Layman's report

Recysite Project

LIFE15 ENV/BE/000204

Recysite project: Production of fully recyclable and reusable green composites based on bioresins and natural fibres.

Summary

Within the Recysite project it has been demonstrated the production possibility of green composites based on natural fibres sources from agro-wastes, bio-based resin (ELO-epoxidized linseed oil) and humins (a side stream of bio-polymers production). Additionally, it is possible to recycle these composites up to 10 times.

Several real demonstrators were manufactured and validated for the transportation and construction sector. However, high mechanical requirements and fire protection legislation have to be fulfilled in some end applications, for these reasons, the amount of bio-based content varied from 7,5% for railway wagon floor up to 90% for the modular panels and ventilated facades in the construction sector.

This project provides the solutions for the composite industry where bio-based and renewable materials can be introduced in different percentage according to sector requirements, without compromising the quality and also lowering the prices.

Project motivation

Environmental problem

The increase of the awareness of sustainable solutions created demands for more sustainable and efficient materials among industry and European strategic sectors such as transportation and construction. The demand of fuel efficient vehicles, green buildings and environmental regulations to decrease CO_2 emissions drive the growth of composite market.

There are numbers of advantages, already well acknowledged by the several sectors of industry, utilizing of composite designed and produced in fibre-reinforced composite materials instead of metals, such as aluminium or steel. The main profits are higher strength, lower weight and less maintenance. Additionally it is expected that by 2020 more than 1 million tonnes of composite materials will be produced with revenues over EUR 85 bn. In the other hand, present composites face some problems and are not environmental friendly but based on the fossil based materials.

Problem 1

Dependence of petroleum based materials

Current composites applications mainly use petroleum-based plastics. There is also some trend observe to use some bio-based thermoplastics. The **Recysite project is focused on natural fibre and thermoset bio-based resins that are not the main stream products but side products. Therefore the environmental impact is low.**

Problem 2

Management of agricultural waste

In agriculture often only a part of the crop is really harvested and valorised. This is the case for harvesting linseed; a special type of flax that has a high seed yield. The "linseed straw" (up to 2500 kg/hectare), is, for the moment, rarely valorised and either worked into the ground or directly burned on the fields (due to its low disintegration in the ground). Natural fibres used in the project are manufactured from **agricultural bio-waste**. The Recysite bio-based **resins also are produced during a bio-polymer production as the side-stream**.

Problem 3

Absence of methods and technologies for recycling composites materials.

Recent high growth in the use of composites and relatively recent environmental legislation such as the EU-directive for end-of-life vehicles causes increasing demand for recycling techniques that realise the material recycling. **Recysite composites can be recycled**.

Project partners & project location

The project is implemented in Spain, France, Belgium and The Netherlands from July 2016 to June 2019.

Centexbel is the Belgian scientific and technical centre for the textile industry, located in the heart of the Belgian textile industry with strong links to the majority of the textile companies. Centexbel coordinates the project and support Dierickx Visschers in adaptation of natural fibre to novel formulations.

Dierickx Visschers is a family business working with especially natural fibre products, has several in house production technologies with focus on needle felt production.

Avantium is a worldwide recognized leader in catalytic biomass conversion into furanic building blocks and high throughput technology. Two Avantium linked enterprises participates in the project: Avantium Support BV and YXY Technologies BV. In Recysite project they are in charge of the production of natural resins from renewable resources.

IK4-CIDETEC Technology Centre is working in the fields of materials, surfaces and energy. In the frame of the project they are responsible for the adaptation of recyclable composition, use of hardener adapted to biocomposites formulations.

AITIIP Technology Centre is an innovation and technology centre for the development of projects in design and manufacturing of plastic material and transformation processes. The centre is responsible for the adaptation and optimization of processing technologies LRI and manufacturing of samples.

Sispra is a Spanish based SME founded in 1998 and exclusively devoted to R&D and production of composite materials and parts. In the scope of the project they are in charge of validation of proposed solution and manufacturing of proposed demonstrators.

L'Institut de Chimie de Nice IS specialized in materials science and peculiar behaviours of condensed matter. In the frame of the project they are collaborating with Avantium for resins characterization and optimization of thermo-mechanical performances.

How was the project carried out

In order to design and produce the Recysite bio-composites, the following elements were researched and developed:

The elements

A composite material consists of two components, reinforcement and the matrix. The connection of these components into the composite material generates superior properties in comparison to those represented by the individual component.

The main differences in the structure of the present, standard used fossil based composite and the Recysite composite, are shown below in the table.

Comparison standard composite material and Recysite material:

	Current composites	Recysite composites
Layers	Glass or carbon fibre	Natural fibres from agrowaste
Resin	Petrol based resin	Bio-based (Epoxidsed Linseed Oil)
Other components	-	Humins (side-stream of bio-polymer production)
	Not recyclable	Recyclable up to 10 times

Carbon fibre layers

Natural fibres

Bio-based resin

Let's go in deep and understand how these materials interact.

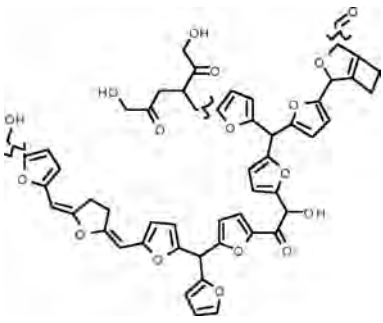
Textiles

Different natural fibres were tested in the form of nonwovens such as linseed fibres (a good quality and expensive one and a lower quality and cheaper one), flax fibre (side product of long flax fibre production), hemp and the recycled jute (the cheapest fibre). The natural fibre based reinforcement offers quite good mechanical properties and additionally these fibres have much lower density (1.3-1.5 g/cm^3) in comparison to glass fibre (2.5 g/cm^3).

Resins

The primary functions of the resin are to transfer stress between the reinforcing fibres, to act as a glue to hold the fibres together and to protect the fibres from mechanical and environmental damage.

Standard resins used in reinforced polymer composites are petrol based thermosets. In the frame of the Recysite project **these resins are bio-based**, consists of ELO (Epoxidized Linseed Oil) and humins, which are a side-stream of the biopolymer production.



Humins molecular structure

Humins are one of the main innovations introduced in the project. Humins, besides being bio-based, are a biopolymer production side-stream that has low commercial value. Introducing them in the resin allowed us to at valorise this product, secondly to reduce the proportion of ELO and finally to add new characteristics to construction composites parts e.g. fire-retardant properties.

Finally a **hardener** is used to accelerate the curing process.

How to prepare a good bio-based resin:

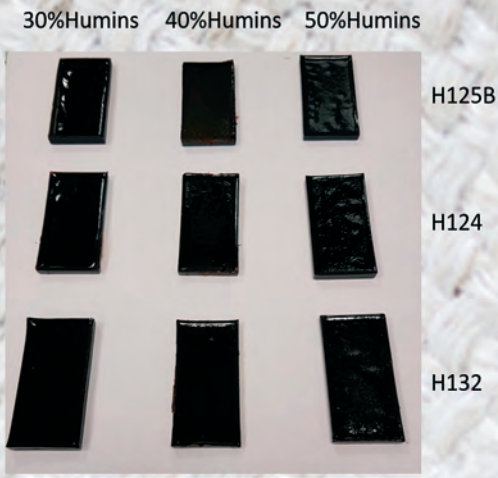
1. **ELO and hardener** must be, at first, mixed together at room temperature in the required proportion. This mixture has to be stirred for 10 minutes at room temperature. A change of coloration should be observed, as indication of a chemical reaction.

2. The mixture containing ELO and hardener has to be mixed with the **humins** at at 80°C. The reaction blend should be thoroughly mixed, until a homogeneous phase is obtained. The resin should reach 80°C under stirring and the temperature should be maintained for 5 minutes.

3. The resin should present small bubbles, again, indicating the chemical reaction. The resin can now be poured into molds at 80°C.

4. The **curing process** takes 4 hours at 80°C and it is followed by post curing at 130°C until completely hardened.

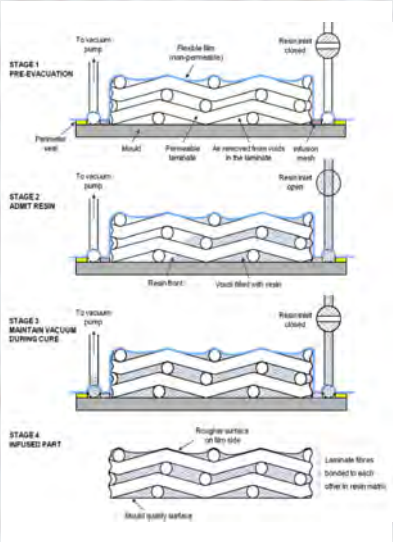
Different mixtures were prepared and tested allowing to reach the final combination of the resin componens represented by the composition consists of 50% humins + 30% hardener + 20% ELO.



50% humins demonstrated higher mechanical properties.

Composites production

Liquid Resin Injection (LRI) is a technique used for the impregnation of natural fibre based structures by the resin. In this technique a vacuum chamber is generated in order to apply some pressure to the piece and pump the resin into the mould, due to the pressure gradient. This way the resin impregnates the reinforcement in the mould.

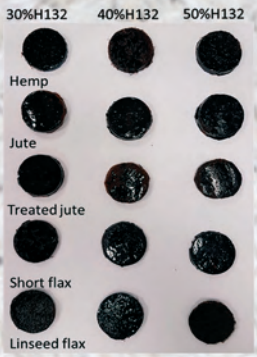


After the reaction process of the resin components is finished the resin solidifies and a unified rigid composite is created. The reinforcement has to fulfil some criteria's to create the good adhesion characteristic such as some porosity of the material, good compatibility with the resin and etc.

Typical materials are inorganic fibres (with glass and carbon fibre being most common) but in this project we have focused on natural fibres such as flax and combinations of natural fibres with other materials such as closed cell foams and honeycomb.

Within this project different combinations of mixtures and fabrics were prepared such as:

- **Non woven bio-fabrics**: hemp, jute, linseed flax good quality, linseed flax lower quality and short flax.
- **Woven bio-fabrics**: Linseed flax (twill 2x2).
- **Glass fibre**: Bidirectional, unidirectional and a general configuration.



Validation of composites in laboratory

Different trials were made with different natural fibres (woven and non-woven structure), glass fibre, bio-based and petrol-based resins and different humins concentration. The objective was to obtain the highest bio-based content in the final piece but mechanical requirements for the construction and the transportation sector were taken into account.

1. Non-woven materials + bio-based resin + humins:

After some mechanical evaluation it was concluded that the natural fibre based nonwoven materials are not suitable if the high mechanical properties are obligatory. This combination is not able to meet the requirements needed for the transport sector application. Nevertheless this combination is perfectly useful for construction sector.

Non-woven composite

2. Bio-based resin + flax fabric + humins:

This second execution route consists of **biobased resin and a flax fabric instead of non-woven materials**. This arrangement assures good flexural and the tensile properties of the pieces In parallel the control specimens were produced in order to compare the properties separately of the bioresin and the properties of the linseed flax fabric.

Once the results of the flax fabric + biobased resin were studied, it was seen that this arrangement it is not enough to fulfil the requirements for the transportation sector. Due to this, Recysite project moves to the third alternative.

3. Flax fabric + petrol-based epoxy resin + humins:

The third option was **the flax woven material combined with a commercial epoxy resin** partially replaced by **humins** up to a 25% in the first stage. This combination as presented below fulfils the transport sector requirements.

The mechanical properties of the obtained materials were studied, especially flexural and tensile properties.

Comparison of the tensile strength of glass fibre.

As the graphic depict, the differences between materials consists of glass fibre or flax fabric epoxy and humins are not significant, therefore it is possible to conclude that **linseed flax fabric might substitute the glass fibre material in some applications with high mechanical requirements.**

It was also proven that natural fibre based **non-woven reinforcing bio-based resins are ready-to-use when no mechanical requirements are needed**. This type of composite structures can be used in the construction sector for ventilated façade and modular walls and roofs.

Validation of composite products in real installations

Once materials and production have been validated in laboratories, real demonstrators were manufactured for both types of applications in the transportation and construction sector.

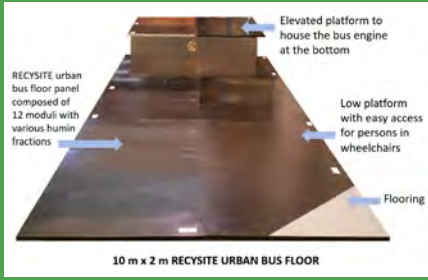
MODULAR PANEL			BUS FLOOR			WAGON FLOOR		
Textile	Resin	Humins	Textile	Resin	Humins	Textile	Resin	Humins
Non woven	Bio-based	50%	Non woven	Bio-based	50%	Non woven	Bio-based	50%
ok			No ok			No ok		
			Flax fabric	Bio-based	50%	Flax fabric	Bio-based	50%
			No ok			No ok		
			Flax fabric	Petrol based	15%	Flax fabric	Petrol based	15%
			ok			No ok		
						Glass fibre	Petrol based	15%
						ok		

Transportation sector

An optimised bio-composite material has been developed to fabricate urban bus and a wagon floors.

Different tests with series of composites materials were evaluated as well as the maximum bio-based content. This research included mechanical tests (tension, compression, bending, sandwich out-of-plane shear), humidity content, fire and smoke, acoustic insulation, adhesion to flooring, fibre content and porosity.

For the wagon floor due to fire & smoke requirements only glass fibres were used. However the bio-based content due to incorporation of humins was increased up to 15%.

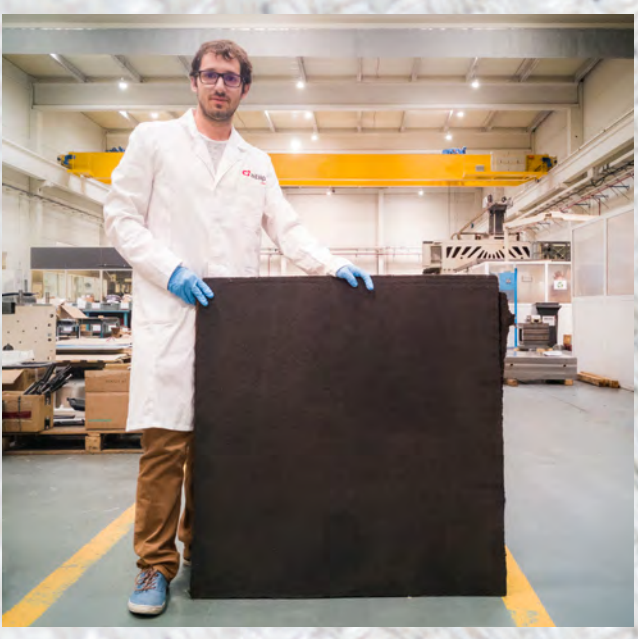


Bus floor. Recysite composites were used to manufacture bus floor. Different trials and materials were tested and finally the proper blend was woven into flax fabrics with a recycled PET core and 15% humins.

The validation test results showed that flax fabrics with epoxy resin and 15% humins with a recycled PET in the core is a competitive material system for bus floor applications.

Construction

The Recysite project aimed to generate modular wall and roof panels for the construction sector with a 70% bio-based resin and 100% non-woven based on natural fibres. In this way it is possible to achieve a **specimen containing up to a 90% wt. bio-based content**.



The use of non-woven based on natural fibres as reinforcement reduces the price of the final composite structure. Additionally, less compact structure of natural fibre based non-woven materials provide lower thermal conductivity than the woven material. This effect makes of the non-woven structures a very useful material not only for **thermal insulation applications**, but also for **sound insulation**.

One of the main possible obstacles preventing to use natural fibres as a material for the construction sector was their low resistance fire. However, the performed screening tests, following ISO 11925-2 (2010) using the bio-based composite have shown that the material has **fire-resistant properties**. According to this screening test we could conclude that the material has reached class E but BCD, S1 and D0 category is possible after the final fire resistance evaluation.

Recysite fire-resistant properties

MAIN CLASIFICATION		SMOKE		DROPS
A1				
A2		s1		d0
B	+	s2	+	d1
C		s3		d2
D				
E		+		d2
F				
NPD				

Results

	Wagon floor	Bus floor	Construction panel
Textile	Glass fibre	Flax fabric	Non-woven
Resin	Epoxy	Epoxy	ELO
Humin	15%	15%	50%
Biobased %	7,5%	57,5%	90%
Cost (compared to non Biobased composite)	-10%	-15%	-35%

The project has demonstrated that green composites are industrially feasible and economically profitable. The grade of bio-based content depends on the mechanical requirements of the manufactured pieces (and also on legal requirements about fire regulations). **Anyhow a minimum of 7,5% bio-based content is possible to incorporate to the high end applications. For some other applications up to 90% bio-based content is reached**.

These composites use **natural fibres**, which origin is agrowastes from different crops. These textiles are combined with a **bio-based resin**, made from epoxidized linseed oil, humins (a bio-polymer production side-stream) and a hardener.

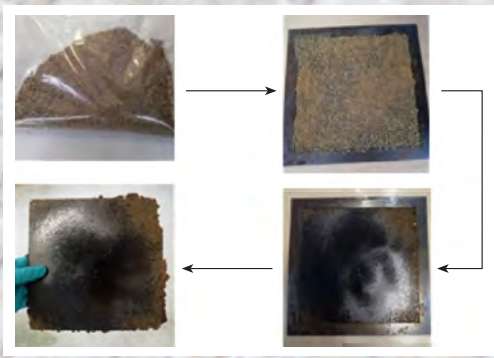
Manufactured pieces can be used in the **construction** sector as modular wall or roof panels, with 90% of bio-based content have shown good fire retardant properties, and noise and thermal insulation characteristics. Additionally, NF based non-wovens fibre reduces the cost of the panel.

However, when mechanical requirements are needed, green composites need reinforcements that could be in example a recycled PET core or glass fibre and petrol-based resins.

This is how greener composites can be also installed as **wagon** and **bus floor** with a 57,5% of bio-based content (reinforced with a PET or glass fibre core due to mechanical requirements).

One of the objectives of the project was to obtain **fully recyclable bio-based composite materials**.

This has not been possible at an industrial scale, since initially selected formulation was a solid paste that did not allow working with the described manufacturing techniques. In order to manufacture the fully recyclable bio-based composite materials that were aimed within the project, some additional work was done at a smaller scale: ELO was substituted by a commercially available bio-based epoxy resin that allowed obtaining a liquid formulation mixture and, therefore, working with commonly employed manufacturing techniques. The elevated cost of this formulation, kept the work at a smaller scale. Different natural fibres were used as reinforcement materials (flax, hemp or jute).



The obtained composites can be grinded to powder and a new composite can be prepared from this powder just by applying heat and pressure. **So far, 10 recycling cycles have been done** on a flat laminate with flax fibre reinforcement and still going (the material has the same thermal properties as those of the pristine material).

Environmental impact and socio economic assessment

In Recysite project several strategies for increasing the sustainability of producing composites haven been developed:

1. Natural reinforcements based on vegetable fibres produced from agro wastes.
2. Resin matrix containing natural oils coming from the valorisation of agro wastes
3. Composites formulations containing side products of the production of bio-based polymers
4. Recyclability of the green composites (so far not an industrial scale)
5. A combination of the aforementioned strategies

During the project the suitability of each of these approaches have been demonstrated in several sectors, defining the most promising applications for a quick market acceptance:

- **Railway** sector is prone to those development including fire retardant requirements, such as humins, therefore Recysite will face the industrialisation focused on conventional fibre and resin but with the highest amount of humins.
- **Road** sector is welcome to lightweight structures in which linseed flax fabric is a good solution. In addition, humins will be also included to increase the bio-based content.
- **Construction** sector, for applications with low mechanical properties and high isolation standards is looking for solutions with the maximum amount of bio-based content. Therefore, the combination of non-woven natural fibres, ELO and humins will be well accepted.

Dissemination activities

Recysite implemented an intensive dissemination campaign, designing different communication tools as a website **(www.recysite.eu)**, brochure, posters, notice boards, videos, newsletters and profiles in different social networks.

It is also important to highlight that the project participated in many conferences and fairs as MatComp, JEC World, Equiplast and Composiforum...

In total more than 50 publications in social networks and project website were published, within this frame the project gained 150 followers in social networks and 1.400 unique visitors in the website. It was also presented in more than 15 national and international conferences and distributed more than 2.500 brochures.

The LIFE programme

LIFE is the EU´s financial instrument supporting environmental and nature conservation projects throughout the EU as well as in some candidates, acceding and neighboring countries. Since 1992, LIFE has co-financed some 4,500 projects, contributing approximately 3,400 million euros to the protection of the environment.

http://ec.europa.eu/environment/life/

LIFE15 ENV/BE/000204 RECYSITE

Production of fully recyclable and reusable green composites based on bio-resins and natural fibres.

This Project is co-financed by the European Union through the LIFE Programme.

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% EU contribution: 1.001.411€

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