1 Final publishable summary report.

1.1 Executive summary.

BRIGIT consortium developed a cost-competitive and environmentally friendly process to produce biopolymers (PHB, PBS, its copolymers and blends) from waste-derived lignocellulosic sugar feedstock liquor from the wood sulfite pulping process. The fermentation process to produce PHB and succinic acid (SA) for the production of PBS was carried out in the spent sulfite liquor (SSL) through a without alteration of the quality of current lignosulfonates (LS) contained in liquor.

The main innovations in BRIGIT are the use of an existing sugar-rich waste stream from the production of cellulose and the process integration with the existing industrial operation. A selection of efficient microorganisms, and optimized fermentation and downstream technology allowed to reduce resources consumption, and operational costs. The obtained biopolymers, PHB and Bio-PBS showed properties comparable to commercial materials.

BRIGIT developed new flame retardants (FR) derived from modifications of the LS contained in the liquor, PHB, SSL and from crude biomass containing PHB.

By blending the biopolymers with flame retardants BRIGIT developed bio-based compounds for fire-resistant applications in the transport sector (trucks and buses). The compounds in combination with natural fibres and cork were used to produce 3D sandwich panels alternative to current panels made out with thermosetting resins reinforced with glass fibres. The panels were obtained by a continuous compression moulding (CCM) process in contrast to current available sandwich panels.

To fulfil the global objectives, BRIGIT developed cost competitive and environmental friendly bio-based composites for high-tech fire-resistance applications based on:

- The use industrial waste by-product as raw material (SSL) with no competition with food supply.
- Increase of the amount of suitable sugars resulting from the digestion of hardwood chips.
- Removal and handling of the inhibitors in the SSL to maximize biopolymer yield.
- PHB and SA process integration with the existing industrial operation (sulfite pulping).
- Development of environmental friendly and high performance separation technologies for PHB and SA purification.
- A PHB obtained using new fermentation culture conditions in a well-characterized sugar waste stream (SSL).
- Increase the SA yields, integrated fermentation and new separation technologies.
- Reduction of costs of current SA at purities higher than 90%. Process integration.
- Methodologies to obtain, Bio-PBS (Polybutylene-succinate) and some of its copolymers with improved performance to current market PBS.
- To enhance the low fire retardant properties of PHB based biopolymers by modifications of LS, and PHB with phosphate and nitrogen moieties.
- Biopolymer PHB/PBS blends and compounding with different halogen-free and new FR
- Development of multilayer structures to improve the final performance of sandwich panels.
- Continuous compression moulding in combination with natural fibres to produce 3D sandwich panels suitable to replace panels made of thermoset resins reinforced with glass fibres in applications for trucks and buses.
- Recyclability of biocompounds, up to a 30%.

1.2 Description of project context and objectives Executive summary

BRIGIT project aims to develop a cost-competitive and environmentally friendly process to produce biopolymers (PHB, PBS, its copolymers and blends) from waste-derived lignocellulosic sugar feedstock liquor from the wood sulfite pulping process. The fermentation process to produce PHB and succinic acid (SA) for the production of PBS will be carried out in the spent sulfite liquor (SSL) through a without alteration of the quality of current lignosulfonates (LS) contained in liquor.

The digestion of wood by sulfite pulping process lead to high quality cellulose fibres and black liquor containing lignin, sugars from the hydrolysis of hemicelluloses and the other wood components. In this process, lignin is converted to lignosulphonate (LS) salts and hemicellulose is hydrolysed to sugars usually by reaction with calcium or magnesium sulphite. Lignosulfonates from sulfite process are still the main source of lignin for industrial additives; dispersing, binding, complexing and emulsion-stabilizing agents. BRIGIT will propose new high added value applications for them as the base production of flame retardant additives.

Nowadays, sugars produced during sulfite pulping process (near 0.5 Million tons/ year) are mainly destroyed using different chemical products as they have a negative effect over lignosulfonates quality.

As summary, BRIGIT aims to transform an existing sugar-rich waste in a high added value biopolymer to be used in the manufacture of panels for passenger transportation industry. The multidisciplinary consortium set up in order to carry out the R&D working areas and technologies addressed in BRIGIT has representatives from the entire value chain from feedstock, biosynthesis of the polymer or polymer precursor, through to the optimization of product recovery, purification and further conversion towards the final product (3D sandwich panel validation on prototype trucks and buses). Proposed applications represent a significant step respect to the current applications of biopolymers, based only in low-cost low-demanding applications.

In comparison with previous projects to obtain biopolymers from different sources, the main innovation in BRIGIT is the use of an existing sugar-rich waste stream and the process optimization and integration with the existing pulping industrial operation that will permit an overall reduction in resource consumption and reduction of operational costs. To minimize the potential problems with inhibitors, different routes will be studied such especial separations devices and new microorganisms strains under inhibitors conditions.

Bio-composites manufactured from natural materials, such as fibres and bio-based polymers have become an alternative to conventional thermoplastic materials in several low added-value commercial applications in sectors such as packaging and agriculture. These limitations can be overcomed by developing improved bio-based polymers and by engineering new composite materials from renewable resources combining proposed biopolymers with flame retardant additives (including new developments in intrinsically fire-resistant copolymers) and natural fabrics.

Polyhydroxybutyrate (PHB) is one of the most promising alternative bio-polymers due to its highly crystalline structure, which contributes to improve thermal and chemical resistance, having fast biodegradability in different environments and a positive environmental impact during production, despite of limited mechanical properties (fairly stiff and brittle), narrow processing windows and high production costs

To solve these limitations, PHBs are normally blended with thermoplastic aliphatic biodegradable polyesters, normally synthesized from petrol-based monomers. One of them is the polybutylene succinate (PBS), which is expected to have the highest rate of market penetration due to its favourable properties. PBS, which is produced by polymerization of succinic acid and 1,4-butanediol is biodegradable and has low young modulus, thermal and chemical resistance and an excellent processability.

For these reasons, BRIGIT aims to develop cost competitive and environmental friendly bio-based composites for high-tech fire-resistance applications based on:

- Increase of the amount of suitable sugars resulting from the digestion of hardwood chips (*Eucalyptus globulus* and *Eucalyptus nitens*) in at least 35% in the sulphite pulping process.
- Removal and handling of the inhibitors in lignocellulosic waste materials to maximize biopolymer yield. Our objective is to remove more than 75% of acetic acid, furfural and hydroxymethylfurfural.
- PHB and succinic acid (SA) process integration with the existing industrial operation (sulfite pulping) to achieve an overall reduction in resource consumption and emissions.
- Assessment of the use of bio-based and biodegradable solvents and high performance separation technologies for PHB and SA purification.
- A PHB obtained using new fermentation culture conditions in a well-characterized sugar waste stream.
- Increase the SA yields from current 22-24 g/L to more than 40 g/L. Integrated fermentation and new separation technologies are the suitable way proposed to achieve this yield.
- Reduce the cost of current SA from 1.2 €/kg to less than 1 €/kg at purities higher than 90%. Process integration, and direct SA ester production from fermentation broth may lead to such low production cost.
- Methodologies to obtain, via enzymatic route, new PBS (Polybutylene-succinate) and some of its copolymers with improved performance to current market PBS.
- To improve the cost competitiveness of PHB and PBS biopolymer by optimization of the fermentation process (increasing yield) and the use of waste sugar of sulphite pulping process as fermentation main raw material.
- To use industrial waste by-products as raw materials, no competition with food supply chain exists. Currently, the sugars of spent pulping process are destroyed to avoid interaction with other high value liquor components.
- To enhance the low fire retardant properties of PHB based biopolymers.
- Enzymatic modifications of lignosulfonates (LS) to increase their molecular weight between 5-20 times than the initial one, and thus to improve the processability and fireproof capacity of develop biopolymers.
- Production on new FR additives based on chemical modifications of lignosulfonates (LS) and PHB with phosphate and nitrogen moieties.
- Biopolymer PHB/PBS blends and compounding with different halogen-free fireproof additives, lignosulfonate based fillers and intrinsically FR-polymers to fulfil the fire resistance requirements of the selected case-studies (inner panels of buses, trams, industrial vehicles or trucks)
- Development of multilayer structures to improve the final performance of sandwich panels and reduced the amount of additives, mainly flame retardant.
- Continuous compression moulding in combination with natural fireproof fabrics to produce 3D sandwich panels as a real alternative to current thermoset-glass fibre reinforced panels at low weight (less than 2.30 kg/m^2 in comparison with 2.65 kg/m^2 of current ones).

- Biopolymer sandwich panels that have to meet tough design requirements of the collective passenger/goods transport industry.
- The cost of final panel at industrial level will be lower than 22€/m^2, 15% cheaper than current thermoset based panels.
- Fully recyclable biocompounds, up to a 30% of recycled materials will be added to the virgin material without significant loss of mechanical properties (less than 10%).

Partners involved in the project:

Partner name	Logo / Website	Main role in the project
AIMPLAS Asociación de investigación de materiales plásticos y conexas	AIMPLAS INSTITUTO TECNOLÓGICO DEL PLÁSTICO www.aimplas.es	PHB/PBS blends optimization. Fire resistant compounds based on lignosulfonate fillers, intumescent additives and intrinsically FR PHB copolymers. Co-extrusion and thermoforming tests at pilot plant level. Support co-extrusion case studies. Recyclability analysis, biodegradation tests. Regulatory analysis report.
ULUND Lunds Universitet	www.lunduniversity.lu.se	Establishment/upgrading of genome-scale models to assist for optimized medium (liquor) supplementation targeting optimum productivities. Protocol for the adaptation of microorganisms to substrates containing (potential) inhibitors. Production of succinic acid using the xylose-metabolising yeast
UNICAN Univesity of Cantabria	UC UNIVERSIDAD DE CANTABRIA www.unican.es	Study at lab scale the spent pulping liquor process to increase the amount of hydrolysable sugars and reduce the amount of inhibitors. New source of sugars based on waste sugarcane bagasse.
BIOTREND Biotrend - Inovação e Engenharia em Biotecnologia, S.A.	biotrend Inovação e engenharia em biotecnologia www.biotrend.biz	BIOTREND actively participate in the fermentation process optimization of the PHB and in the process integration activities of the project. BIOTREND perform the technical and economic viability assessment of the process options.
SILICO Silicolife LDA	SILICOLIFE Computational Biology Solutions for the Life Sciences www.silicolife.com	Computational solutions, including text-mining algorithms, to search genomic and bibliographic databases as support to finding candidate microorgansims that are able to produce one of the target biopolymers using xylose as main raw material, preferably isolated from complex residues (more likely to have inhibitors).

AUA Agricultural University of Athens	www2.aua.gr/el	AUA optimise succinic acid production (WP2) regarding fermentation conditions and separation of lignosulfonates and succinic acid. AUA is involved in technology scale-up. Environmental (LCA) studies.
AVECOM Avecom N.V.	www.avecom.be	Technical and economical feasibility evaluation of all tested options and/or combinations and/or integrations of the steps of liquor pre-processing (or not), fermentation, separation, purification.
BANGOR Bangor University	www.biocomposites.bangor.ac.uk	Production and purification of different monomers for PBS-co polymers. Enzymatic production of PBS co-polymers.
NEXTEK Nextek Ltd.	www.nextek.org	NEXTEK participate in the fermentation process optimization and scale-up of Succinic Acid and PBS production and in the process integration activities of the project.
DLAB Daren Laboratories & Scientific Consultants Ltd.	www.darenlabs.co.il	DLAB, using different chemical routes, prepare different PHB copolymers containing chemical modified lignosulfonates and intumescent flame retardant components.
GSOUR Green Source S.A.	www.sniace.com	Optimize the current spent pulping liquor process to increase the amount of hydrolysable sugars and reduce the amount of inhibitors. New source of sugars based on waste sugarcane bagasse.
ADDCOMP Addcomp Holland B.V.	www.addcomp.nl	Scale-up of the new PHB/PBS composites including, compounding process modification, blend compatibilization and provide compounds for multilayer studies and case-studies production and validation.
PROFORM PROFORM IPARI ÉS KERESKEDE LMI KFT	www.pro-form.eu	Study multilayer structures (Co-Extrusion & thermoforming) using combinations of different PHB/PBS compounds containing flame retardant. Case studies definition design, thermoforming tool production and co-extrusion and thermoforming processing

		optimization.
XPA Xperion Aerospace Gmbh	xperion www.avanco.de	Production of flat sandwich panels using their patented Continuous Compression Moulding (CCM) process.
SOLARIS Solaris Bus & Coach S.A.	SOLARIS www.solarisbus.com	Definition and design of at least two case studies using develop sandwich panels for the coach industry. Full case-studies validation.
CRF Centre Ricerche Fiat SCPA	CRF CENTRO RICERCHE FIAT www.crf.it	Definition and design of at least two case studies using develop sandwich panels. Full case-studies validation.