithic structures in many applications, thus saving input materials and reducing the overall weight. Sandwich structures with a low cost core material can be more lightweight and more cost effective. Moreover, the reduced use of materials in sandwich composites, compared to monolithic structures, can reduce their environmental impact. In transportation, lightweight structural materials mean fuel savings and reduction of environmental burden.

The core development focused on developing methods to produce expanded, low density core structures for use in lightweight sandwich panels. Three parallel approaches were used for the production of these low density materials:

- Polyurethane foams from bio-based materials, reinforced with NFC (LTU)
- Physical foaming of thermoplastics, bio-based polylactide, PLA (VTT)
- Development of honeycomb cores (Econcore et.al.)

Rigid polyurethane foams from bio-based materials, reinforced with NFC

Semirigid and lightweight (<50 kg/m3) bio-based PU foams reinforced with nanocellulose fibre were developed by LTU. Nanofibres are usually dispersed in water and aggregated when drying. Nanofibre dispersion was mixed in polyol together with water & dioxane co-solvent, which were removed by heating. The mixture resulted in well dispersed nanofibres. Foams with lowest density of 35 kg/m^3 showed highest impact.

The addition of NFC had a positive effect on foam properties. The nanocomposite foams were performing in a level of commercial PU foams. The addition of the NFC brings both the modulus and the strength of the BPU-based foams in line with the commercially available rigid foams as shown in the Figure 19. Thus, when density is considered, these nanocomposite foams show a commercially viable performance with regards mechanical properties. Their use in a sandwich structure was also investigated by measuring the flexural stiffness of the foams with and without a skin of paper impregnated with epoxy (Figure 20). The chart shows that the flexural properties of the foams are in line with those of rigid PU foams, as was the case for the compressive properties of the nanocomposite foam. It also shows that sandwich structure can be successfully made from the foams and that as expected, enhances the flexural modulus.

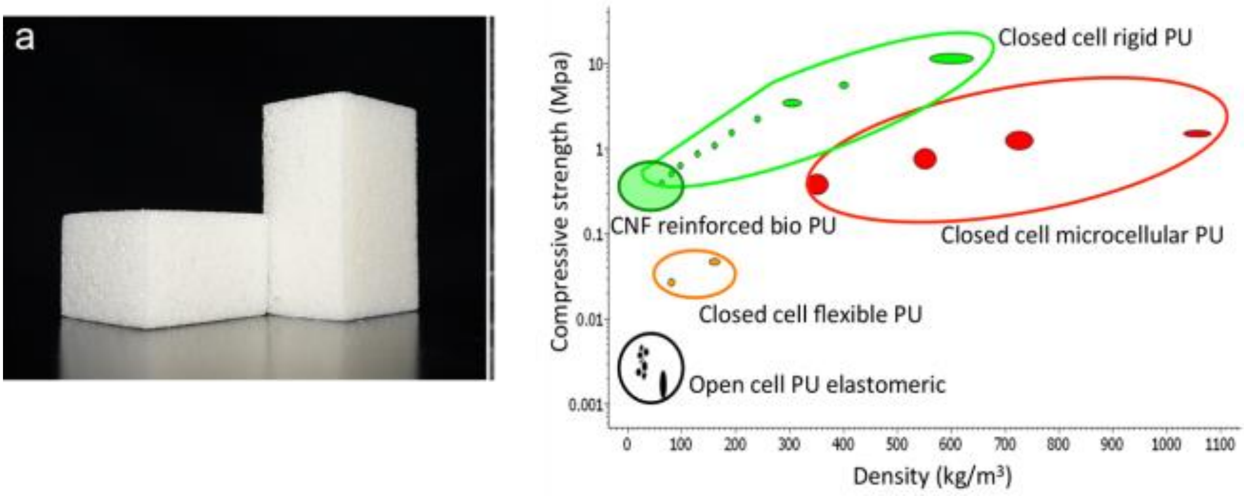


Figure 19. Rigid bio-based polyurethane foam reinforced with carrot NFC. Addition of NFC improved mechanical properties to the level of commercial rigid PU foams

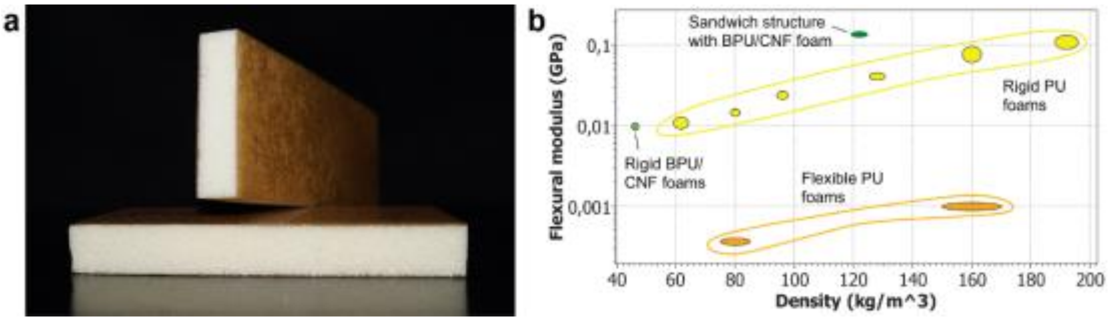


Figure 20. *a) Vacuum infused sandwich composite with CNF reinforced BPU core and cellulose skin layer, and b) a chart of the flexural modulus verse density of the prepared nanocomposite foam and its sandwich structure in comparison with other PU foam families. Data from CES Edupack 2015 software (Granta Design Ltd., Cambridge (2015)).*

Physical foaming of thermoplastics, bio-based polylactide, PLA

At present, synthetic polystyrene (PS) polymer is primary used in the manufacturing of foamed structures for packaging and large area insulation products. The most common production technologies are expanded polystyrene (EPS) and extruded polystyrene (XPS). Highly potential bio-based material to replace petroleum-based PS in these lightweight structures is polylactide (PLA).

Core prototype structures from bio-based thermoplastic polymer PLA were studied in the laboratory scale (Brabender extruder) and pilot scale tandem extrusion foaming line at VTT. The initial screening of PLA foamability was performed in small scale extrusion foaming trials. The aim was to produce optimised foam structures and define the production parameters and properties based on extrusion foaming of thermoplastics. Blowing agents CO_2 and the mixture of CO_2 and isobutane were used. Small and even cell size was achieved by using mixture of CO_2 and isobutane. Targeted foam density <50 kg/m^3 was reached and the compression stress varied between 0.12 – 0.19 MPa depending on the process parameters and the right combination of chain extender/nucleating agent. The thermal conductivity of PLA foam was measured to be similar compared to a measured XPS sample. Optional particle foaming process for PLA was also studied. The single expanded particle density of 20 kg/m^3 was achieved.

Figure 21. a) Extruded PLA foam and expanded PLA foam particle samples prepared at VTT. b) Small sandwich samples using extruded PLA foam and carbon fibre reinforced skins produced by CSI Composites.

Development of honeycomb cores

The objective for development of thermoplastic honeycomb structure, based on EconCore's patented ThermHex process, was to achieve improvement of performance or/and optimisation of core's weight.

The development of thermoplastic honeycomb structure can be divided in three different approaches:

- Nanoreinforced material applied (e.g. as a coating) onto the honeycomb walls
- Cellulose fibre reinforced thermoplastic
- Flame retardant honeycomb

EconCore's patented ThermHex production technology for production of Honeycomb Sandwich Panels was used to develop new light weight core materials, Figure 22.

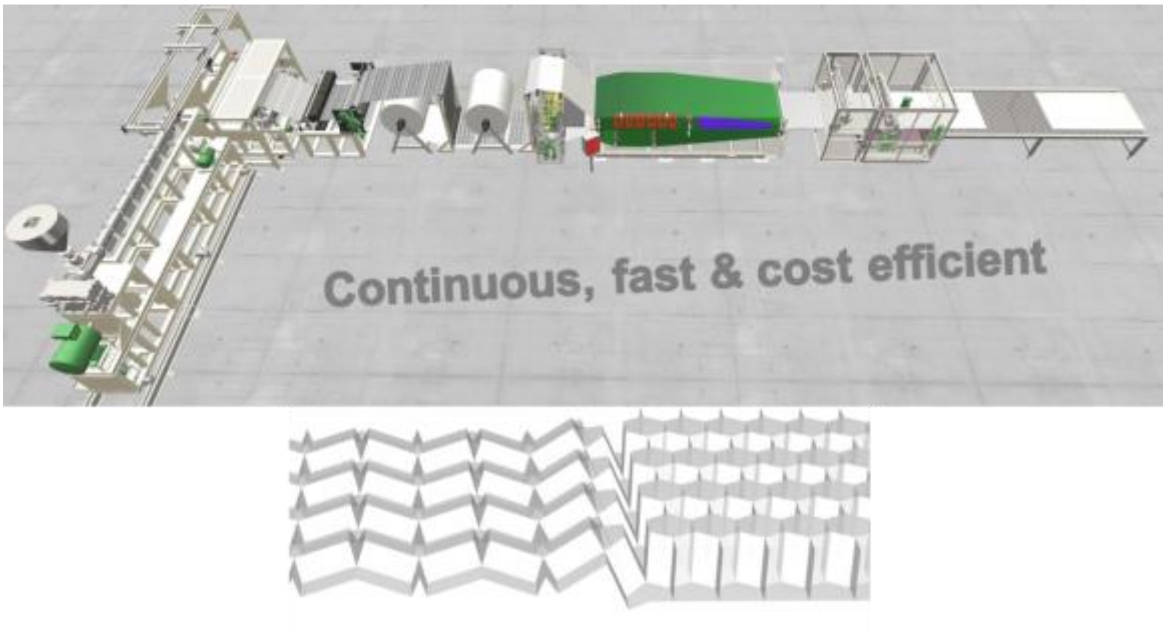


Figure 22. ThermHex production process

The nanoreinforced material applied (e.g. as a coating) onto the honeycomb walls

The aim was to develop a nanoreinforced material, or specifically nanocellulose reinforced resin, that can be applied (e.g. as a coating) onto the honeycomb walls in order to achieve a reinforcement effect. The development of the coating was performed by project partners Fh-ISC, Millidyne and SurA and is described in the Coating development chapter. Trials for applying the coatings were done by EconCore and Millidyne. DTU and EconCore measured the properties of the coated honeycombs.

Thermally curable coatings were applied on honeycomb structures and the compression strength was measured. The developed epoxy hybrid coating by Fh-ISC showed an increase in the compression strength of honeycomb structures from 0.3 to 0.4 MPa (= N/mm²), which is an improvement of 30 %, even without addition of NFCs (Figure 23).
NFC filled epoxy hybrid coatings with 2 %, 5 % and 10 % load of NFCs were applied on Polycarbonate (PC) honeycombs by spraying, Figure 24c, but the mechanical property results were not available before the end of the project.

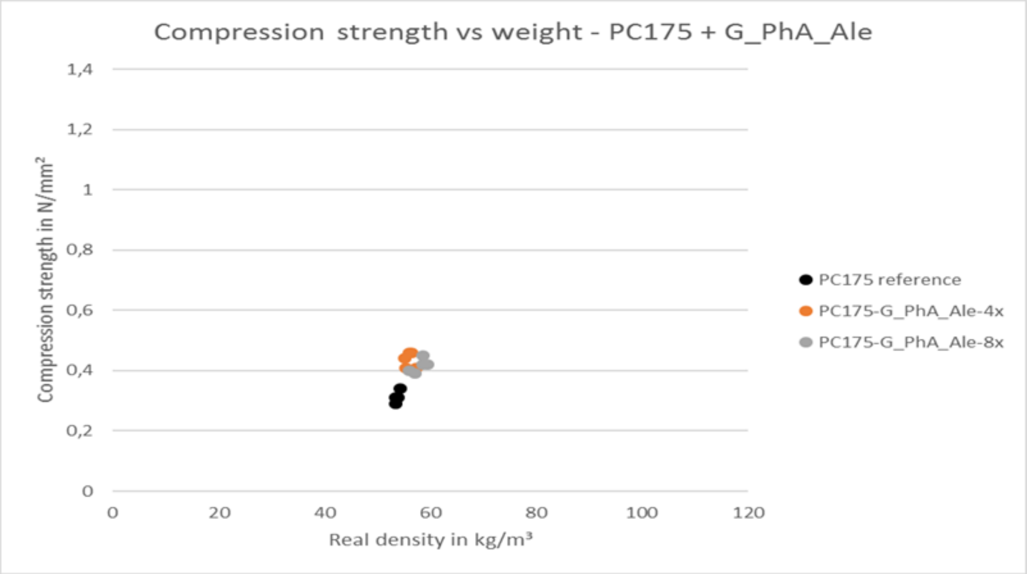


Figure 23. Compression strength of PC honeycombs, coated with G_PhA_Ale_epoxy.

In order to better visualize the wetting, coatability and spreading of the coatings coloured UV-curable coatings were spray coated also on folded honeycomb-structures, Figure 24 It could be observed that with proper spray management also folded structures can be coated homogeneously.

Figure 24. a) and b) UV curable non-filled colored coating. c) thermally curable coating with 10 % NFC. (Fh-ISC)

The approach of using sol-gel based hybrid coatings to coat lightweight polymer-based honeycomb structures is promising for achieving an increase in stability and strength. However, some challenges still need to be overcome.

Cellulose fibre reinforced thermoplastic

Cellulose fibres give the opportunity to improve the stiffness of the core of sandwich structures ecologically without increasing the weight and at the same time decrease the use of oil-based polymers. EconCore and VTT developed cellulose fibre reinforced thermoplastic film for honeycomb structures.

Bio-based polylactide (PLA) and petroleum-based polypropylene (PP) films were reinforced with 10, 20 and 30 wt-% microcellulose. Based on study of their mechanical properties and thermoformability, PP film with 20 wt-% of cellulose fibres was extruded and thermoformed into honeycomb structure. The process steps are illustrated in the Figure 25. Figure 26 shows the sample of the thermoformed web and (laminated) honeycomb core based on PP with 20 wt-% cellulose content.

The production of these environmental friendly lightweight material constructions for different applications is feasible to scale up to industrial production processes.

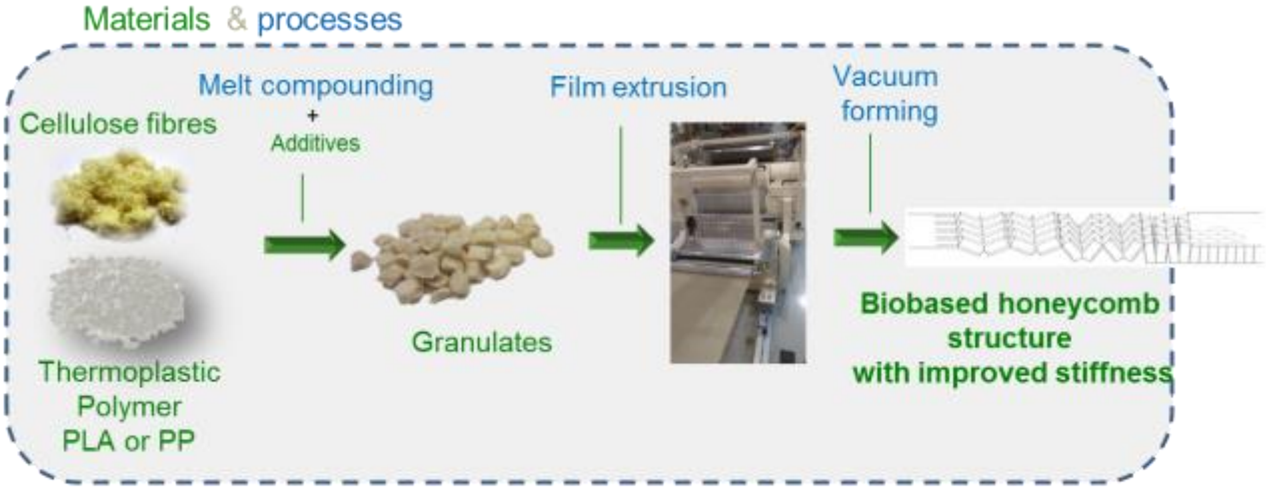


Figure 25. Process steps for producing cellulose fibre reinforced honeycomb structures.

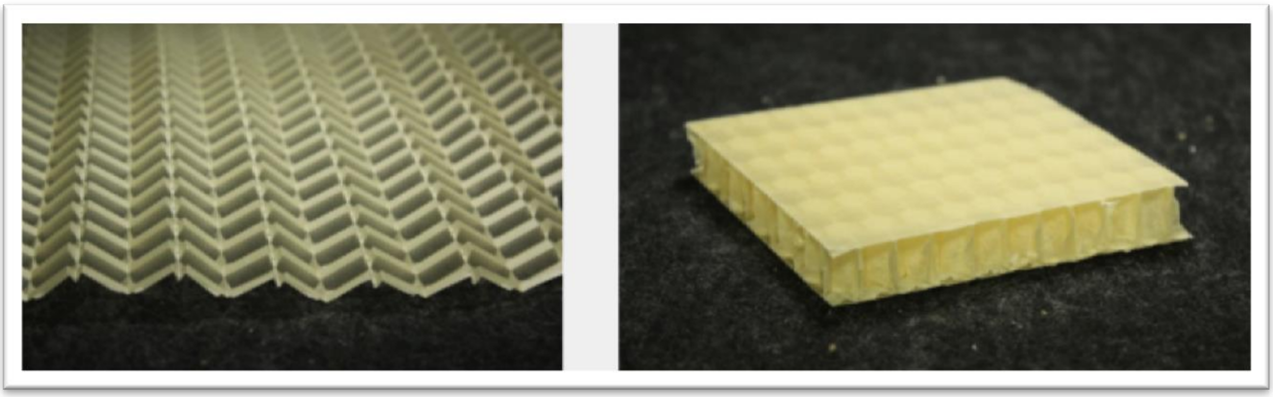


Figure 26. Sample of the thermoformed web and (laminated) honeycomb structure based on PP-core with 20% cellulose content.

Flame retardant honeycomb structures

An interesting development route with high potential of commercialization has been identified by EconCore and Diehl Aircabin. The two companies envisaged a new highly cost-effective process for creating modules by thermoforming and functionalizing mono-material sandwich panels in a single step. EconCore's honeycomb production process was proven to be able to deliver honeycomb cores based on Fire-Smoke-Toxicity qualified polycarbonate material, Figure 27. Such honeycomb prototypes, produced by EconCore for the needs of the project as per validation of Diehl Aircabin GmbH, show very promising results in view of commercial and mechanical aspects. Fire resistance of these thermoplastic honeycombs was tested and the results were shows a high potential: in combination with aerospace qualified thermoset skin layers all requirements towards flammability, heat release, smoke density and smoke toxicity are met. The cost however is much lower making the product an option to be considered in wider range of applications, including ground public transportation such as railway transportation.

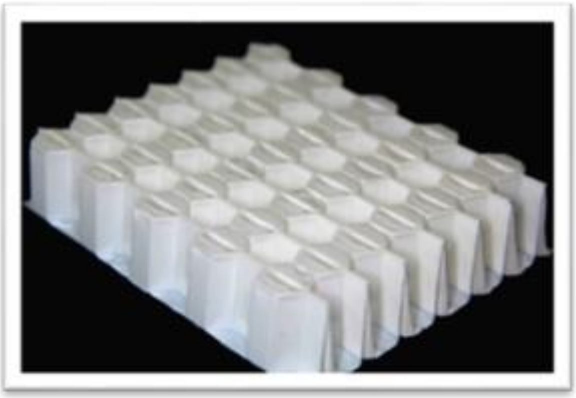


Figure 27. ***Sample of the 16mm fire-smoke-toxicity-qualified PC core.***

Numerical modelling of the mechanical performance of nanocellulose composites

3D computational simulations of deformation and damage behaviour of nanocellulose (NFC) reinforced composites were developed, taking into account the real nanocellulose structures, and hydrogen bonding effect. In order to carry out such simulations, a new software for the automatic generation of 3D unit cell finite element models of nanocellulose reinforced polymers with “snake”-shaped nanocellulose fibrils was developed. By varying the structures of nanocomposites (content of nanocellulose, availability of hydrogen bonds, etc), the relationships between structures and damage behaviour were investigated. With the developed 3D simulations the mechanical performance of the nanocellulose composites can be predicted.

Demonstrators

Aircraft stowage unit demonstrator

The use of cost effective, thermoplastic flame retardant PC honeycomb core manufactured by Econcore was demonstrated in Diehl’s stowage unit of the Airbus A350, so called “Doghouse”.

The stowage unit consists of flat panels, highly stressed parts (crash loads) and quite high level of complexity. Highly stressed areas (front panel and slide) were reinforced with embedded nanocellulose paper from LTU (process described under coating development).

Composite structures with PC core and NFC paper were successfully manufactured and no problems occurred in the subsequent process steps: milling, grinding, filling, varnishing and final assembly.

Figure 28. Stowage compartment of the Airbus A350 so called "Dog House" manufactured in INCOM project.

The environmental impact of the aircraft stowage unit prototype was quantified by means of LCA by 2B and resulted in a hot spot analysis, identifying the main contributors to the environmental burden of the prototype: electricity consumption during manufacturing, refrigerant due to the release of tetrafluoroethane, aluminium inserts and the pre-peg resins. Improvement options were suggested and ecodesign feedback was given to the consortium partners.

The contribution of the INCOM materials NFC (0.3%) and EconCore Thermhex (1.7%) to the environmental burden of the stowage unit prototype was assessed to be relatively small, while the materials seem to give a positive effect to the structure and weight reduction of the prototype. Both are important aspects in the context of sustainable lightweight structures applied in aeronautical applications. It was therefore recommended to evaluate the possibility to use more INCOM materials in the prototype.

The GWP of the aircraft stowage unit seems to be high, but in the context of possible fuel savings due to the weight reduction of the unit, this impact is entirely counterbalanced. Considering that a weight saving of 0.1 kg is equal to a saving of 942 kg in terms of kerosene and 2835 kg in terms of CO_2 life span emissions, the stowage unit prototype, designed as the interior part in the A350-program, represents large environmental advantages. Despite the high manufacturing energy consumption and thereby consequently high environmental impact, the impacts of the stowage unit are entirely compensated due to the weight reduction of the unit and associated fuel savings, which has been possible by applying the light weight sandwich core of the INCOM project.

Bulkhead demonstrator

The aim of the Axon Automotive demonstrator was to evaluate the potential of honeycomb cores and NFC reinforcement in the automotive sector. For this purpose, the front bulkhead of Axon Automotive Far Platform Chassis was selected as the demonstrator component. The comparison between the two prototypes, the monolithic and sandwich bulkhead, has shown significant improvements, both in terms of environmental impact and costs, due to the application of INCOM materials. The mechanical testing suggested that improved fatigue properties are achievable with the addition of nanocellulose in the resin.

The primary purpose of the front bulkhead is to isolate the passenger cabin from the harshness of the engine bay. It protects against fumes, and prevents the progression of fire into the cabin. The bulkhead equally provides some level of structural rigidity in a crash by mitigating against lozenging of the vehicle frame, and preventing intrusion of engine bay contents into the occupant zone within the cabin.

Figure 29. The front bulkhead of Axon Automotive Far Platform Chassis was selected as the demonstrator component.

Two automotive demonstrators were manufactured, one monolithic version and one sandwich version, Figure 30. Both demonstrators were manufactured via wet hand lay-up process using NFC reinforced resin produced by VTT and VMA-Getzmann by bead milling process. Honeycomb core was provided by EconCore. A hand lay-up technique was chosen due to the high viscosity of the NFC - resin. Other manufacturing methods such as wet pressing, vacuum infusion and press consolidation of sandwich panels were also studied and utilised to produce samples for testing to assess the material to be used for the final demonstrators. The materials used for the demonstrators are summarised in Figure 30. In Figure 31, it is possible to see the process adopted for the manufacturing of the sandwich version of the bulkhead.

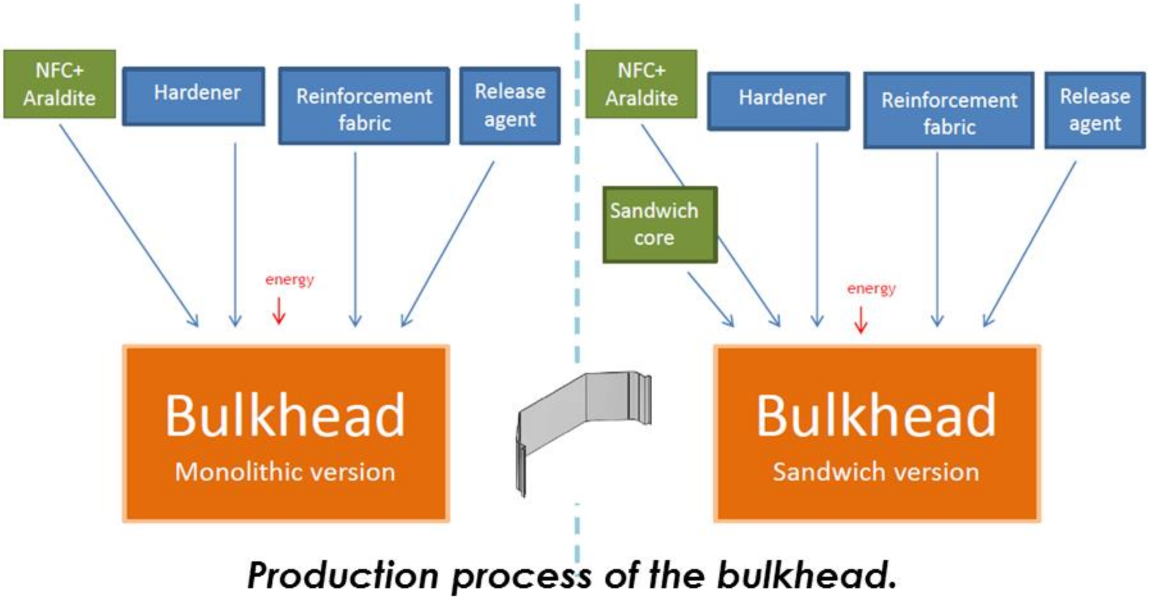


Figure 30. Two production routes were chosen for the bulkhead production: monolithic and sandwich versions.

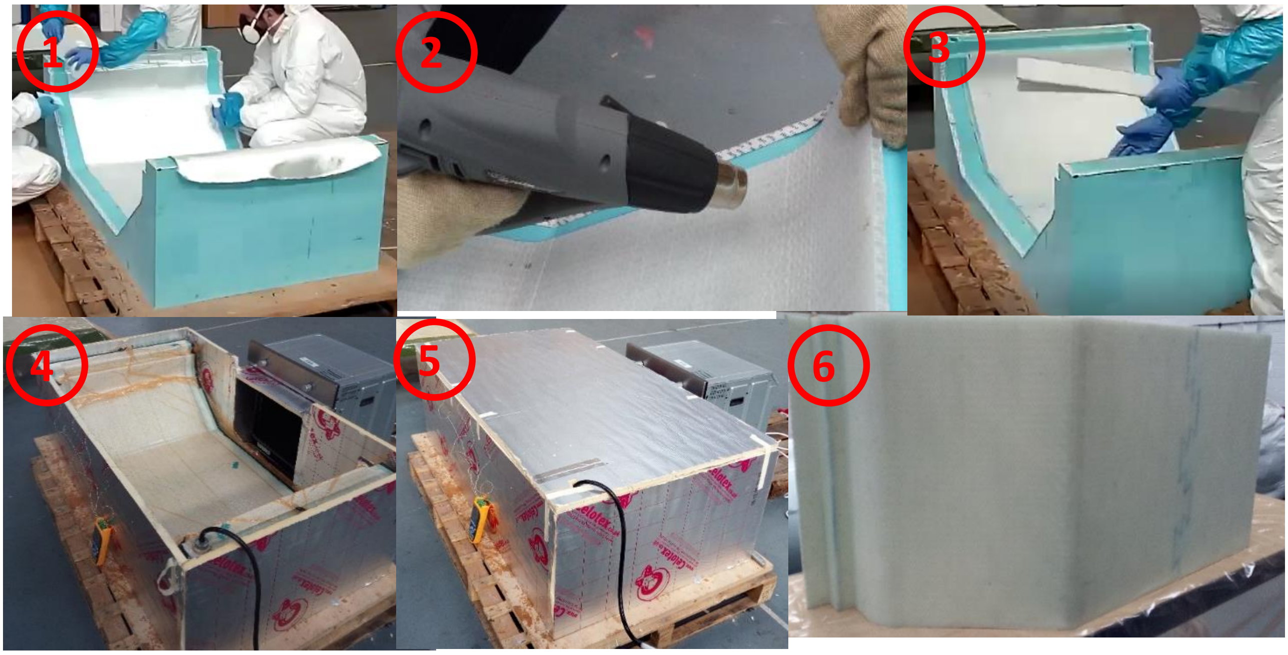


Figure 31: manufacturing of the sandwich version of the bulkhead. 1) E-glass dry fabric placement in the tool 2) thermoforming of the PP honeycomb core 3) placement of the honeycomb core in the bulkhead tool 4) the bulkhead bagged and under vacuum 5) curing of the part in the oven 6) final part after demoulding operation

The fatigue test performed by DTU on samples produced with the INCOM resin and E-glass fabric used in the demonstrator manufacturing showed improvement in the fatigue properties compared to laminates produced with standard resin (Figure 32). This could potentially lead to components with longer life. However, more testing need to be carried out to understand better the fatigue behaviour of this material.

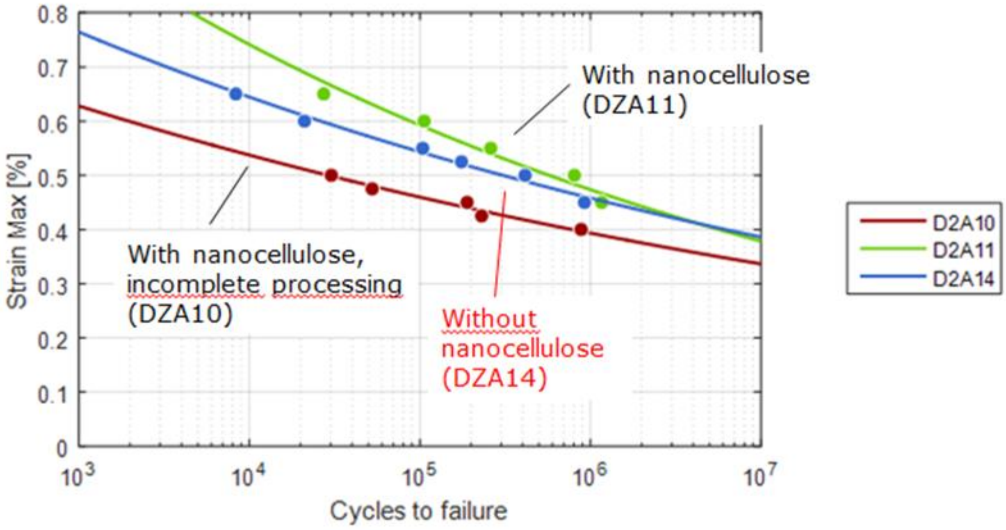


Figure 32. ***Fatigue S-N curves for biaxial glass fibre/epoxy composites, with and without nanocellulose in the epoxy matrix.***

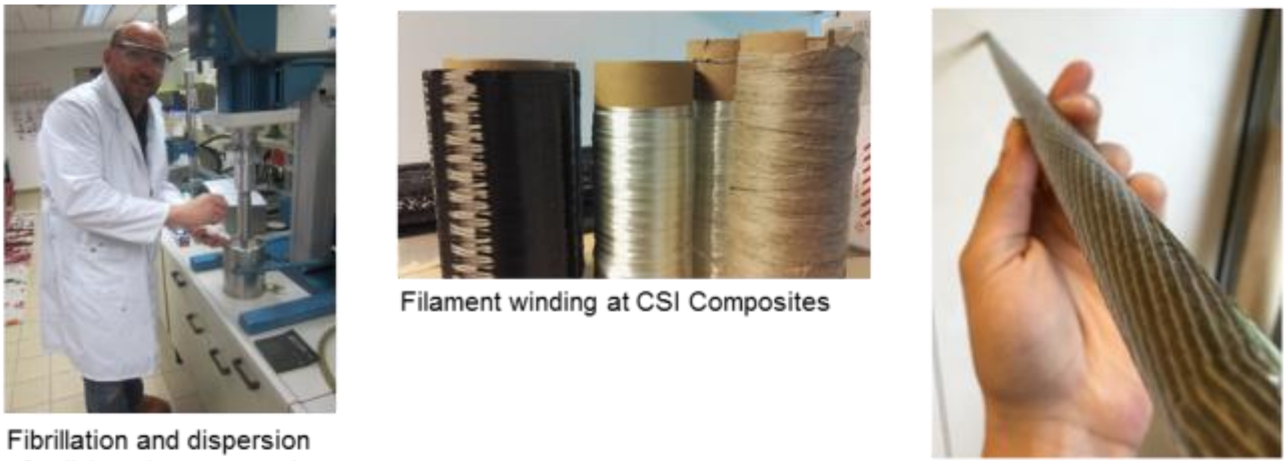
Life cycle assessment study by 2B has shown significant improvements in terms of both environmental impact and costs, due to the application of INCOM materials in comparison between the two prototypes, the monolithic and sandwich bulkhead. The mechanical results in conjunction with the LCA results show that the INCOM materials provide a competitive solution to increase resin properties with negligible effect on the manufacturing cost of the resin.

The contribution of the NFC dispersion to LCA results is relatively small while the contribution of the honeycomb core in the sandwich bulkhead is significant. This is justified since the application of the honeycomb core results in weight savings and thus avoids use of other materials, present in the monolithic bulkhead. For both versions, the processes with the largest contribution are the resin and the glass fibre used in the reinforcement fabric. Mainly due to weight savings, the environmental impact of sandwich bulkhead compared to monolithic bulkhead is clearly lower. This advantage can be very positive in terms of fuel savings and associated CO_2 emission reductions.

Sporting goods demonstrator

During the INCOM project, properties of NFC-resin were found to be very interesting in terms of gaining enhanced technical features for sporting goods. A process called filament winding was used to produce the sporting goods demonstrator by CSI Composites. Filament winding was found to operate extremely well with nano/microscale cellulose basket bead milled with epoxy resin. In terms of commercial potential and up-scaling, sporting goods were found to be very suitable for demonstrating high volume production. The base line for producing sporting good demonstrators was to study several material combinations. Secondly, a study of a semi-automated production line with NFC resins was carried out. All the produced demonstrators were tested by DTU in line with the testing program. The capability to meet the material needs of high volume production was investigated in co-operation with VMA-Getzmann and VTT. The prototypes were manufactured by CSI. The aim of the CSI demonstrator was to evaluate the potential of the INCOM resin, a nanocellulose fibre epoxy resin, in the sporting sector.

With NFC resins there are certain limitations in infusion, resin transfer or pressing type of manufacturing processes, mainly because of the fact that the nano or micro sized cellulose fibres tend to be filtered when impregnating to fibre network. The increased viscosity of the NFC – resin causes also challenges in processing. In the filament winding process, this phenomenon does not exist, because of the nature of the process. Fibres are lead through the resin tank and get wet so that no filtering is taking place. Based on the results in terms of processability of the NFC resins, filament winding is extremely promising process to be used when utilizing the features of NFC resins.


Filament winding at CSI Composites

Fibrillation and dispersion of cellulose in epoxy resin at VMA- Getzmann

Fibre reinfroced sporting goods

Figure 33. Process steps at VMA-Getzmann and CSI Composites to produce sporting goods.

Samples of the sporting equipment demonstrator were characterized by DTU by X-ray tomography to evaluate the internal microstructure, Figure 34. Indications of voids were found, which should be addressed by improved processing conditions. The skiing pole samples have been tested by 4-point bending to evaluate their mechanical performance. The addition of NFC to the matrix of the composites was not found to lead to increased mechanical performance. Any positive effect of NFC might be masked by the existence of voids. It was found that the skiing poles have been developed to a satisfactory level. The performed measurements are valuable for benchmarking further developments.

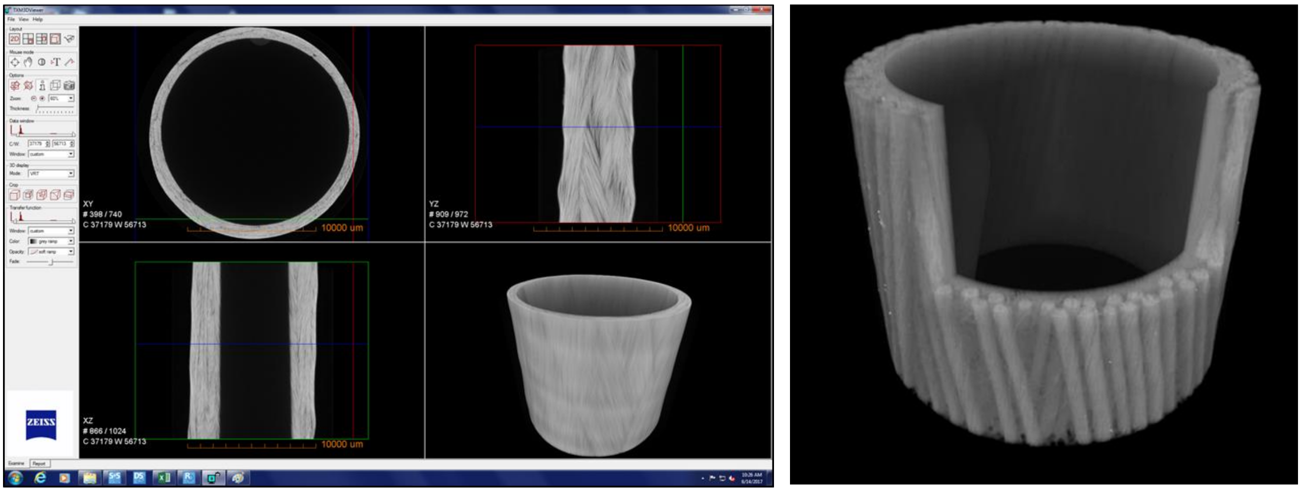


Figure 34. ***X-ray tomography images of glass fibre and carbon + flax fibre composite sporting good samples, DTU.***

The LCA performed by 2B reveals that the processes with the largest contribution to the INCOM sporting goods are the polyacrylonitrile fibres, used in the carbon fibre. The NFC was found to have a relatively low contribution to the overall environmental impact of the INCOM sporting goods (0.1%). Improvement options were suggested by 2B: reduction of the use of carbon fibres and to increase the amount of INCOM materials applied in the prototype.

The environmental impact of the baseline and INCOM sporting goods appear to be more or less the same. This is due to the small amount of NFC applied in the INCOM sporting goods; in the used INCOM resin only 0.5 % of NFC was applied.

The eco-efficiency performance is similar for the baseline and the INCOM sporting goods, because the environmental impacts and the life-cycle costs of the two prototypes are also very similar. For the same costs and environmental burden, additional functionality can be created for the sporting goods by applying the NFC materials of the INCOM project. Potentially, NFC plays an important role in the weight and structural properties of sporting goods.