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## D.3.5.2

# Life Cycle Assessment (LCA) for the different biocomposites production routes

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#### **Abstract**

Keywords: acrylic; biocomposites; lactide; LCA; life cycle assessment

The Activity Description in the bid document said:

"Eco impact of biocomposites studied will be analysed through Life Cycle Analysis for which standard software (SimaPro) is available.

Since only demonstrators are produced under pilot conditions, and no real industrial end products are produced a full LCA will not be possible or makes little sense. Instead we will concentrate on the main differences between the used ingredients and the newly developed production routes, extrapolating how the upscaling and will further optimize production conditions. So differences between LCA for natural fibres and glass fibres, for biopolymers and bioresins or oil-based plastics, injection moulding versus layer by layer deposition, ....

Further the improved recycling potential as well as durability and ecotoxicity impacts are taken into account".

The SimaPro software, and ecoinvent life cycle inventory (LCI) database, were acquired and first used for this project. However, the LCI data has now been identified to not be as robust as should be expected from commercial product. In particular while the functional unit is clear, the goal and scope, and the system boundary are inadequate to provide confidence in any results generated. In consequence, the authors caution that any results in this report are qualitative. In particular, cross-comparisons between different material systems are unlikely to be valid.

There was an intention to use a 5G telecommunications dome as a demonstrator component. The commercial component is rotomoulded polyolefin. The infused component would be flax fibre reinforcement in a variety of resins. In order to realise the bio-based product, where the matrix would be poly(lactide) processed at >120°C, a high-performance mould tool is required (which could be used for all matrix systems). In the event, the company contracted to produce the tool encountered a series of technical and supply chain issues which delayed the delivery of the tool beyond the end date of the Work Package.

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#### List of abbreviations

DG Directorate-General.
EC European Commission.
ENV (EC DG] for Environment.

EU European Union.

FEA Finite element analysis. FLOW FLoating Offshore Wind.

FM Flow medium.
FVF Fibre volume faction.

GF Glass fibre.

GFPP Glass Fibre Peel Ply. gsm grams per square metre.

ILCD International Reference Life Cycle Data System.
ISO International Organization for Standards (Geneva CH).

ISP In Situ Polymerisation.
JET James E Thomas.

JRC Joint Research Centre (JRC, Ispra ~ Italy).

LCA Life Cycle Assessment.

LCI Life Cycle Inventory analysis.

LCIA Life Cycle Impact Assessment.

MATS347 University of Plymouth undergraduate module.
MIFT Monomer Infusion under Flexible Tooling.

MW Molecular Weight. NFM No Flow Medium.

NREL National Renewable Energy Laboratory (Golden CO, USA).

PY Princess Yachts (Plymouth, UK).

PP Peel ply.

RIFT Resin Infusion under Flexible Tooling.

SCRIMP Seeman Composites Resin Infusion Molding Process.

SDLPRT File format for a 3D model created by Dassault Systemes SolidWorks.

SMB Smeaton Building (University of Plymouth).

STEP Standard for the Exchange of Product Data (a 3D model file ISO 10303 standard

exchange format).

VARTM Vacuum Assisted Resin Transfer Moulding.

V<sub>f</sub> Fibre Volume Fraction.

#### 1: Introduction

The InterReg SeaBioComp project (2 Seas Mers Zeeën 2S06-006) seeks to develop durable bio-based composites for the marine environment. The University of Plymouth activity in WP1 was focussed on the development of *in situ* polymerisation (ISP) during monomer infusion under flexible tooling for bio-based polymers, and in WP3 on life cycle assessment (LCA).

#### 1.1 Manufacture of large composite structures

Large composite structures (marine vessels to 80 m length and wind turbine blades to >115 m length (Lowde et al) are manufactured using Resin Infusion under Flexible Tooling (RIFT) (Williams et al, 1996. Cripps et al, 2000. Summerscales and Searle, 2006. Beckwith 2007. Summerscales 2012. Hindersmann 2019, Tamakuwala 2021.). The process is often referred to as Seeman Composites Resin Infusion Molding Process (SCRIMP) or Vacuum Assisted Resin Transfer Moulding (VaRTM). The system normally uses a thermosetting resin (e.g. unsaturated polyester, vinyl ester, epoxy or phenolic) which sensibly meets the service requirements but is difficult to deal with at end-of-life (Summerscales et al 2015).

1.2 *In Situ* Polymerisation during Monomer Infusion under Flexible Tooling (ISP-MIFT) The end-of-life issue could be resolved by the use of thermoplastic matrix composites. However, the "preferred melt viscosity range for most thermoplastics forming processes" is 100 000 - 1000 000 mPa.s. Given that the limiting viscosity for RIFT processes is 800-1000 mPa.s (Becker undated. Pearce et al 1998.), the melt infusion manufacture of large thermoplastic structures is impractical. A number of thermoplastic matrix systems are amenable to *in situ* polymerisation (van Rijswijk and Bersee 2007). Qin et al (2020) down-selected those systems to only include those which can be processed below the degradation temperature of natural (plant) fibres and were sensibly useful in wet conditions. The most appropriate systems for *In Situ* Polymerisation during Monomer Infusion under Flexible Tooling (ISP-MIFT) appear to be acrylic (methyl methacrylate/MMA) and lactide systems.

#### 1.2.1 Acrylic matrix

The acrylic system is a "drop-in" alternative for composite manufacture with the above resin systems. The SeaBioComp WP1 demonstrator was a floating offshore wind (FLOW) turbine blade at scale 1:50. This model represents the NREL 5 MW reference wind turbine. The blades are not scaled geometrically, but instead have been adapted to produce Froude scaled thrust, in spite of much lower Reynolds numbers at reduced scale and when using Froude scaled wind. The blade offsets and aerodynamic profiles have been published (Kimball et al 2014) with additional detail of the application in Guichard (2022).

Bhudolia et al (2017) investigated the optimisation of infusion manufacture of carbon fibre fabrics with a room temperature cure epoxy or Elium<sup>®</sup> MMA thermoplastic matrix systems. They identified that a three-stage vacuum during infusion and consolidation (90 mbar pre-infusion, 500 mbar infusion on mesh, 400 mbar post flow mesh, 330 mbar consolidation) led to panels with consistent fibre volume fractions and minimal void content.

#### 1.2.2 Lactide matrix

Poly(lactic acid), also known as poly(lactide), is a promising matrix system for durable composites with a glass transition temperature  $\sim\!65^{\circ}\text{C}$  and a melt temperature  $\sim\!175^{\circ}\text{C}$ . The lactide monomer (a cyclic dimer of lactic acid) is amenable to infusion albeit at elevated temperatures. The lactic acid monomer (CH<sub>3</sub>.CHOH.COOH, molecular weight 80) will release significant quantities of water (molecular weight 18 = 22.5% of lactic acid MW) during dimerization or polymerisation and is thus unsuitable for MIFT processes.

The lactide monomer for WP1 was sourced from Total Corbion (Gorinchem, The Netherlands). The company has rebranded as Total Energies (Gorinchem, The Netherlands) and divested the supply of the monomer. Initial advice was that an alternative source of monomer was Corbion (Barcelona, Spain) but they in turn recommended [the Total Corbion spin-off] Corbion (Gorinchem, The

Netherlands). Alternative sources for the monomer were investigated but the majority of suppliers focus on scientific/pharmaceutical grade at process up to €18000/kg (gold is <€50000/kg!).

The original lactide was supplied with a Product data sheet that recommended storage in dry conditions as the monomer is deliquescent (absorbs moisture from the air and dissolves in it). The new lactide has similar requirements in the Product Data Sheet, but the labelling recommends storage under dry nitrogen, leading to additional handling costs (and a small increase in environmental burdens).

The literature on the use of poly(lactide) in Liquid Composite Moulding processes beyond the SeaBioComp process is limited to a single paper by Louisy et al (2019). They produced glass fibre reinforced poly(lactide) but the focus was on degree of polymerisation rather than mechanical performance. As such, the MIFT process for lactide was, and remains at, Technology Readiness Level (TRL) 1, with the SeaBioComp project possibly moving the technology to TRL2.

#### 2: Life Cycle Assessment for composites (principal author JS)

#### 2.1 ISO Standards

Life Cycle Assessment (LCA) is a methodology used to assess the environmental impacts associated with the complete life cycle (raw materials acquisition, product manufacture, use phase, and disposal) of a product, process, or service. LCA is the subject of a series of International Standards. ISO14040:2006(E) defines four different phases for Life Cycle Assessment. Brady (2005) defines the four phases as follows:

- Goal and scope definition of the LCA: the goal and scope of the study are defined in the context of the intended application.
- Life Cycle Inventory analysis (LCI) phase: this involves the collection of data, and the calculation procedures, resulting in a table that quantifies the relevant inputs and outputs of the analysed system.
- Life Cycle Impact Assessment (LCIA) phase: this translates the results of the inventory analysis into environmental impacts (e.g. eutrophication) with the aim of evaluating the significance of the respective impacts.
- Life Cycle Interpretation phase: conclusions and recommendations for decision makers are drawn from the inventory analysis and impact assessment.

The framework set out in the standard then requires:

- reporting and critical review of the LCA
- limitations of the LCA
- relationships between the phases of the LCA, and
- conditions of use of value choices and optional elements.

ISO14040:2006(E) suggests that when "setting the system boundary, several life cycle stages, unit processes and flows should be taken into consideration, for example, the following:

- acquisition of raw materials
- inputs and outputs in the main manufacturing/processing sequence
- distribution/transportation
- production and use of fuels, electricity and heat
- use and maintenance of products
- disposal of process wastes and products
- recovery of used products (including reuse, recycling and energy recovery)
- manufacture of ancillary materials
- manufacture, maintenance and decommissioning of capital equipment

ISO 14044:2006 then sets the requirements and guidelines for the conduct of an LCA. The standard defines minimum requirements and is open to wide interpretation by the individual experts,

practitioners and data developers. The range of important options that can be selected within a specific LCA, leads to differences in the consistency, reliability and comparability of the assessment outcomes. Furthermore, the assumptions underlying the methodology behind the life cycle data can differ widely. Data from different sources is rarely comparable so relative assessments are fundamentally flawed.

#### 2.2 International Reference Life Cycle Data System (ILCD)

The Joint Research Centre (JRC, Ispra ~ Italy) and the European Commission Directorate-General for Environment (EC DG ENV) took the initiative to develop a more coherent system. The International Reference Life Cycle Data System (ILCD) aims "to provide guidance and standards for greater consistency and quality assurance in applying LCA" established through a series of extensive public and stakeholder consultations (a parallel draft literature review is included at Appendix A).

#### 2.3 Product/Organisational Environmental Footprint

Ekvall (2016) has criticised the internal inconsistency of the ILCD Handbook and Ekvall (2022) has suggested that the frameworks for Product/Organisational Environmental Footprint have largely replaced the ICLD Handbook (European Commission, 2021).

#### 2.4 Life Cycle Inventory databases

Ecoinvent (https://ecoinvent.org/) claims to have "the world's most consistent and transparent life cycle inventory database. However, close examination of the data appropriate to the SeaBioComp analysis has generated doubt about the data quality. A preliminary summary of the published data pertinent to composites has been compiled (Summerscales, 2022). Outside the project, esteemed colleagues at École Polytechnique Fédérale de Lausanne (EPFL, Switzerland) and at Katholieke Universiteit Leuven (KUL, Belgium) have opened discussion about the parallel issue of the wide diversity and weak audit situation for carbon fibre data in the commercial databases.

One useful resource is the flowchart on the Plastics Europe website: <u>Eco-profiles comprise Life Cycle Inventory datasets (LCI) and Environmental Product Declarations (EPD) for plastics</u>. The Eco-profile datasets are the source for the disaggregated ecoinvent database and the aggregated SimaPro and Shera (previously Gabi) databases.

#### 2.4.1 Unsaturated polyester resins

The information for unsaturated polyester resins can be traced back to a single report (Rietveld et al 2014). The goal of the project was to "develop and build a data-set of resins as produced in Europe containing the (average) eco-impact indicators for each resin. The functional unit of the analysis is 1 ton of resin. Included life cycle phases are raw material extraction, production of materials, transport to production location, and the production of the resins. This is also called a "cradle to gate analysis". The system boundaries are claimed to be displayed in Figure 1 although that diagram is simply a "Schematic representation of the analysed system". Further, the report states that "The data inventory as obtained from the producers is available on request". The request for that information was not satisfied by the document provided.

#### 2.4.2 Epoxy resin

The Epoxy Resin Production (liquid, global) dataset documentation points to a company report (Werk Gendorf, 2015) which is not available via the <u>company server</u>., although reports for 2016, 1017 and 2021. The ecoinvent document notes that "Epoxy resins can have different characteristics, these depend on additional products that can be added to the liquid resin". The database does not have specific inventory data for epoxy hardeners (curing agents) or sensible alternatives that might act as proxies.

## 3: Life Cycle Assessment using SimaPro software with the ecoinvent database (principal authors Nanting Yu and Ruadan Geraghty)

#### 3.1 Life Cycle Assessment

The core report on Life Cycle Assessment is included at Appendix B, supported by Appendix C and Appendix D.

#### 3.2 Parallel research activity

In activity parallel to, and informing, SeaBioComp WP3 at University of Plymouth there are projects also undertaking life cycle assessment. Princess Yachts (PY) have sponsored a PhD student to undertake Life Cycle Assessment of a yacht production line. That work is comparing SimaPro to MarineShift360 LCA software. The PY work is also taking a deep critical look at data quality across a number of databases (ecoinvent, openLCA Nexus, Sphera (was GaBi), etc.). An abstract submitted to the 23<sup>rd</sup> International Conference on Composite Materials to be held in Belfast in 2023 is included at Appendix E. The InterReg INdIGO project is using SimaPro in the context of handling Abandoned Lost and Discarded Fishing Gear. (ALDFG).

#### 4: Optimisation of ISP-MIFT processes

Optimisation of the ISP-MIFT process was undertaken with a view to minimising environmental burdens arising from the processes. Infusion of flax composites resulted in the flow medium becoming permanently attached to the laminate even with sensivble iuse of peel-ply and release film. Natural fibres tend to produce lower fibre volume fractions than synthetic reinforcements fabrics. The lower FVF results in higher permeability laminate stack which can be sensibly infused without flow medium (RIFT I: resin infusion under flexible tooling in the plane of the reinforcement) (Appendices F-H).

Subsequent experiments were conducted with bio-epoxy (Appendix I), conventional epoxy (Appendix J) and the Elium acrylic system (Appendix K) using the Plymouth standard RIFT procedure without flow medium (Appendix L).

## 5: Demonstrator mould tool and energy monitoring

The demonstrator chosen for the ISP-MIFT study was a 5G telecommunications dome to protect the electronic systems for a marine communications network. The commercial 5G dome is a 7 mm thick rotomoulded poly(ethylene) component. The substitution of a stiffer material will permit a reduction in wall thickness. Initial calculations indicate that a 4 mm flax epoxy component will have equivalent structural stiffness (Appendix M).

The aerospace industry uses composite tooling for infused components but the mould tool does not normally incorporate integral heating. For example, the RTM-6 epoxy resin system is normally preheated from solid at ambient to become viscous liquid at 80 °C to degas. It is then infused (<5 mbar) into a mould at a constant temperature between 120-140°C (Parsons et al, 2022), then undergoes a free-standing oven post-cure at  $\sim$ 180°C,

The mould tool for the demonstrator component was ordered from a reputable composites processing equipment supplier in December 2021 after the appropriate tendering process. In February 2022, the company advised "with this revised schedule with earlier design & build slots we are looking at completion in w/c 30 May with commissioning possible at ... or your facility immediately after. Of course we will take any and every opportunity to improve on this timeline and keep you updated at key milestones, or on a regular update schedule if you prefer". The equipment acquired to monitor energy usage during the process is described at Appendix N and Appendix O.

The mould tool for the demonstrator component proved to be less straightforward than anticipated. The cost of metal tooling would have exceeded the available budget. The company contracted to build the mould tool had experience of infusing epoxy tooling with standard maximum operating temperatures of 150°C (and an expectation that the glass transition temperature would be ~170 °C (and hence an expectation it would "stand a few cycles up to 170°C"). An epoxy composite tool, complete with oil heating, was procured. However, a series of production/quality issues lead to the third iteration mould tool not being available before conclusion of the work package on 31 October 2022. The mould tool required for ISP-MIFT manufacture of poly(lactide) matrix composites has emerged as needing state of the art composite mould tool (or an unaffordable metal mould tool) for poly(lactide) infusion in the range 120-180°C. The reinforcement geometry for the demonstrator component creates low permeability volumes which are difficult to fill, and in the limit remain as dry spots. The high-temperature resin system is more viscous than is normally used for an infused tooling resin (Confidential-to-the-Consortium Appendix P).

An abstract on the prospects for ISP-MIFT of the lactide monomer has been submitted to the 23rd International Conference on Composite Materials to be held in Belfast in 2023 is included at Appendix Q.

#### 6: Summary

Two monomers were identified as candidates for ISP-MIFT. Bio-based acrylic was being investigated in academic laboratories at the start of the project. Bio-based grades are staring to become available commercially but there is no infusion grade to date. Lactide is bio-based by default but requires processing at elevated temperatures (hence higher energy consumption with consequent environmental burdens).

Life Cycle Assessment (LCA) of materials for composites is constrained by data quality issues. Initial assessments have been undertaken with the available data but the authors are cautious about release of the information beyond the consortium. There is already a significant number of publications in the public domain which need to be critically analysed to ascertain the value of the information presented.

The practical data acquisition to inform the Life Cycle Assessment has been constrained by the political context (Appendix Y).

### Acknowledgments

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## **Appendices**

## Appendix A: Literature review (principal author John Summerscales).

#### Life cycle assessment for composites: a critical review and polemic

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#### Abstract

#### **Keywords**

Life Cycle Assessment; Allocation, Proxy

#### Introduction

The world is currently subject to two existential crises: (i) Climate Change and (ii) Loss of Biodiversity. The United Nations Brundtland Report (Our Common Future, 1987) [1] defined sustainability as "meeting the needs of the present without compromising the ability of future generations to meet their own needs". After decades of international cooperation, the UN Sustainable Development Summit in September 2015 adopted the 2030 Agenda for Sustainable Development, with 17 Sustainable Development Goals (SDG).

Technical, Economic, Environmental, Social and Governance (TEESG)

The development of products should seek an optimum solution with respect to the technical, economic, environmental, social and governance (TEESG ~ "the banana anagram") criteria:

- Technical: conformance to the performance targets set by a sensible Product Development Specification.
- Economic: industry, innovation and infrastructure (SDG9)
- Environmental: responsible consumption and production (SDG12)
- Social: good health and well-being (SDG3). Decent work and economic growth (SDG8).
   Reduced inequalities (SDG10)
- Governance: Administration with a sound ethical and moral basis, especially openness, transparency and no corruption. Peace justice and strong Institutions (SDG16).

Truth *vs* the viewpoint of the man on the street

In the context of governance, some currently accepted views might be challenged:

- In the UK, the government is selected by the First Past The Post (FPTP) electoral system, sits on benches across from the opposition in the House of Commons, and "whips" members of parliament through the lobbies when voting. This repeatedly produces administrations with a short-term focus (winning the next General Election) at the expense of long-term strategic planning for the benefit of the majority of the population of the country.
- The Western media (television and newspapers) are controlled by a small group of powerful
  people who hold a disproportionate amount of wealth, privilege and political power.
  Advertisements enable their control of the messages the public do see, but the more
  something is advertised, the less it is essential to a good life.
- In China, President Xi Jinping declared that China had eliminated poverty in 2020. However, the Western media portray the situation for the Uyghur population in the Xinjiang region as "demographic genocide". Reporting of the situation can often be traced back to work by Adrian Zenz which has been revealed to have flagrant data abuse and outright

falsehoods. Would you trust the story of the "troubles" in Northern Ireland if told solely by the Irish Republican Army?

#### **Life Cycle Assessment**

The development of products or services in the context of TEESG guidelines, should be demonstrated by systematic analysis, not reliant on the uninformed perceptions of the public. The Stern report identifies agrochemicals as the second most energy intensive industry producing physical entities (i.e. not energy) after building materials. With the exception of the small proportion of organic produce, the whole food industry has high dependence on those agrochemicals. We need to exercise similar caution in all data provided for Life Cycle Assessment!

The ISO14040 series of standards describe the principles and framework for life cycle assessment (LCA). ISO 14040:2006 [2] covers LCA studies and life cycle inventory (LCI) studies, but does not describe the technique in detail, and does not specify methodologies for the individual phases of the LCA. ISO 14044:2006 [3] specifies requirements and provides guidelines for the LCA. Brady [4] defines the four phases of LCA as follows:

- a. **Goal and scope definition of the LCA:** the goal and scope of the study are defined in the context of the intended application.
- b. **Life Cycle Inventory analysis (LCI) phase:** this involves the collection of data, and the calculation procedures, resulting in a table that quantifies the relevant inputs and outputs of the analysed system.
- c. **Life Cycle Impact Assessment (LCIA) phase:** this translates the results of the inventory analysis into environmental impacts (e.g. eutrophication) with the aim of evaluating the significance of the respective impacts.
- d. **Life Cycle Interpretation phase:** conclusions and recommendations for decision makers are drawn from the inventory analysis and impact assessment.

The framework set out in the standard then requires:

- e. reporting and critical review of the LCA
- f. limitations of the LCA
- g. relationships between the phases of the LCA, and
- h. conditions of use of value choices and optional elements.

Wolf et al [5] provide an overview of the The International Reference Life Cycle Data System (ILCD) Handbook. The ILCD Handbook [https://eplca.jrc.ec.europa.eu/ilcd.html] provides technical guidance for detailed Life Cycle Assessment (LCA) studies in the context of the international standards. The Handbook provides the technical basis for the derivation of product-specific criteria, guides, and simplified tools. The principle target audience for the Handbook is LCA practitioners, but the advice is equally relevant to technical experts in both the public and private sectors who deal with environmental decision support related to products, resources, and waste management. The main ILCD Handbook comprises of ten parts (main text/total pages) listed below as sequenced in §5 of [5]:

- EUR24708 General guide for LCA detailed guidance (322/417 pp) [6].
   Based on, and conforms to, the ISO 14040 and 14044 standards on LCA, this guide provides technical guidance for detailed LCA studies, and provides the technical basis to derive product-specific criteria, guides, and simplified tools.
- EUR24378 General guide for LCA provisions and action steps (124/163 pp) [7]. This "cook-book" style document provides the provisions and action steps for daily reference when performing ILCD-compliant, detailed Life Cycle Assessment (LCA) studies.
- EUR24709 Specific guide for LCI data sets (105/142 pp) [8].
   This guide provides more details for the generation of specific types of data, e.g. it describes

- how to create LCI data sets that best reflect the average situation regarding emissions and resource consumption.
- EUR24586 Framework and requirements for LCIA models and indicators (82/116 pp) [9]. This guide lists the criteria to be used in assessing the impact assessment models and indicators, in terms of scientific robustness and stakeholder acceptability.
- EUR24571 Recommendations for LCIA in the European context (107/159 pp) [10].
   This guide describes the indicators and models recommended for LCIA to be used for the ILCD methods.
- EUR24710 Review schemes for LCA (14/34 pp) [11]. This guide includes detailed provisions on the review types required for various life cycle data and studies in the form of review schemes. It conforms to the ISO 14040 and 14044 and other related standards on LCA and its applications.
- EUR24379 Reviewer qualification for LCI data sets (15/34 pp) [12].
   This guide specifies the requirements on the experiences and expertise of reviewers for LCI datasets.
- [not released] Review, scope, methods and documentation.
- EUR24384 Nomenclature and other conventions (37/58 pp) [13].
   This guide provides the detailed provisions for nomenclature of emissions, resource, processes, units and some other conventions (e.g. flows and units classification) in support of LCA practices.
- [not released] Terminology.

#### Key supporting documents include:

- EUR24380 Compliance rules and entry-level requirements (12/18 pp) [14, 15].
- EUR24381 Documentation of LCA data sets (46/57 pp) [16].
- EUR25167 Characterisation factors of the ILCD recommended LCIA methods (17/31 pp) [17]. This guide supports the correct use of the characterization factors (CF) and points out some known limitations. The CF dataset, entailing metadata and *errata-corrige*, is available as ILCD formatted xml files or MS Excel files. The complete list of CF is available in the tool page (ILCD developer section).
- Analysis of Environmental Impact Assessment methodologies for use in LCA (65/115 pp) [18].
   This guide provides an overview of the impact assessment methods as they existed in 2010, and their main features.
- Management of UUID [Universally Unique Identifiers] and version number of data sets (19/25 pp) [19]

The LCA Cookbook [20] presents a selection of provisions and actions from the ILCD Handbook to permit an LCA practitioner to undertake a typical process LCA within the ISO framework (in just 86 pages!).

## Goal and scope definition of the LCA

System boundary

ISO14040:2006(E) suggests that when "setting the system boundary, several life cycle stages, unit processes and flows should be taken into consideration, for example, the following:

- acquisition of raw materials
- inputs and outputs in the main manufacturing/processing sequence
- distribution/transportation
- production and use of fuels, electricity and heat
- use and maintenance of products
- disposal of process wastes and products

- recovery of used products (including reuse, recycling and energy recovery)
- manufacture of ancillary materials
- manufacture, maintenance and decommissioning of capital equipment
- additional operations, such as lighting and heating

LCA studies can be targeted at many levels. <u>Ricardo</u> propose six principal divisions:

- 1. Product LCA: streamlined to reveal "hot spots".
- 2. Product Environmental Footprint: European Commission PEF.
- 3. Intervention LCA: ensure change without unexpected impacts.
- 4. Social LCA: social and financial impacts.
- 5. Total business LCA: identify new directions in business development.
- 6. LCA Critical Review: third-party critical review process prior to publication

#### Allocation

Ekvall and Finnveden [21] undertook a critical review of the adequacy and feasibility of methods recommended for allocation by the (then) current international standard on life cycle inventory analysis with a focus on multi-functional systems. They demonstrated that different approaches to the allocation problems result in different types of information. They recommended, "that all of the environmental burdens of the multifunction process be allocated to the product investigated". LCA results appear to be largely dependent on the chosen allocation methods used. ISO14040/ISO14044 [2, 3] also recommend avoiding allocation whenever possible either through subdivision of certain processes or by expanding the system limits to include associated additional functions.

Summerscales and Dissanayake [22] considered allocation in the LCA of flax fibres for the reinforcement of composites. Their study compared allocation of environmental burdens to two different primary products: (i) flax seed as a nutritional supplement with fibre generated from the waste stream, or (ii) flax fibre as the primary product. Harvesting flax at mid-point flowering makes separation of the fibres easier, but does not yield seed. Considering flax seed as the primary product results in improved environmental credentials for the flax sliver production assuming the fibre extraction and preparation methods are similar.

#### Life Cycle Inventory analysis (LCI) phase

Inventory data is available across the scientific literature and has been compiled into various databases, e.g ecoinvent [23] which claims to be "the world's most consistent and transparent life cycle inventory database".

Dissanayake [23a] has identified that data "quality and traceability within LCA is a significant issue that needs to be addressed in developing LCA as a decision support tool and its wider adoption within the composites industry" and that it "will be extremely difficult for a practitioner to find highly relevant data among secondary sources". Primary sources are defined as "plant-specific, measured, modelled or estimated data that are directly accessed by the LCA practitioner or for which the practitioner has input into the data collection process".

#### **Proxy**

Canals et al [24] how LCA practitioners might address data gaps for bio-based products. They suggested "either proxy data sets (e.g., use existing environmental data for apples to represent pears) or extrapolated data (e.g., derive new data for pears by modifying data for apples considering pear-specific production characteristics)". They identified that use of proxy data sets was the easier and quicker solution for bridging the data gaps but was associated with highest uncertainty. The use of data extrapolation methods requires extensive expert knowledge, and hence is a more difficult technique, but can produce more robust results.

Meron et al [25] considered the use of generic or country-specific site datasets given that comprehensive site-specific LCI datasets require considerable time and effort. They proposed a methodology for systematic selection of the most appropriate proxy dataset for a specific site, from the available LCI datasets for a specific background process. They used the term site-specific as a broad term, denoting a site, a region, or a technology. Their methodology is generally applicable to various background processes. The selection of a dataset that represents the missing dataset leads in most cases to a much better approximation of environmental impacts.

A search for "resin" in the ecoinvent database version 3.8 (2021) on 23 March 2022 returned 72 entries. The options likely to be used as the matrix for fibre-reinforced composites (excluding phenolic resins) are listed in Table 1 with GFR – glass fibre-reinforced, GLO = global, HLU = hand lay-up, RER = Europe, UPR = unsaturated polyester resin, VER = vinyl ester resin,  $\checkmark$  is in the list without a synonym, and  $\times$  is not in the list. The respective names are not always clear whether the data is for resin as supplied, or for fully cured resin.

Masnadi et al [26] proposed a data-driven framework to capture and replace complex engineering simulation models with statistical proxy models. The reduced-order proxy models require less input data and replace core models for accurate and computationally efficient estimation of the parameters required for LCA studies (e.g. product composition or energy consumption). They can also be trained and utilized on behalf of commercial software to support open-source LCA simulators. The proposed methodology was used for energy return on investment (EROI) studies in oil and gas modelling, but has potential for general use.

Rietveld et al [27] undertook an cradle-to-[manufacturing facility exit] gate LCA of four generic unsaturated polyester resins and one vinyl ester resin which underpins the environmental data for polyester resins in the ecoinvent database. The system boundary is poorly defined. The impact methodology is simply described as "ReCiPe midpoint (H) as implemented in SimaPro ... using the Eco-invent 3 database" for the raw materials to quantify the respective impact categories. The report suggests that the "data inventory as obtained from the producers is available on request, but no point of contact is given!

The inventory data for epoxy resins is limited to the base resin without the ~25% addition of curing agent (hardener). The ecoinvent database includes diethanoldiamine (DEA) and dimethylaminopropylamine (DMAP/DMAPA, available commercially as epoxy hardeners) and methylamine and trimethylamine (patented as curing agent/accelerator). However, there is no data for some popular hardeners (e,g, dicyandiamide (DCDA/DICY) a.k.a. cyanoguanidine, and diaminodiphenylsulphone (DDS) a.k.a. sulfonyldianiline). The molecular structure of DDS has an aromatic ring and sulphur so may not be sensibly represented by the potential proxies in the database.

Where the database provides information for uncured resin, then additional energy (oven or autoclave cure) will be required after lamination to fully crosslink the molecules and achieve optimal physical and mechanical properties in the composite. The specific heat of a material is the quantity of heat needed to raise the temperature of unit mass of material by one degree Celcius. For a composite, a weighted sum of the respective specific heats should allow calculation of the energy required to convert the liquid resin to solid. However, it will normally be necessary to heat the mould tool to hold the form of the component. Further, the oven or autoclave will not achieve 100% efficiency, so a time-temperature record for the cure cycle, or energy metering, will be necessary to improve the reliability of the LCA.

**Table 1:** Unsaturated polyester, vinyl ester and epoxy resin production in the ecoinvent database

Name	GLO	RER synonym	Time period	Phases	Participating companies	Source
	synonym					
Unsaturated polyester						
dicyclopentadiene based UPR: production	UP	DCPD//UP	2013-2019	cradle-to-gate	Reichhold, Polynt, Ashland	Rietveld et al, Davies
	resin//DCPD	resin				
isophthalic acid based UPR: production	UP resin	UP resin	2013-2019	cradle-to-gate	Reichhold, Polynt, Ashland	Rietveld et al, Davies
maleic UPR: production	UP resin	UP resin	2013-2019	cradle-to-gate	Reichhold, Polynt, Ashland	Rietveld et al, Davies
orthophthalic acid based UPR: production	UP resin	UP resin	2013-2019	cradle-to-gate	Reichhold, Polynt, Ashland	Rietveld et al, Davies
GFR UPR HLU: production	GFK//GRP	GRP//GFK	2015-2020	gate-to-gate	(placeholder)	Kellenberger et al
UPR production	$\checkmark$	$\checkmark$	1995-2002	gate-to-gate	~	Althaus et al
Vinyl ester resin						
bisphenol A epoxy based VER:	BPA resin	BPA resin	2013-2019	cradle-to-gate	Reichhold, Polynt, Ashland	Rietveld et al
production					·	
Epoxy resin	·	·	·	·		
epoxy resin production, liquid	$\checkmark$	$\checkmark$	2015-2020	~	Guichon Valves	Gendorf +5

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Life Cycle Interpretation phase

#### **Case studies**

Owsianiak et al [28] have presented an exemplary report on an LCA to benchmark a prototype wood/glass fibre composite window against three alternative materials systems: wood, wood/aluminium or PVC. The study also sought to identify environmental hotspots for each window system.

#### Fibre reinforcements

Duflou et al [29] compared flax fibres to glass fibres for the reinforcement of polypropylene matrix composites. Flax FRP could be a valid substitute provided high fibre volume fractions are used and that the component lifetime is not significantly shorter than for the equivalent glass FRP. The agricultural activities produce higher impact for land use and freshwater eco-toxicity.

#### **Matrix polymers**

Chard et al [30] conducted an environmental life cycle assessment using GaBi to understand the environmental impacts of the thermosetting resin systems from the Scott Bader Company. A single urethane methacrylate/unsaturated polyester resin system was the focus of the assessment with a breakdown of the reaction steps. Extraction of raw materials was the most significant factor in the "cradle to factory gate" study with resin manufacture rated below 0.2% for the impact categories considered.

#### **Building materials**

La Rosa et al [31] used SimaPro 7.2 to study the environmental impacts in the production of the hemp fibre mat/bio-based epoxy/cork core eco-sandwich relative to a traditional E-glass mat/synthetic epoxy/polyurethane foam sandwich. The major environmental impact (>85%), for a functional unit of one thermal insulation panel, in both cases, was the epoxy resin. La Rosa et al [32] used a Life Cycle Assessment (LCA) methodology in SimaPro 7.3 software to evaluate the environmental impacts from four different proposed systems for use as external walls for buildings. The analysis included thermal conductivity, thermal resistance and thermal transmittance.

#### **Marine applications**

Cucinotta et al [33] undertook an LCA comparing hand lamination and vacuum infusion technologies for the yacht industry. The infused yacht was 9% lighter with the same mechanical properties), required less rew material, reduced fuel consumption and reduced mass to landfill at end-of-life.

#### **Conclusions**

A good LCA will use validated data, honest allocation, and morally sound interpretation to confirm the environmental credentials of the product or service. There is a need for a more comprehensive and transparent life cycle inventory database. The purveyors of materials should be obliged to add an Environmental Information Sheet to the Technical Data Sheet (TDS) and Materials Safety data Sheet (MSDS) to enable material specific LCA.

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#### **Declaration of Interest**

The author is not a member of any political party or political lobbying organisation. The author has been a member of the Society for Anglo-Chinese Understanding for over 33 years.

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## Appendix B: Life cycle analyses of natural fibre reinforced thermoplastic matrix marine composites

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#### Background

Bio-based polymers are potentially viable substitutes for oil-based synthetic polymers. The biomaterials may reduce greenhouse gas emissions and/or provide better End of Life (EoL) disposal options through biodegradability. The InterReg SeaBioComp project aims to explore the potential for bio-based polymers, or polymers from other renewable resources, to replace the synthetic (fossil fuel-based) polymers as the matrices for flax fibre reinforced composites to be used in the marine environment (e.g., boats, offshore renewable energy turbine blades or telecommunication buoys). Moreover, for natural fibres (flax) applied in the composite products, the environmental burdens could have a significant reduction due to the lower energy consumption. However, it is a very complex issue to assess the environmental soundness of a material through all the phases of its life.

Life cycle assessment (LCA) is a useful technique to evaluate the potential environmental impacts of products during their entire life cycle from raw material extraction and acquisition to EoL (recycling, incineration, or landfill) according to the inputs and outputs of the product system [1]. In the scope of SeaBioComp project 'monomer section for the natural fibre reinforced thermoplastic-matrix marine composites [2], the life cycle analyses of In Situ polymerization (ISP) of lactide during the Monomer Infusion under Flexible Tooling (MIFT) process for the manufacture of large natural fibre reinforced polymer matrix composite structures was conducted by the University of Plymouth. The aim of the present study is to compare the environmental benefits (majority of impact categories within LCA) of novel flax fibre reinforced thermoplastic-matrix (polylactic acid or acrylic) composites to those when using thermosetting matrix systems (unsaturated polyester or epoxy resin).

#### Methodology of LCA

According to ISO 14040: 2006 [3] and ISO 14044: 2006 [4], a LCA study starts with goal and scope definition, continues to life cycle inventory (LCI) analysis, life cycle impact assessment (LCIA) and ends with interpretation of results. The stages of a LCA study are shown in Figure 1.1.

In the goal and scope stage, the functional unit of the studied materials needs to be defined. The system boundaries are dependent on the subject of the study (cradle-to-gate, cradle-to-grave, cradle-to-cradle etc.) or in an ideal situation. The allocation procedure needs to be followed. In the LCI stage, the inventory of input or output data about the system needs to be collected to calculate the embodied energy consumption and pollution emissions of the studied materials during the entire life cycle. The relevant data should meet the quality requirements, the cut-off criteria (mass, energy or environmental significance) need to be employed. In the LCIA stage, specific impact assessment methods (CML, ReCiPe, CED or IMPACT, etc.) are used to assess the environmental impacts of the studied materials. In the interpretation stage, the findings of LCI or LCIA need to be summarised or discussed for conclusions and recommendations. A sensitivity study can be conducted to determine the influence of the critical parameters.

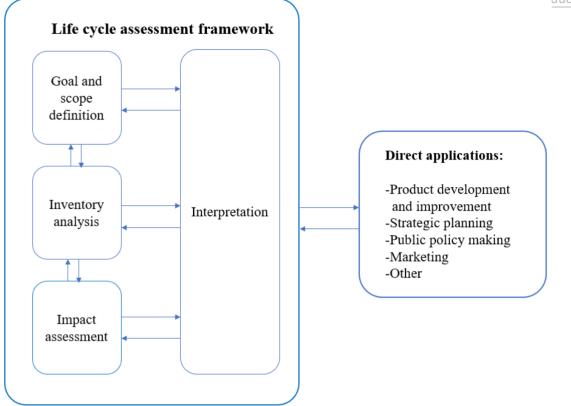


Figure 0.1: Procedure of life cycle assessment (in accordance with ISO 14040)

#### Environmental impact categories of LCA

ISO/TR 14047 (2003) [5] defines eight environmental impact categories (acidification potential, aquatic toxicity potential, human toxicity potential, eutrophication potential, global warming potential, non-renewable/abiotic resource depletion potential, ozone depletion potential and photochemical oxidants creation potential) which needs to be analyzed in a LCIA. A good LCA should not only consider the greenhouse gas emission but require the consideration of at least the environmental impact factors mentioned above [6].

The detailed descriptions of the relevant environmental impact categories follow [7],

#### Acidification potential (AP)

AP is the result of acids being emitted into the environment, subsequently released into the land and water. The classification factor of AP can be expressed as  $SO_2$  eq. (equivalent), which caused by the combination of  $SO_2$ ,  $NO_x$ , HCl,  $NH_3$  and HF to the potential acid deposition.

#### Aquatic toxicity potential (ATP)

ATP quantifies the concentration of different harmful or poisonous substance which affect aquatic organisms. Given current concerns about marine plastics pollution, there is an urgent need to additionally address displaced stomach capacity, suffocation and strangulation.

#### Human toxicity potential (HTP)

HTP results from the release of toxic chemicals, which may be harmful to human health, released into the environment (air, water and soil). The human toxicological factors can be measured by the acceptable or tolerable daily intake of the toxic element.

#### Eutrophication potential (EP)

EP is the potential for the increase of nutrients which may lead to over-fertilization of water and soil. The overuse of fertilizers (nitrate or phosphate) for plant growth purpose can be regarded as the pollution in an aquatic system which may result in the growth of biomass especially algal blooms. The classification factor of EP can be expressed as relative to  $PO_4^{3-}$  based on the weighted sum of the release of nitrogen and phosphorus derivatives ( $N_2$ ,  $NO_x$ ,  $NH_4^+$ , P,  $PO_4^{3-}$  and chemical oxygen demand (COD)).

#### Global warming potential (GWP)

GWP is the measure of emission of greenhouse gas ( $CO_2$ ,  $N_2O$ ,  $CH_4$  and volatile organic compounds (VOCs)) into the atmosphere. The classification factor of GWP can be expressed as  $CO_2$  eq.

#### Non-renewable/abiotic resource depletion potential (NRADP)

NRADP estimates the proportion of the world reserves of metals, minerals and fossil fuels used.

#### Ozone depletion potential (ODP)

ODP is the measure of the depletion of the ozone layer caused by emissions of chlorofluorocarbon compounds (CFCs) and chlorinated hydrocarbons (HCs). The classification factor of ODP can be expressed as CFC<sup>-11</sup> eq. This category lead to the banning of chemicals under the Montréal Protocol.

#### Photochemical oxidants creation potential (POCP)

POCP is the measure of the depletion of volatile organic compounds (VOCs) including alkanes, halogenated HCs, acetylenes, aromatics, olefins, alcohols, aldehydes, ketones, esters, ethers etc. in the sunlight and the oxides of nitrogen ( $NO_x$ ). The POCP is quantitatively classified relative to 1 kg of ethylene.

#### Monomer and manufacturing techniques selection

In the context of previous work conducted by the University of Plymouth [2], the life cycle inventory of the natural fibre (flax), selected monomer/resin (PLA or acrylic) as well as the relevant manufacturing techniques (MIFT or compression moulding) has been reviewed in this section.

#### Flax production

Natural-fiber reinforced composites are regarded as good alternatives for glass-fiber reinforced composites due to the multiple benefits. Among different natural fibers (flax, hemp, jute and cotton etc.), flax is one of the most widely used bio-fibers which represented almost 50% of the natural-fibre composite market [8]. Furthermore, flax has been long utilised as the source of textile fibre [9]. Since 1994, Canada became the largest flax producer around the world, during 2005 and 2006, Canada produced over one million tonnes of flax in which 60% was exported to EU countries, 30% was exported to USA and 4% was exported to Japan. Other countries like France, Belgium and Netherland also leads in production of flax, EU countries produced almost 12200 ton of flax fibre in 2007 [8]. The growing cycle of flax in the western European region is about 100 days from sowing in March to harvesting in July.

Dissanayake et al. [10] investigated the energy requirement for the production of flax fibre to be used as reinforcement in composites. It was found that sliver (post-carding fibre) required an embodied energy of 59 GJ/ton through no till and warm-water retting. For yarn, the spinning process could significantly increase the embodied energy to 86 GJ/ton. Later, González-García et al. [11] identified the environmental impacts of the production of flax and hemp for non-wood pulp mills by using LCA methodology. The LCI data was obtained from Spanish plantations and data for the background system was selected from Ecoinvent database, the LCIA phase was carried out following the CML baseline 2000 methodology. Le Duigou et al. [12] quantified the environmental impact of the production of hackled flax fibers to be used as composite material reinforcement. Their results showed that flax fibre has better performance according to most environmental indicators (climate change, acidification, non-renewable energy consumption etc.) compared to that of glass fibre.

More recently, Gomez-Campos et al. [9] conducted a detailed cradle-to-gate LCA to evaluate the environmental performance of a flax-based technical textile. The LCI data considered the flax cultivation, retting, scutching, combing, spinning and weaving process, the fate of co-products was documented as well. In their calculation model, the cultivation and the initiate stages (retting, scutching and combing) took place in France, last stages of the process chain (spinning and weaving) took place in China. The comparisons of potential environmental impacts associated to different types of natural fibers (hackled flax, flax textile and hemp) can be can be found in

Table 2.13. It should be noted that these results may not be obtained through the same assumptions and model setup, therefore are only for indicative use

Table 0.1 Potential environmental impacts associated to different types of natural fibers

		Natural fibers			
Impact Category	Units	Hacked flax (ton)	Scutched flax (ton)	Hemp (ton)	
Abiotic Depletion (ADP)	kg Sb eq.	1.7 [12]	1.3 [12]	4 [18]	
Acidification Potential (AP)	kg SO₂ eq.	3.22 <sup>[11]</sup> 2.2 <sup>[12]</sup>	1.8 [12]	9.39 <sup>[11]</sup> 2.6 <sup>[18]</sup>	
Eutrophication Potential (EP)	kg PO <sub>4</sub> -3 eq.	2.28 <sup>[11]</sup> 1.4 <sup>[12]</sup>	1.4 [12]	14.6 <sup>[11]</sup> 0.6 <sup>[18]</sup>	
Global Warming Potential (GWP)	kg CO₂ eq.	437 <sup>[11]</sup> -1400 <sup>[12]</sup>	-1450 <sup>[12]</sup>	1600 <sup>[11]</sup> 531 <sup>[18]</sup>	
Ozone layer Depletion Potential (ODP)	kg CFC <sup>-11</sup> eq.	2.4×10 <sup>-5 [12]</sup>	2.1×10 <sup>-5 [12]</sup>	6.88×10 <sup>-5 [18]</sup>	
Human Toxicity Potential (HTP)	kg 1.4DB eq.	215 [12]	150 <sup>[12]</sup>	136 [18]	
Freshwater Aquatic Ecotoxicity Potential (FAEP)	kg 1.4DB eq.	59 <sup>[12]</sup>	54 <sup>[12]</sup>	57.1 <sup>[18]</sup>	
Photochemical Oxidation Potential (POP)	kg C₂H₄ eq.	0.114 <sup>[11]</sup> 0.073 <sup>[12]</sup>	0.058 [12]	0.213 [11]	
Land Use	m² year	850 <sup>[12]</sup>	970 <sup>[12]</sup>	1540 <sup>[18]</sup>	
Terrestrial Ecotoxicity Potential (TEP)	kg 1.4DB eq.	8.7 [12]	2.6 [12]	1.52 [18]	
Cumulative Energy Demand (CED)	MJ eq.	59190 <sup>[10]</sup> 11700 <sup>[12]</sup>	4400 [12]	8890 [18]	

#### ThermoSet Matrices

Thermosetting resins are often applied for fibre-reinforced polymer (FRP) composites due to their good mechanical properties, durability and thermal stability [13]. The most common commercial thermosetting resins are phenolic resins, epoxy, unsaturated polyester and vinyl ester. They are normally produced as liquid resin and are solidified by chemical agent with heat sometimes needed to complete the crosslinking [14]. However, the thermosetting resins cannot be melted or reformed after the reaction, so recycling can be a big issue for thermosetting resins at the EoL stage.

#### Unsaturated polyester (UP)

UP resins are generally manufactured by the condensation reaction of unsaturated dibasic acid and di-functional alcohols. The UP resin is cured by an addition chain reaction initiated by a peroxide and a reducing agent [14]. Rietveld and Hegger [15] performed cradle-to-gate LCA analyses to develop and build LCI data of four generic UP resins, the environmental impact indicators of each resin were also studied.

#### Ероху

Epoxy resins are normally manufactured by the condensation reaction of a phenolic and epichlorohydrin. The epoxy system is cured by ring-opening of the epoxide (cross-linking reaction) initiated by hardeners [14]. They have been widely used in the engineering applications (e.g., civil engineering and automotive industry) due to its good material properties [16]. Koči and Loubal [17] performed a cradle-to-gate LCA to compare the environmental impacts of the production of liquid epoxy resins with epichlorohydrin based on propylene or glycerin. The bio-based epoxy resin has a better environmental performance. Recently, La Rosa et al. [18] [19] used cradle-to-gate LCA approaches to investigate the environmental impacts and energy use of epoxy-based composite materials. Quintana et al. [20] conducted a comparative cradle-to-grave LCA of gypsum plasterboard and bio-based epoxy composites reinforced with different natural fibers. It should be mentioned that, due to the poor recyclability, the disposal solutions of epoxy composites in the industry are landfills or incineration [21].

#### Thermoplastic matrices

Thermoplastic resins have been frequently used with natural fibers to create natural fibre-reinforced polymer (NFRP), because they can be easily applied into complex part of the fibre and provide good impact resistance [13]. Primary thermoplastic resins for the composites are polypropylene, polyamide, polyester and PEEK [14]. It should be mentioned that the thermoplastic resins require high processing temperature, the melting point value of crystalline polymers is about 200 °C (±50 °C) higher than the glass transition temperature limiting use in highly-stressed components (heating mould tools for large structures may require very high energy inputs). Unlike thermosetting resins, thermoplastics can readily be recycled at the EoL stage.

#### Polylactic (acid) (PLA)

PLA can be manufactured by direct condensation polymerization of lactic acid [2] which is produced from the fermentation of 100% natural resources (from corn or sugarcane). It has been widely used in the packaging industry and various biomedical applications due to the biocompatibility. The full life cycle of PLA can be divided into five phases, including feedstock, conversion, manufacturing, application and EoL options where the manufacturing processes include blow moulding, blending compounding, electrospinning, casting, thermoforming, foaming, injection moulding, extrusion and additional manufacturing [22]. The PLA manufacturing company Total Corbion PLA [23] and NatureWorks<sup>TM</sup> [24-26] have provided the detailed LCI data for the production of PLA, the LCA was performed for measuring environmental sustainability.

De Andrade [27] compared the environment burdens of three PLA disposal approaches (mechanical recycling, chemical recycling and composting), it was shown that the mechanical recycling gave the lowest environmental impact. More recently, Maga et al. [28] presented a LCA study for different recycling technologies (mechanical recycling, solvent based recycling and chemical recycling) for industrial PLA wastes. The results showed that the recycling of PLA waste can reduce the relevant environmental impacts.

#### Acrylic (acrylates)

Acrylic (e.g., methyl methacrylate (MMA)) monomer) is converted by vinyl (addition) polymerization to polymethy methacrylate (PMMA) monomer [2]. It is a common plastic material which has strong, stiff and transparent properties. Having similar characteristics to thermosetting resins, acrylic was proved as the first thermoplastic resin which can be used for the production of composite materials by using resin transfer moulding.

Table 0.2: Potential environmental impact of 1 kg of unsaturated polyester, 1 kg of liquid petroleum-based epoxy, 1 kg of liquid bio-based epoxy, 1 kg of polylactic acid and 1 kg of polymethyl methacrylate

SeaBioComp-Version 1	Matrices	D. 3.5.2 LCA for	the uniterent	biocomposit	es production
Impact Category Units	Unsaturated polyester (kg)	Liquid petroleum- based epoxy (kg)	Liquid bio- based epoxy (kg)	Polylactic acid (kg)	Polymethyl methacrylate or acrylic (kg)
Abiotic Depletion (ADP) (kg Sb eq.)	-	2.1×10 <sup>-5 [17]</sup> 0.0594 <sup>[18]</sup>	0.9×10 <sup>-5 [17]</sup> 1×10 <sup>-5 [18]</sup>	-	2.54×10 <sup>-15 [29]</sup>
Acidification Potential (AP) (kg SO <sub>2</sub> eq.)	-	0.0384 <sup>[17]</sup> 0.0403 <sup>[18]</sup>	0.0276 <sup>[17]</sup> 0.0254 <sup>[18]</sup>	7.26×10 <sup>-3</sup>	-
Eutrophication Potential (EP) (kg PO <sub>4</sub> -3 eq.)	-	5.5×10 <sup>-3 [17]</sup> 6.6×10 <sup>-3 [18]</sup>	7.6×10 <sup>-3 [17]</sup> 6.9×10 <sup>-3 [18]</sup>	1.38×10 <sup>-3</sup>	-
Global Warming Potential (GWP) (kg CO <sub>2</sub> eq.)	-	8.654 <sup>[17]</sup> 6.663 <sup>[18]</sup>	4.632 <sup>[17]</sup> 4.079 <sup>[18]</sup>	0.5 <sup>[23]</sup> 1.3 <sup>[25]</sup> 0.62 <sup>[26]</sup>	8.43 <sup>[29]</sup>
CO <sub>2</sub> emissions (kg)	-	5.9 <sup>[1]</sup>	-	0.827 [25]	-
CO emissions (g)	-	2.2 [1]	-	4.167 [25]	-
SO <sub>x</sub> emissions (g)	-	19 <sup>[1]</sup>	-	7.401 [25]	-
NO <sub>x</sub> emissions (g)	-	35 <sup>[1]</sup>	-	12.311 <sup>[25]</sup>	-
Ozone layer Depletion Potential (ODP) (kg CFC <sup>-11</sup> eq.)	-	5×10 <sup>-7 [17]</sup> 1.26×10 <sup>-9 [18]</sup>	2×10 <sup>-7 [17]</sup> 0 <sup>[18]</sup>	3.99×10 <sup>-</sup> 13 [26]	2.63×10 <sup>-9</sup> [29]
Human Toxicity Potential (HTP) (kg 1.4DB eq.)	-	0.276 <sup>[17]</sup> 0.49 <sup>[18]</sup>	0.566 <sup>[17]</sup> 0.545 <sup>[18]</sup>	-	-
Freshwater Aquatic Ecotoxicity Potential (FAEP) (kg 1.4DB eq.)	-	0.0166 <sup>[17]</sup> 0.2465 <sup>[18]</sup>	0.0682 <sup>[17]</sup> 0.0664 <sup>[18]</sup>	-	-
Terrestrial Ecotoxicity Potential (TEP) (kg 1.4DB eq.)	-	0.0109 <sup>[17]</sup> 0.0291 <sup>[18]</sup>	0.232 <sup>[17]</sup> 0.228 <sup>[18]</sup>	0.0348 [23]	-
Marine Aquatic Ecotoxicity Potential (MAEP) (kg 1.4DB eq.)	-	-	-	0.0139 [23]	-
Particulate matter (kg PM2.5 eq.)	-	0.015 [1]	-	0.0017 [23]	0.00407 [29]
BOD to water (mg)	-	1200 [1]	-	2.746 [25]	-
COD to water (mg)	-	5.1×10 <sup>4</sup> [1]	-	4895.4 <sup>[25]</sup>	-
Nitrates to water (mg)	-	1 [1]	-	-	-
Phosphates to water (mg)	-	220 [1]	-	-	-
Non-Renewable Energy (NRE) MJ primary)	-	146.313 [17]	102.788 [17]	28.8 <sup>[23]</sup> 42 <sup>[25]</sup> 40.05 <sup>[26]</sup>	-
Photochemical ozone formation (kg NMVOC eq.)	-	2.958×10 <sup>-3</sup> [17]	2.083×10 <sup>-3</sup>	6×10 <sup>-4</sup> [26]	0.0329 [29]
Cumulative Energy Demand (CED) MJ eq.	76.9~92.5 <sup>[15]</sup>	140.71 <sup>[1]</sup> 2.16×10 <sup>-3 [18]</sup>	119.3 <sup>[15]</sup> 1.9×10 <sup>-3 [18]</sup>	89.2 <sup>[23]</sup> 67.8 <sup>[25]</sup> 66.66 <sup>[18]</sup>	-

investigate the eco-profiles of polythylene terephthalate (PET) and PMMA, the inclusive LCI model was built for the assessment of environmental impacts. Table 2.2 compares the potential environmental impacts of 1 kg of UP, liquid petroleum-based epoxy, liquid bio-based epoxy, PLA and acrylic. As expected, the thermoplastic resins (PLA or acrylic) have a better environmental performance than thermosetting resins (UP or epoxy). It should be noted that these results may not be obtained through the same assumptions and model setup, therefore are only for indicative use.

#### Manufacturing techniques

Up to now, some manufacturing techniques such as injection molding, hand lay-up, compression moulding (CM), resin transfer moulding (RTM) and pultrusion have been developed for producing composites. These techniques might be applicable to different types of composites, e.g.:

Parvez Mahmud and Farjana [29]

performed a LCA study to

- injection moulding [30] can be regarded as a suitable process for manufacturing short fibre reinforced thermoplastic matrices;
- hand lay-up [31-33] is a convenient technique for making low performance composites;
- compression moulding [30] [34] [35] [36] can be used to produce high-quality thermoplastic composites;
- RTM [37] [38] is one of the most popular composite manufacturing processes for small and medium sized natural fibre reinforced polymer composites with complex shapes;
- pultrusion [39] is more suitable for thermoplastic or partially cured thermoset composites.

In the InterReg SeaBioComp project, the University of Plymouth adopted in situ polymerisation (ISP) of lactide and/or methyl methacrylate during the Monomer Infusion under Flexible Tooling (MIFT) to manufacture large scale natural-fibre reinforced polymer matrix marine composites. Meanwhile, IMT Lille-Douai applied compression moulding (CM) to produce flax reinforced PLA composites. In this report, life cycle analyses of flax fibre reinforced thermoplastic-matrix (PLA or acrylic) through the relevant manufacturing techniques (MIFT or CM) were carried out by using the commercial LCA software SimaPro (Version 9.4).

In situ polymerisation during monomer infusion under flexible tooling

RTM is one of the most popular liquid composite moulding technologies for material engineering. In RTM, the dry fibers are placed in two solid mold tools, then the liquid resin will flow into the fabric by means of pressure or vacuum pump [40] (see Figure 2.1). However, RTM is not suitable for the large area structures due to the high tooling cost. To overcome this problem, vacuum bag replaces one of the matched pairs of solid molds. The process is known as resin infusion under flexible tooling (RIFT) [41]. Summerscales and Searle [42] [43] classified four-stage variants for the RIFT processes:

- RIFT I: in-plane flow parallel to the layers of reinforcement.
- RIFT II: through-plane flow from a flow medium or second core.
- RIFT III: resin film infusion.
- RIFT IV: partially pre-impregnated materials.

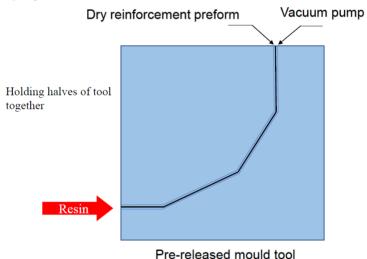


Figure 0.2: Schematic of resin transfer moulding (RTM)

RIFT II was used to produce flax reinforced thermoplastic matrix composites by the University of Plymouth. As shown in Figure 2.2, the liquid resin (epoxy or acrylic) is pulled through the flax fiber technical textile by means of a vacuum pump. The composite is normally cured at room temperature for 24 hours and then post-cured into the oven or with heater blankets at 80°C for 3 hours.

production routes

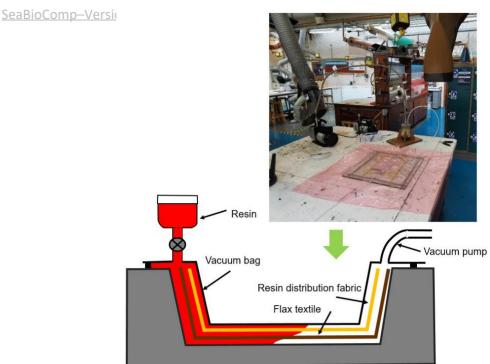


Figure 0.3: Schematic of standard resin infusion under flexible tooling (RIFT II with a flow medium)

The production of PLA/flax composite requires an elevated temperature, the relevant manufacturing process can be found in Figure 2.3. PLA monomers are first melted over 120°C, after adding the catalyst, the liquid PLA resin infiltrates the flax textile by means of vacuum pump. For curing the PLA/flax reinforcement, the whole experimental equipment will be placed in the oven at 80°C for 3 hours.

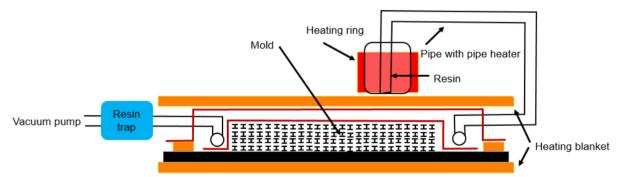


Figure 0.4: Manufacturing process of Flax/PLA composites through RIFT at elevated temperature

#### Compression moulding

Compression moulding (also known as 'stamping') is a standard composite manufacturing approach for laboratory-level research of natural-fibre composites using heat and pressure. The cycle time of compression moulding is relatively short. Figure 2.4 demonstrates a simplified process of compression moulding. The material can be preheated and placed into a heated mould cavity directly. After closing with a top cover, the pressure is applied on the top cover so that the molten material can reach all mould area. Meanwhile, heat and pressure would be maintained until the materials have been cured. It should be mentioned that the pre-impregnation or film stacking stage have a significant influence on the quality of composites due to the limitation of the matrix flowing in the mould tool [44].

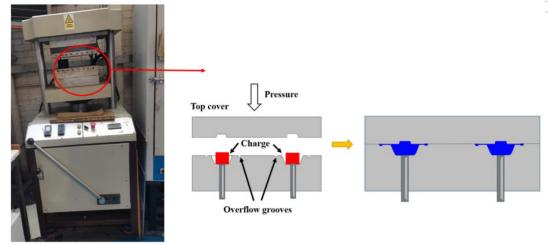


Figure 0.5: Simplified process of compression moulding

To maintain the strength of flax fibres, the processing temperature of compression moulding should be below the degradation temperature (not exceed 190°C). For the flax/PLA composites produced by compression moulding in IMT Lille-Douai, the specimen was heated at 180°C for 8 minutes and then cooled at 20°C for 5 minutes, the pressure was set between 3.5 MPa and 14 MPa depending on the weight of the material [45]. The energy intensities of the relevant composite manufacturing techniques reported on [45-49] for the onsite energy use and primary energy use can be found in Table 2.3.

Manufacturing methods	Primary energy (MJ/kg)	Onsite energy (MJ/kg)
Resin infusion process (RIFT I)	-	9.9 <sup>[46]</sup>
Vacuum assisted resin infusion (RIFT II)	30.6 [47]	10.2 [48]
Resin transfer moulding	38.4 [47]	12.8 [48]
Pultrusion	9.3 [47]	3.1 [48]
Sheet molding compound	-	3.5 <sup>[48]</sup>
Compression moulding	37 4 <sup>[45]</sup> or 34 3 <sup>[47]</sup>	11 4 <sup>[49]</sup>

Table 0.3: Energy intensities of manufacturing process

# Life cycle assessment (LCA)

The composite life cycle begins with raw material extraction, processing, transport manufacturing, use and disposal. LCA provides the quantitative environmental impact assessment by evaluating the major inputs and outputs of the selected materials and energy. Based on the framework of LCA, the life cycle analyses of fibre-reinforced composites (flax-epoxy composites, flax-UP composites, flax-PLA composites, and flax-acrylic composites) are described in the following sections.

# Goal and Scope

The following work is a cradle-to-grave study to evaluate the main environmental impacts related to the production and disposal of different material configurations for fibre reinforced composites intended for marine application.

The LCA based on ISO 14040: 2006 [3] and ISO 14044: 2006 [4] methodology was conducted by using commercial software SimaPro 9.4.

# Configurations

#### Table 3.1 details the configurations used for the LCA.

Table 0.4: LCA material configurations (FVF = fibre volume fraction)

LCA Case	Material	Manufacture Method	Part Description
1	Flax-Epoxy, FVF 0.5	RIFT	Medium Plate
2	Flax-UP, FVF 0.5	RIFT	Medium Plate
3	Glass Epoxy, FVF 0.5	RIFT	Medium Plate
4	Glass UP, FVF 0.5	RIFT	Medium Plate
5	Flax PLA, FVF 0.4	RIFT	250*150*3 mm plaque
6	Flax-PLA, FVF 0.6	CM	250*150*3 mm plaque
7	Flax-PMMA, FVF 0.5	RIFT	Medium Plate

#### **Functional Unit**

The functional unit considered is 1 kg of fibre reinforced composites used in the marine environment for 20 years. This work does not consider difference in durability or mechanical performance.

# **System Boundaries**

The system boundaries of the composite parts are detailed below:

- a. Production of matrix and reinforcements, including raw material extraction, and subsequent processing.
- b. Transportation of materials and tooling at all stages of the life cycle.
- c. Manufacture of composite including required material preparation, laminating process through either RIFT or compression moulding, subsequent heat treatment, and Finishing processes.
- d. Waste of material in any previous steps.
- e. End of life disposal of the composite, Including processing.

What will not be considered within the system boundaries:

- The overhead energy use for the composite manufacture, this is considered the same for all composites.
- The embodied impacts of tooling/equipment used in the composite manufacture

#### Other Assumptions

• The use phase will be the same for all composites, and so will not be investigated here.

# System Model

The chosen system model is allocation cut-off, due to wide use in literature, and ease of modelling. This system is a method where impacts and emissions from wastes and co products are the responsibility of the producer, and so using any of these wastes or co products will be burden free.

# Impact Methodology

The chosen methodology will be Recipe Hierarchist Midpoint, covering a range of impact categories to give a holistic view on the environmental impacts of the production. This model was chosen because of its good representation of data and widespread use within literature and published LCA, ensuring good comparability. For network diagrams using single point scores, the Recipe Hierarchist Endpoint methodology was used, assigning weight factors to midpoint results, to form a single impact value. Processes are disregarded if below 0.1% relative contribution.

## Life Cycle Inventory

Based on the framework of international LCA standard ISO 14044: 2006 [4], an inventory analysis was conducted to quantify the input and output flows of the systems in terms of materials and emissions through a mass and energy balance. The full inventory table can be found in the attached file Appendix\_C\_Life\_Cycle\_inventory.

# Flax Fibres

The flax fibres will be modelled according to Gomez-Campos's work [9], with alterations made to fit in with the chosen system model. It is assumed that the flax is grown and spun within France, and shipped to Devon. The following outputs described in this process are considered as waste, and a description of their subsequent processing and emissions/inputs are shown in Table 3.2:

Waste	Attributed to	Fate	Modelling
Retting Dust	Retted Flax	Left on Field	Waste wood, untreated
			{IN}  market for waste
			wood, untreated   APOS, U
Retting Shives			Waste wood, untreated
			{IN}  market for waste
			wood, untreated   APOS, U
Scutching Shives	Short Fibres	Mulch, animal bedding,	Waste wood, untreated
	Grains	building filler	{IN}  market for waste
	Long Fibres		wood, untreated   APOS, U
Scutching Inert Residue		Soil Amendment	Waste wood, untreated
			{IN}  market for waste
			wood, untreated   APOS, U
Combing Dust	Flax Tow	Soil Amendment	Waste wood, untreated
	Flax Sliver		{IN}  market for waste
			wood, untreated   APOS, U
Spinning Wastewater	Flax Yarn	Waste water	Wastewater from textile
			production {GLO}  market
			for wastewater from textile
			production   APOS, S

It is assumed that flax fibre density is 1300 kg/m<sup>3</sup> [52].

# **Epoxy Resin**

The LCA data of epoxy 'Epoxy resin, liquid {RER}| market for epoxy resin, liquid | Cut-off, S' and epoxy curing agent 'Diethylene glycol {GLO}| market for | Cut-off, S' were used from ecoinvent. It should be mentioned that the source document for epoxy resin cited by the ecoinvent database is Ernst and Young's report [15] which is not currently in the public domain.

The assumed is density will be 1100 kg/m<sup>3</sup>.

# **Unsaturated Polyester**

The LCA data of unsaturated polyester (UP) resin 'Maleic unsaturated polyester resin {GLO}| market for | Cut-off, S' was used from Ecoinvent database. Similarly, to the epoxy resin, the data for UP resin cited by the Ecoinvent is a publication that appears not to be in the public domain.

The assumed density is 1200 kg/m<sup>3</sup>.

#### PLA

The inventory data for the production of lactide monomer was provided by the Regulatory and Sustainability manager of Corbion (Mutual NDA has been signed for sharing LCA information).

The assumed density is  $1240 \text{ kg/m}^3$ .

# **PMMA**

The LCA data of methyl methacrylate (MMA)) monomer 'Methyl methacrylate {RER}| market for methyl methacrylate | Cut-off, S' was used from Ecoinvent database.

The assumed density is 1010 kg/m<sup>3</sup>.

#### **RIFT Process**

The energy consumption measured in the lab for this process is approximately 0.12 kWh, however due to questionably low value literature sources will be used. According to the data sources in [47], the embodied energy for vacuum assisted resin infusion is about 10.2 MJ/kg.

## **Compression Moulding**

In the IMT Lille-Douai, the flax-PLA composites were manufactured using compression moulding. The embodied energy for compression moulding was 11.4 MJ/kg referring to the data in [47].

# Transportation

Where market sources from ecoinvent were used for materials, the transportation is included.

The flax textile produced in Tiverton was transported 230 km back to Portsmouth ferry port by truck, and then delivered 200 km back to Le Havre port by ship. Finally, the flax textile was delivered 310 km from Le Havre port to IMT Lille-Douai by truck.

The lactide monomer was purchased from Corbion (Lyon, France) and transported 680 km to IMT Lille-Douai by truck for the test

The considered EoL scenario for all composites was landfilling, the data of landfilling was used based on Ecoinvent database for landfilling process.

# **Results and Discussion**

Full Results can be found in the attached file Appendix\_D\_LCA results.

# Flax Epoxy (Figure 4.1 and Figure 4.2)

The flax fibre had the majority contribution in 16 out of the 18 Recipe Midpoint impact categories, including GWP, human toxicities, and eutrophication. For fossil resource scarcity, as expected the epoxy is the main contributor, due to requiring organic feedstock obtained from crude oil. The fine particulate matter was also higher for the epoxy, though the reason for this is unknown.

The greater relative impact of the flax over the epoxy could be due to the higher density of the flax fibre. With a FVF of 0.5 the weight of the flax makes up 54.2%, whilst the epoxy makes up 45.8%, as there is more material by mass, it follows that impacts are higher.

The majority of the GWP contribution came from the flax fabric, this is mostly due to the embodied energy requirements in producing the fertilisers, and tillage work. The tillage is mostly completed using diesel tractors, which have low efficiency and high fuel consumptions, resulting in large CO2 emissions. Due to high water requirements for growth and preparation of the fibres, flax fabric production utilised roughly 65% of the water consumption during production.

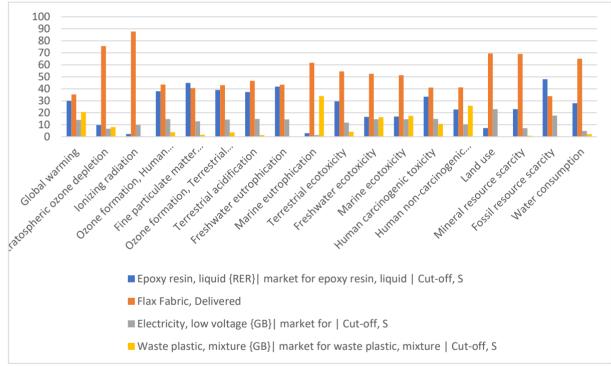


Figure 0.6: Relative contribution of Flax Epoxy composite

Figure 0.7: Network diagram of Flax Epoxy composite single point score

# Flax UP (Figure 4.3 and Figure 4.4))

The flax dominated the environmental impacts, being the major contributor in 17 out of the 18 categories. Polyester resin was the major contributor to fossil resource scarcity due to requiring fossil fuel feedstock. Again, the flax has a higher density than the polyester, of 1300 kg/m³, with UP being 1200 kg/m³. This means greater weight of flax than the UP in the composite and can explain some of the increased environmental burdens.

Both the freshwater and marine eutrophication impacts are dominated by the flax in this composite, this is expected and is due to the high use of fertilisers in the crop cultivation. Land use is significantly higher for the flax due to the large land requirements for both growth and retting of the fibres.

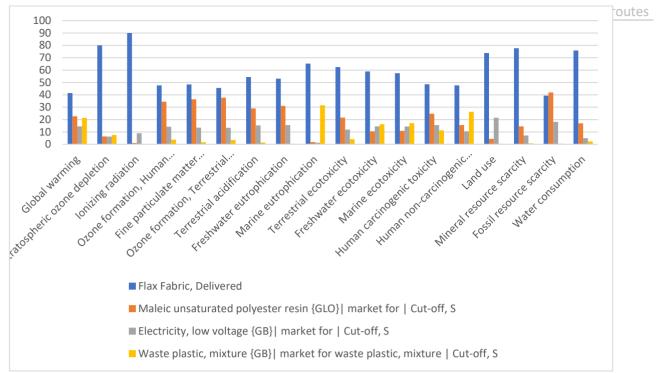


Figure 0.8: Relative Contribution of Flax UP Composite

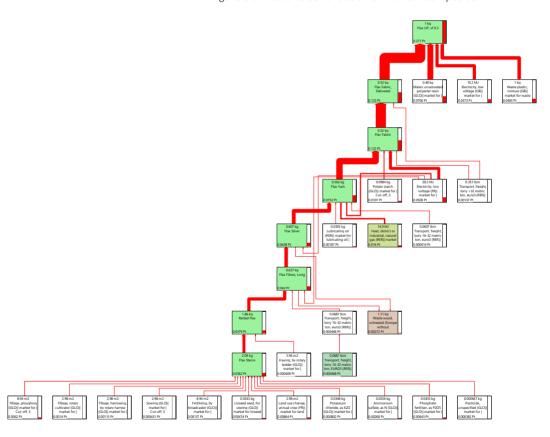


Figure 0.9: Network diagram of Flax UP composite single point score

# Flax PLA, FVF 0.4 (Figure 4.5 and Figure 4.6)

The results shows that flax contributes most of the environmental harm in 16 out of the 18 impact categories. The PLA shows high contribution to impact categories most associated with agriculture, almost matching flax in terrestrial acidification, whilst surpassing flax in marine eutrophication and land use. This is due to PLA requiring feedstock from the agricultural industry, and so embodies high land and fertiliser use, which is compounded due to inefficiencies of fermentation for lactic acid.

The electricity use during the composite manufacture is relatively higher for the PLA materials due to the high processing temperature, up to 150 degrees.

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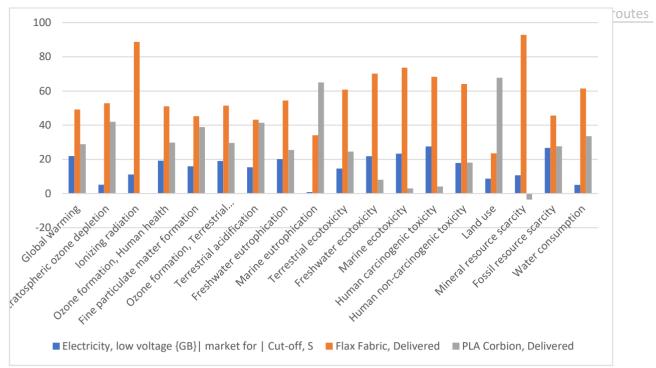


Figure 0.10: Relative Contribution of Flax PLA, FVF 0.4 Composite

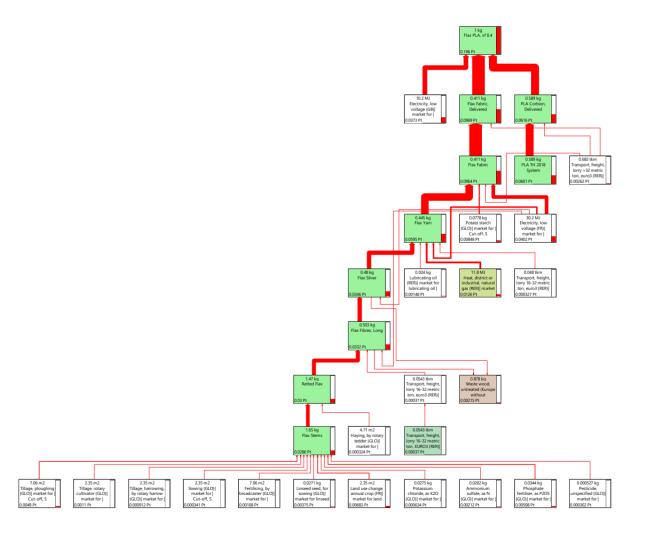


Figure 0.11: Network diagram of Flax PLA, vf 0.4 composite single point score

# Flax PLA, FVF 0.6 (Figure 4.7 and Figure 4.8)

With the increase in the FVF, the relative contribution of the flax to the environmental impact increases, resulting in being the main contributor in 17 impact categories. As with the Flax PLA composite with a lower volume fraction, land use is higher for the PLA, though less prominent due to changes in matrix/fibre proportions.

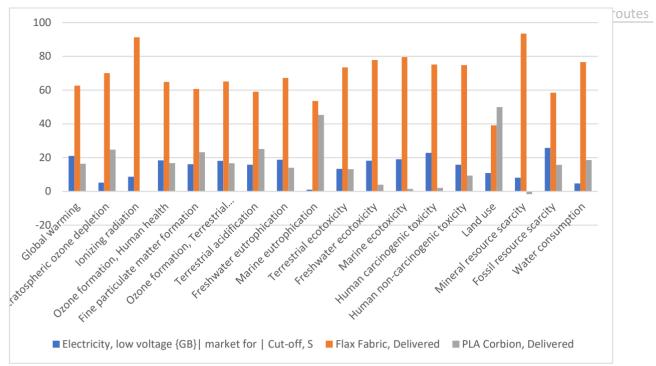


Figure 0.12: Relative Contribution of Flax PLA, FVF 0.6 Composite

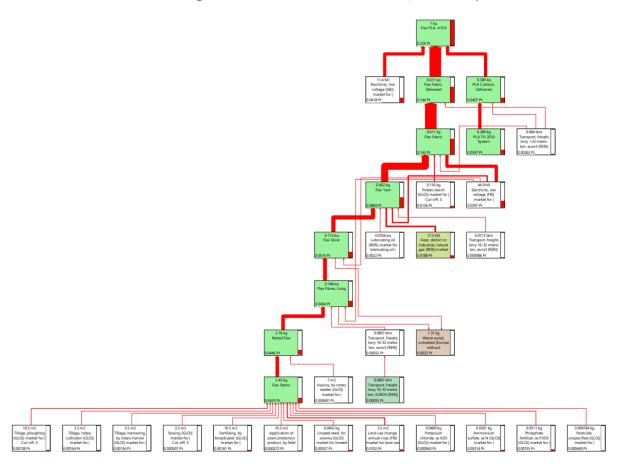


Figure 0.13: Network diagram of Flax PLA, vf 0.6 composite single point score

# Flax PMMA (Figure 4.9 and Figure 4.10)

As with the other configurations, flax is the leading cause of the environmental impact, with its relative contribution being the major part in 15 of 18 of impact categories.

Where the other fossil derived resins were significantly higher in the fossil resource scarcity than the flax, the MMA is relatively low. This could be due to feedstock being able to be sourced from both oil and natural gas [53]. The MMA is contributing highly in ozone formation, fine particulate matter and ozone formation, approaching that of the flax fibre. The high terrestrial acidification of the MMA is a surprising result, as this impact is typically

associated with agriculture and so expected to be dominated by the flax. This could be down to emissions during the production of ammonia, a major precursor.

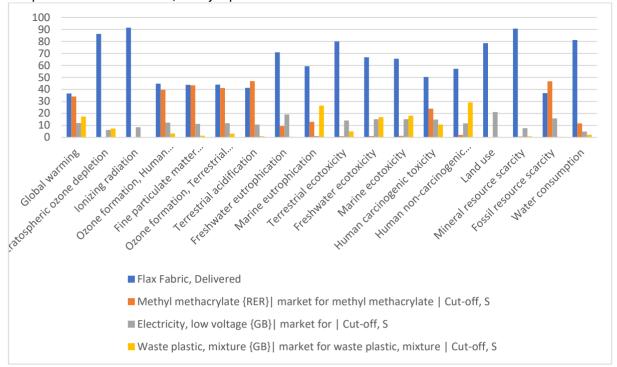


Figure 0.14: Relative Contribution of Flax PMMA Composite

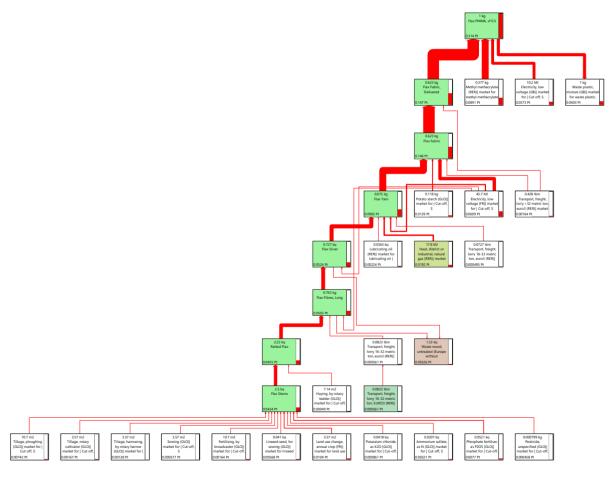


Figure 4.10:

Network diagram of Flax PMMA composite single point score

# Comparison

Figure 4.10 details the relative impact of the different materials to the highest material for the given impact category. Large variation of the GWP is present with the Flax/PMMA composite showing the greatest CO2 eq Copyright © SeaBioComp

emissions followed by epoxy, glass, and PLA having the least. This is likely due to the carbon sequestration within the PLA manufacturing.

As expected, the PLA containing materials were the poorest performing in marine eutrophication and land use, both impacts associated with biomaterials. However, they performed well in freshwater eutrophication and water consumption.

The Flax PMMA is the worst performing material in several categories including GWP, ozone formation, fine particulate matter formation, terrestrial acidification, and fossil resource scarcity.

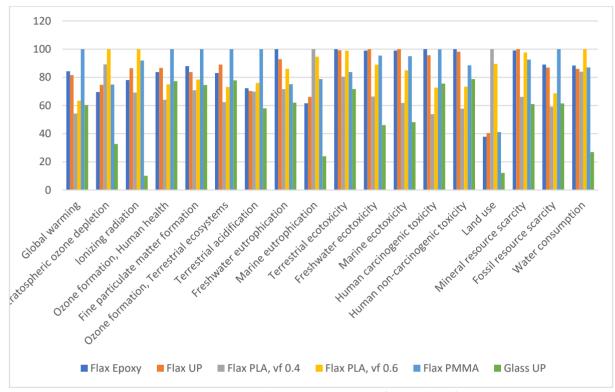


Figure 0.15: Relative LCA Comparison of Material Configurations

The epoxy and glass have similar relative contributions, this is due to the similar environmental impacts of the two resin systems as shown in Table 4.1. Though epoxy is consistently higher it has a lower density than glass, and so when compared by volume their impacts are similar.

Impact category	Unit	UP, 1kg	Epoxy,1kg
Global warming	kg CO2 eq	3.55E+00	5.06E+00
Stratospheric ozone depletion	kg CFC11 eq	1.20E-06	1.80E-06
Ionizing radiation	kBq Co-60 eq	1.58E-01	3.14E-01
Ozone formation, Human health	kg NOx eq	9.85E-03	1.10E-02
Fine particulate matter formation	kg PM2.5 eq	5.51E-03	7.48E-03
Ozone formation, Terrestrial			
ecosystems	kg NOx eq	1.16E-02	1.18E-02
Terrestrial acidification	kg SO2 eq	1.11E-02	1.53E-02
Freshwater eutrophication	kg P eq	1.11E-03	1.69E-03
Marine eutrophication	kg N eq	7.62E-05	1.22E-04
Terrestrial ecotoxicity	kg 1,4-DCB	1.14E+01	1.64E+01
Freshwater ecotoxicity	kg 1,4-DCB	1.70E-01	2.80E-01
Marine ecotoxicity	kg 1,4-DCB	2.22E-01	3.55E-01
Human carcinogenic toxicity	kg 1,4-DCB	1.18E-01	1.76E-01
Human non-carcinogenic toxicity	kg 1,4-DCB	3.15E+00	4.90E+00
Land use	m2a crop eq	5.19E-02	8.43E-02
Mineral resource scarcity	kg Cu eq	1.01E-02	1.66E-02
Fossil resource scarcity	kg oil eq	1.77E+00	2.17E+00
Water consumption	m3	3.70E-02	6.55E-02

For impacts such as marine eutrophication, land use, and water consumption the flax is the largest contributor. Therefore, the changes in the matrix play little difference, and any major differences between the configurations is due to the change in the relative difference of FVF in matrix and reinforcement.

#### Discussion

# System Model

The model used was 'allocation cut-off', where inputs and emissions are allocated to products and co products by either mass, financial value, or exergy. Because of this, the system relies heavily on non-uniform allocation assumptions often decided on what is easiest to model [54].

The cut off system model gives no credits for producing waste that can be used as inputs to other value creating processes. However, this means that utilising wastes from other processes results in a lower burden, resulting in worse performance for materials that primarily use virgin feedstock such as epoxy, UP, and PMMA, whilst favouring PLA due to the high by-product use.

#### **Functional Unit**

The functional unit was defined as 1 kg of material, with an expected lifespan of 20 years, and no impacts associated with the use stage. However, this is not representative of real world application, where many factors contribute to the amount of material required, and how it is manufactured. Different fibres and matrices will produce different mechanical properties, with many alternatives requiring thicker laminates to achieve the same function. The durability of composites is also paramount to their environmental performance, if parts have to be replaced at a greater frequency due to material change, then more parts have to be produced resulting in unexpected harm.

Therefore, the functional unit should encompass these aspects, it should be application specific and take into consideration any changes of durability or performance due to novel materials being used.

#### End of Life

Both PLA and PMMA are seen as potential alternatives to conventional materials due to viability of thermal recycling methods. As landfill was the only EoL scenario modelled, the benefits of these thermoplastic resins are not fully represented. It might be expected that modelling this will benefit the systems and show improvements on environmental benefit, however if considered against landfill, this may not be the case. LCA poorly captures

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the harm occurred through landfill or environmental plastic debris therefore the transport and processing demand of recycling materials may outweigh the advantages in current LCA impact methodologies [55].

## **Data Quality**

Most of the used data is reliant on the external providers ecoinvent and Corbion, both having IP restrictions on full disclosure. For example, the ecoinvent data set for epoxy resin, a major influencer of this LCA, is based on the paper by Ernst and Young's report [15] which is not currently in the public domain. Because of this the data cannot be scrutinised for the assumptions and modelling to be consistent with our own, and the accuracy of the data to actual production.

#### **Future Work**

This work solely focusses on a weight by weight comparison between different matrices of flax reinforced composites. As discussed, this does not reflect the real world application and so mechanical properties and durability should be properly considered to improve the accuracy of the analysis.

In addition, the present study LCA data was mainly collected from the literature at laboratory level, relevant LCA study with the data acquired at plant level will be the future work. The LCA study will be updated to a life cycle cost analysis when the relevant information is available.

# **Additional Notes**

Previous work at University of Plymouth relevant to the SeaBioComp project are summarised in open access webpages:

- Book chapters and review papers relevant to environmental issues and life cycle assessment for bast fibres and their composites: <u>Bast fibres and their composites webpage</u> {Summerscales, 2023 #886}.
- Quantified Composite Life Cycle Inventory data has been identified and is summarised: <u>LCA quantified environmental impacts webpage</u> {Summerscales, 2022 #887}.

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# Appendix Carife Cycle Inventory supporting Appendix Biocomposites production routes

SeaBioComp\_D3.5.2\_Appendix\_Spreadsheets\_Consolidated.xlsx

Sheets: Appendix B\_LCI\_Flax fibers Appendix B Composites

# **Appendix D: Life Cycle Assessment results supporting Appendix B.**

SeaBioComp\_D3.5.2\_Appendix\_Spreadsheets\_Consolidated.xlsx

Sheets: Appendix D\_Flax Epoxy

Appendix D\_Flax UP Appendix D\_Flax PLA, 0.4 Appendix D\_Flax PLA, 0.6 Appendix D\_Flax PMMA Appendix D\_Comparison Appendix D\_Graphs

Appendix D\_UP\_Epoxy Comp

# **Appendix E:**

# **Quality Assessment of Life Cycle Inventory Data for Composites**

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**Keywords**: Life Cycle Assessment (LCA), data quality, proxies

#### 1 General Introduction

Environmental sustainability over the life cycle of a product is rising as a key driver in the selection of materials and manufacturing processes. Life cycle assessment (LCA) is increasingly used as a standardised, science-based decision-making support tool to quantify and identify potential environmental impacts through all phases of the product life.

A major step involved in completing a Life Cycle Inventory (LCI), and so an LCA study, is the collection, analysis, and quality check of data on the investigated processes, data to quantify the inputs and output (e.g., elementary flow of raw materials, energy, waste, and co-products) of the product system that crosses the LCA study system boundary.

Complete data is a critical aspect and an integral part of any scientific endeavor and protocol. Data quality in LCI is the key success factor in the acceptance of LCA results, due to the nature of the validity methodology. Data quality is rarely considered by the LCA community (i.e., Sensitivity and Uncertainty Analysis, expert peer and critical third-party reviewers) leading to low confidence in the LCA interpretation.

With LCA, there is no feedback mechanism to filter out faulty analyses. Here, confidence in outcomes can be based only on the quality of the input data and the quality of the models used [1].

The survey of unresolved problems in LCA conducted by Reap *et al* [2], identified that the data quality was one of the problems to be only partially solved by existing methods. Consequently, this limited the power and reliability of LCA reported by a number of authors [3].

The first Society of Environmental Toxicology and Chemistry (SETAC) and United Nations Environment Programme (UNEP) workshop on LCA in the late 90s and early 2000s recommended broad guidelines on reporting data quality characteristics and tools in response to current LCA data quality standards of ISO 14040 series (currently ISO 14044/44:2006).

Various entities within the LCA community have developed different methodologies to address and communicate the data quality of LCI data. There are two dominant examples of semi-quantitative methods. First, the pedigree matrix approach refined and used by Ecoinvent database. Second, the data quality ranking system and the related guidelines used by the International Reference Life Cycle Data System (ILCD) which are included in the Sphera (was Gabi) database as one of the Data Quality Indicators (DQI). Also, a qualitative pass/fail method is used by the USDA LCA Commons.

The LCA community is still plagued by the lack of reproducible data quality, and data quality assessment is not currently widely practiced in LCA studies [4].

A clear example is the data quality assessment (DQA) of the sources and the generation of the major LCA databases (i.e., Ecoinvent, Sphera). Despite that, they are most informed in the metadata and in the integrated LCI documentation. The metadata are not fully transparent. Questions arise about the clarity and the quality of the data generation and the reliability of the original sources.

generator Ethernto-the sidatal selection user. When LCA for the main objectives of the study oduction routes specific materials data is not available, it is common practice to select "best fit proxies". The closeness of the match will affect the total study DOA. It is common to make full LCA studies and environmental declarations from LCA results using different sources and different proxies without considering the DQA of the LCI and the implications on the quality of the LCA results. This is a risky approach, as the environmental conditions can have different study goals and scopes. Also they may be very different supply chains, technologies, models, different regions, and countries [5].

## 2. Background, Goal, and Scope.

Over the past decade, the composites industry has recognised the importance of environmental sustainability as a major emerging contributor in all sectors leading to a substantial increase in the number of LCAs conducted and published in both the academic and grey literature.

The current rigid standards and guidelines use the world's most consistent and transparent LCI databases (Ecoinvent, Sphera) in addition to the European reference Life Cycle Database (ELCD) and, specifically for composites, the European Composites Industry Association (EuCIA) Eco Impact calculator database. Close examination has revealed concerns about quality of the data used in the conducted LCA studies.

The concerns were related mainly to the following issues. The LCI data in databases is often harvested from the academic literature which is peer reviewed but not subjected to rigorous audit. All the polyester resin data traces back to the same industry report that has not explicitly described the goal and scope and does not have a clear system boundary. Further, the report is not directly accessible in the public domain.

Similar concerns relate to the reproducibility of the available LCAs studies in composites, the variability of data results, the variety of background data set modeling, in addition the proxy selection methods for the composites in lieu of specific data, to name a few.

The potential broad range of values available in composites LCI databases and the proxies selection

may prove an issue for comparative LCA and provide misleading results leading to inaccurate conclusions and potentially condemning a composite solution relative to other materials.

The goal of this study is to provide LCA practitioners in the composites industry with an approach to assess the overall quality of LCI results by integrating the qualitative and quantitative information of input data, uncertainty, and sensitivity analysis of results.

- To review several LCA data assessment methods.
- To review the quality of composite materials data in the LCI databases and literature.
- To consider the implication of composites DQA on composites comparative LCA product results.
- To assess and integrate the DOA in the proxy selection methodology in LCI composite dataset.

Life cycle assessment (LCA) data quality issues and the approach are under investigation using a case study of a yacht production line.

#### Software Used

- Primary: SimaPro with Ecoinvent Database.
- Secondary: Gabi with Sphera Database.
- Eco Impact Calculator for composites.

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# **Appendix F:**

# **Investigation to Determine Whether Flow Medium is Needed for Natural Fibre Composites**

Lloyd Vance and John Summerscales School of Engineering, Computing and Mathematics (SECaM), University of Plymouth



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Following the "Initial test to determine whether flow medium in needed for natural fibre composites", further investigation was required to ascertain meaningful results. This investigation involved producing 4 sample flax fibre/Epoxy resin plates, 2 using a flow medium and 2 without, extracting samples from each to undergo 3-point bending tests and analysing their associated mechanical properties. This analysis would then be used to decide whether a flow medium is required when manufacturing a flax fibre composite using Resin Infusion under Flexible Tooling (RIFT).

# Manufacture

The manufacturing process and details for each plate are summarised in Tables F1-F4.

Table F7: Manufacturing Details - Flax/Epoxy Flow Medium 1

Manufacturing Details - Flax/Epoxy Flow Medium 1			
Composite Type Flax fibre/ IN2 Epoxy infusion resin			
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 77.5g, actual weight 240gsm		
Resin Used  Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)			
Manufacturing Method	nufacturing Method RIFT as per manufacturing method at end of this document.		
Cure Details	24-hour ambient cure, followed by overnight cure at 60 in oven		
Vacuum Details	vacuum achieved -> 12mbar, leak rate -> 0.5		
Lab Conditions	temperature 24°c, 62% humidity, 25/07/22		
Laminate Details 8-ply, 77.5g fabric used, Footage obtained			
Additional Details correct fabric/resin/catalyst, flow medium used			

Table F9: Manufacturing Details - Flax/Epoxy Non Flow Medium 1

Manufacturing Details - Flax/Epoxy Flow Medium 2				
Composite Type Flax fibre/ IN2 Epoxy infusion resin				
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 76.5g, actual weight 240gsm			
Resin Used  Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)				
Manufacturing Method	RIFT as per manufacturing method at end of this document.			
Cure Details	24-hour ambient cure, followed by overnight cure at 60 in oven			
Vacuum Details	vacuum achieved -> 11mbar leak rate -> 0.5			
Lab Conditions	temperature 24°c , 42% humidity 25/07/22			
Laminate Details	8-ply, 77.5g fabric used, Footage obtained			
Additional Details	correct fabric/resin/catalyst, flow medium used			

Manufacturing Details - Flax/Epoxy Non Flow Medium 1				
Composite Type	osite Type Flax fibre/ IN2 Epoxy infusion resin			
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 76.5g, actual weight 240 gsm			
Resin Used	Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight ratio)			
Manufacturing Method	od RIFT as per manufacturing method at end of this document.			
Cure Details	24-hour ambient cure, followed by overnight cure at 60 in oven			
Vacuum Details	vacuum achieved -> 10mbar leak rate -> 0.2			
Lab Conditions	temperature 24°C , 42% humidity 25/07/22			
Laminate Details	8-ply, 77.5g fabric used Footage obtained			
Additional Details	correct fabric/resin/catalyst no flow medium used			

SeaBioCompTable F10: Manufacturing Details of Flax / Epoxy Non Flow Medium 2 ction routes

Manufacturing Details - Flax/Epoxy non Flow Medium 2				
Composite Type Flax fibre/ IN2 Epoxy infusion resin				
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 76g, actual weight 240gsm			
Resin Used	Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)			
Manufacturing Method	acturing Method RIFT as per manufacturing method at end of this document.			
Cure Details	24-hour ambient cure, followed by overnight cure at 60 in oven			
Vacuum Details	vacuum achieved -> 10mbar leak rate -> 0.2			
Lab Conditions temperature 24°c , 42% humidity 25/07/22				
Laminate Details	8-ply, 77.5g fabric used Footage obtained			
Additional Details	correct fabric/resin/catalyst no flow medium used			

# **Composite Mechanical Testing**

Five 100 x 10mm samples were cut from each plate using a diamond cutter (University of Plymouth composites laboratory W7). Each suitable sample was tested in three-point bend configuration with a span-to-depth ratio of 16 using the Instron 5582 machine with a 100 kN load cell (University of Plymouth materials characterisation laboratory SMB001, calibration certificate expired) with the assistance of Katie Shore. The span of the supports was 80mm, matching that of the FEA and analytical calculations, and the crosshead speed was set to 1mm/min. Test data is in Tables F5 and F6. Images of the test rig can be seen at Figures F1 and F2.

**Table F11: Flow medium sample data** 

Flow Medium Sample Data					
Sample	Crosshead Speed	Thickness	Width		
,	[mm/min]	[mm]	[mm]		
1.1	1	5.4	11.5		
1.2	1	5.4	11.5		
1.3	1	5.0	10		
1.4	1	5.0	10		
1.5	1	5.4	11.5		
2.1	1	5.5	12		
2.2	1	5.5	12		
2.3	1	5.5	16		

Table F12: Non-Flow medium sample data

Non-Flow Medium Sample Data					
Sample	Crosshead Speed	Thickness	Width		
	[mm/min]	[mm]	[mm]		
1.1	1	4.6	13		
1.2	1	4.6	13		
1.3	1	4.8	14.2		
1.4	2	4.8	12.7		
1.5	2	4.6	12		
2.1	1	4.5	11.5		
2.2	1	4.5	9.5		
2.3	1	4.5	14		
2.4	1	4.5	10.3		





Figure F16: A test sample mounted in the test rig (Vance, 2022)

Figure F17: Close up of the rig in the test frame (Vance, 2022)

Tables F7 and F8 show the fibre volume fraction (FVF) and flexural modulus of each sample, with those in blue representing the flow medium samples and those in orange representing the non-flow medium samples. The sample nomenclature 1.1 denotes "test plate 1, sample 1". The table for the flow medium samples also includes an 'adjusted' fibre volume fraction, FVF<sub>a</sub>, based on the laminate thickness without the adhered flow medium.

**Table F13: Fibre volume fraction data for flow medium samples** 

	Fibre Volume Fraction (FVF) Data - Flow Medium							
Sample	Thickness	Adjusted Thickness	Fabric areal weight	Component mass	Layers	Fibre density	FVF	FVFa
	[mm]	[mm]	[kg/m2]	[kg]	[n]	[kg/m3]	[ratio]	[ratio]
1.1	5.40	4.40	0.243	0.25	8	1270	0.28	0.35
1.2	5.40	4.40	0.243	0.25	8	1270	0.28	0.35
1.3	5.00	4.00	0.243	0.25	8	1270	0.31	0.38
1.4	5.40	4.40	0.243	0.25	8	1270	0.28	0.35
1.5	5.40	4.40	0.243	0.25	8	1270	0.28	0.35
2.1	5.30	4.30	0.243	0.25	8	1270	0.29	0.36
2.2	5.50	4.50	0.243	0.25	8	1270	0.28	0.34
2.3	5.50	4.50	0.243	0.25	8	1270	0.28	0.34

SeaBioCompTableiF14: Fibre volume fraction data for infor-flow medium samples tion routes

Fibre Volume Fraction (FVF) Data – Non Flow-Medium						
Sample	Thickness	Fabric areal weight	component mass	number of layers	Density of fibre	FVF
	[mm]	[kg/m2]	[kg]	[n]	[kg/m3]	[ratio]
1.1	4.60	0.243	0.25	8	1270	0.33
1.2	4.80	0.243	0.25	8	1270	0.32
1.3	4.803	0.243	0.25	8	1270	0.32
1.4	4.50	0.243	0.25	8	1270	0.34
1.5	4.60	0.243	0.25	8	1270	0.33
2.1	4.50	0.243	0.25	8	1270	0.34
2.2	4.50	0.243	0.25	8	1270	0.34
2.3	4.50	0.243	0.25	8	1270	0.34
2.4	4.50	0.243	0.25	8	1270	0.34

The fibre volume fraction (V<sub>f</sub>) of each sample was calculated using Equation F1:

$$V_f = \frac{n*A_F}{\rho_f*t} \tag{F1}$$

where V = fibre volume fraction, n = number of layers,  $A_F$  = areal weight of fabric,  $\rho_c$  = density of the fibre and t = thickness of the component. The volume fraction data is presented in Tables E7 and E8.

The flexural modulus,  $E_F$ , for each specimen was calculated using Equation F2:

$$E_F = \frac{S^3 * m}{4 * w * t^3} \tag{F2}$$

where S= span between the supports (to  $\pm 0.2\%$ ), m= slope of the linear portion of the load/deflection graph (N/m), t= specimen thickness and w= specimen width.

The flexural moduli data is presented in Tables F9 and F10.

**Table F15: Flexural modulus data for flow medium samples** 

Flexural Modulus Excel Calculations – Flow medium					
Sample	Width	Thickness	Slope	Modulus	
	[mm]	[mm]	[N/m]	[GPa]	
1.1	11.5	5.4	62500	4.42	
1.2	14.0	5.4	56497	3.28	
1.3	10.0	5.0	45000	4.61	
1.4	11.5	5.4	45000	3.18	
1.5	11.5	5.4	45000	3.18	
2.1	12.0	5.3	57500	4.12	
2.2	13.5	5.5	60000	3.42	
2.3	16.0	5.5	91111	4.38	

SeaBioComp-WablerF16: Flexural modulus data for nonf-flow medium samples luction routes

Flexural Modulus Excel Calculations					
Sample	Width	Thickness	Slope	Modulus	
	[mm]	[mm]	[N/m]	[GPa]	
1.1	13.0	4.6	47500	4.80	
1.2	11.5	4.8	50000	5.03	
1.3	14.2	4.8	47500	3.87	
1.4	12.7	4.5	41500	4.59	
1.5	12.0	4.6	47500	5.21	
2.1	11.5	4.5	43000	5.25	
2.2	9.5	4.5	40000	5.91	
2.3	14.0	4.5	51000	5.12	
2.4	10.3	4.5	39231	5.35	

# **Results and discussion**

Non Flow Medium Data

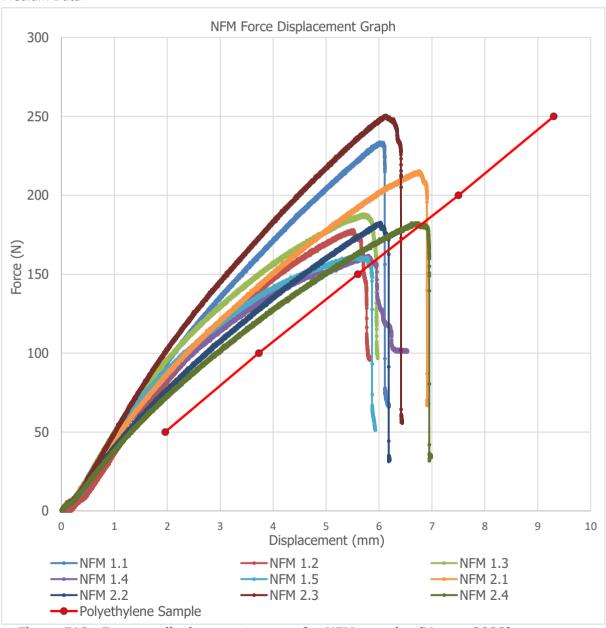


Figure F18: Force vs displacement curves for NFM samples (Vance, 2022)

Figure F3 shows the force/displacement curves for the non-flow medium (NFM) test samples, along with a predicted force/displacement line for a 7 mm thick polyethylene sample which represents the material currently used for the component. Table F11 presents the FVF and flexural moduli for each NFM sample.

SeaBioTable F17: Wolume fractions and flexural moduli for non-flow medium samples outes

	NFM Modulus and FVF Data			
Sample	FVF	Modulus		
1.1	0.33	4.80		
1.2	0.32	5.03		
1.3	0.32	3.87		
1.4	0.34	4.59		
1.5	0.33	5.21		
2.1	0.34	5.25		
2.2	0.34	5.91		
2.3	0.34	5.12		
2.4	0.34	5.35		

Figure F3 shows that 6 of 9 samples failed at a loading forces between 150-185 N, with a failure displacement between ~6-7 mm. The consistency of these results indicates that there is good uniformity of mechanical properties across both of the NFM plates. This therefore suggests that the resin had impregnated fibres throughout the composites, because issues with resin flow would cause dry spots within the composites, which would create local stress concentrations and poor bonding at the fibre/matrix interface and lead to premature failure in affected regions.

Another interesting finding is that the stiffest two curves are samples 2.2 and 2.4, with flexural moduli 5.91 and 5.25 GPa respectively, which were taken from different sections of their plate. This indicates that the composite stiffness is not necessarily related to a specific region of the plate, demonstrating that the resin was effectively permeating the fibres and reaching all areas of the plates without a flow medium. As both of the stiffest samples were from the same plate there could be suggestions that factors of the manufacturing process, such as vacuum pressure obtained, affected the quality of the samples. However, as all of the samples across both plates achieved a FVF between 32% and 34%, this suggests that the difference in manufacturing procedure was relatively small. Consequently, it is possibly more useful to consider that 5 of 9 of the NFM test samples achieved a flexural modulus greater than 5GPa, which reflects the consistency of NFM samples performance and manufacture.

The achieved volume fractions were consistent, but low, which could be due to a number of factors. One reason for the low fibre volume fraction is the effect of fibre swelling during infusion. The swelling of flax fibres means that resin uptake is greater than that seen in other fibre such as glass and carbon, which can lead to a reduced fibre volume fraction (EasyComposites flax fibre in composites). Flax fibres also have a unique microstructure, consisting of four walls of microfibrils with a hollow lumen in the centre (Phillips, 2013). This hollow centre increases possible channels for resin flow compared to carbon and glass, and therefore improves resin flow through the laminate. The increased resin uptake caused a reduced fibre volume fraction compared to reinforcements such as carbon and glass fibres. Poor fibre volume fractions could also be a result of shortcomings in the manufacturing method. For example, the plates manufactured for this investigation were connected to vacuum pumps prior to and during the infusion process, but were then disconnected during the cure period. This means the vacuum bags were continually losing vacuum pressure due to bag leaks, which could significantly reduce the consolidation of the composite and encourage void formation, in turn reducing fibre volume fraction.

On a broader scale, despite the shortcomings discussed previously, the graph above shows that each of the NFM samples produced a curve with a gradient similar to that given by the polyethylene sample. This suggests that the samples have a flexural modulus similar to that of the existing 7mm thick rotomoulded polyethylene component, despite being 43.5% thinner (4.5mm), which will reduce the mass of the component while retaining the desired stiffness.



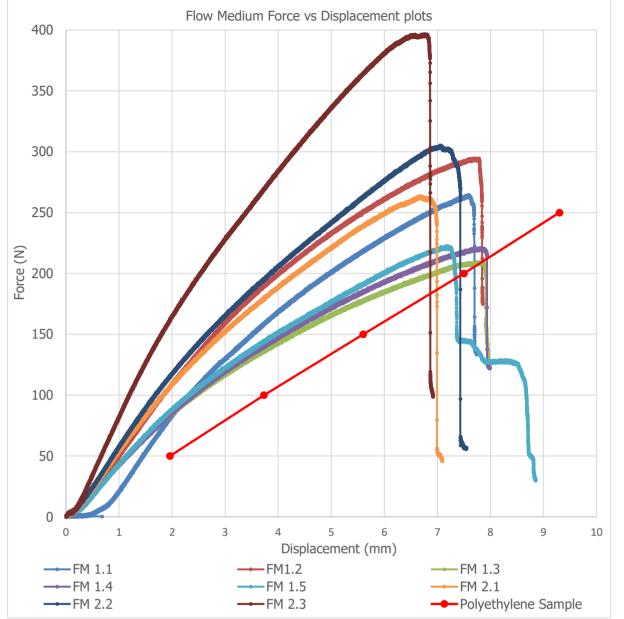


Figure F19: Force/displacement curves for the flow medium samples (Vance, 2022)

Figure F4 shows the force/displacement curves for the flow medium (FM) test samples, along with a predicted force/displacement curve for a 7 mm thick polyethylene sample being the material currently used for the component. Table F12 shows a summary of the modus and FVF for each FM sample.

SeaBioTable F18: Wolume fractions and flexural moduli for non-flow medium samples outes

Flow medium samples					
Sample	Modulus	Adjusted Modulus	FVF	FVFa	
	[Gpa]	[Gpa}	[%]	[%]	
1.1	4.42	8.17	0.28	0.35	
1.2	3.28	6.06	0.28	0.35	
1.3	4.61	9.00	0.31	0.38	
1.4	3.18	5.88	0.28	0.35	
1.5	3.18	5.88	0.28	0.35	
2.1	4.12	7.71	0.29	0.36	
2.2	3.42	6.24	0.28	0.34	
2.3	4.38	8.00	0.28	0.34	

Figure F4 shows the force/displacement curves for each of the FM samples. 7 of the 8 samples failed at a load between 200-300 N, with a displacement between 6-7 mm. Given that each sample had the same length, the failure strain for each sample remained fairly constant, while the failure load was much more varied. This difference in failure load resulted in the flexural modulus of the samples also being varied.

There was only a 3% difference in achieved fibre volume fraction for the FM samples (28%-31%), the difference in flexural modulus between samples does not appear to be caused by an inconsistent fibre volume fraction. The variation in modulus may be influenced to the quality of the bonding at the fibre/matrix interface, or void content. However, the capability of the resin to permeate flax fibres (especially with the use of a flow medium) combined with the high vacuum pressure achieved during the manufacturing process suggests that the laminates should not have a high void content.

Another factor which may have contributed to this may be that the flow medium used in the manufacturing process was unable to be separated from the laminate post curing. As such, all test samples had the flow medium still bonded to the lower (tensile) face during testing. As the author has no experience with this happening before, it is difficult to quantify the extent to which the flexural modulus was affected. However, the flow medium has mechanical properties much lower than that of the composite, and is very easily pulled apart, so it was assumed that the effect of the flow medium itself on the flexural modulus was small.



Figure F20: Image showing flow medium still bonded to test sample (Vance, 2022)

Figure F5 shows the flow medium still bonded to the tensile surface and generating an extremely resin rich volume (RRV) on this face, which may have impacted sample failure. As the upper and lower surfaces are the regions of the sample which lie the furthest from the neutral axis, they experience the greatest deflection, and therefore the largest strain values. As such, given that RRVs are known to negatively impact all composite mechanical properties (Mahmood et al, 2022), this region may have played a role in either reducing the mechanical properties of the composite. The aforementioned source also states that "RRV are strongly implicated in the initiation and propagation routes for crack growth", meaning this RRV could also have had a role in the failure mechanism of the samples, possibly causing a premature failure of samples.

Despite the comments above, the force/displacement graph shows that the samples still meet the most desirable characteristic – that they are stiffer than the existing 7mm thick polyethylene rotomolded component. As such, despite the shortcomings, the composite would meet the stiffness requirements of the project. Consequently, given that these samples were manufactured and tested with the purpose of comparing the general stiffness of flax/epoxy composites manufactured with and without a flow medium, the exact values of the achieved modulus may not be critical to the investigation, if meaningful conclusions could be drawn. As the curves were broadly similar, with no obvious areas of concern or unexpected failure, it was concluded that these samples were suitable in the context of the broader investigation.



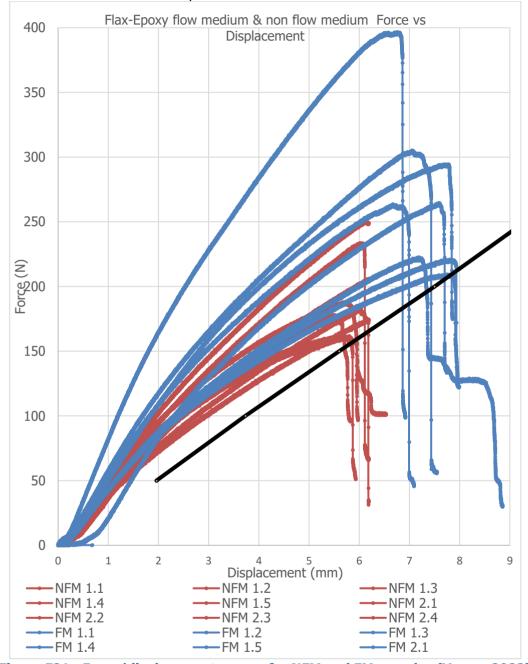


Figure F21: Force/displacement curves for NFM and FM samples (Vance, 2022)

Figure F22: NFM, FM and adjusted FM flexural modulus vs fibre volume fraction (Vance, 2022)

Adjusted Modulus and Vf

Figures F6 and F7 show the force/displacement curves for each of the test samples and a reference polyethylene line, with the NFM samples coloured in orange and the FM samples in blue. At first, this graph appears to show that the FM samples have a stiffer modulus that the NFM samples, however, this is likely to be due to their increased thickness. As such, the fibre volume fraction vs modulus graph appears to be a more accurate representation of the data.

FM Vf vs Modulus

Figure F6 (the fibre volume fraction *vs* modulus graph) shows the data for the FM samples in blue, the data for the NFM samples in orange, and an "adjusted" modulus and FVF in grey. This adjusted data is based on the assumption that the NFM samples would be 1mm thinner without the flow medium being attached, while retaining the same slope.

The FVF vs modulus graph therefore shows that all of the NFM samples achieved a higher fibre volume fraction than the FM samples, but this is likely to be due to the increased thickness caused by the flow medium still being bonded to the samples, rather than a significantly improved laminate. As such, the "adjusted" data shows that if the flow medium was able to be separated, the FVF would be between 34% and 35%, which is slightly greater than that of the NFM samples. The "adjusted" data also shows that the FM samples would have a modulus of between 6 GPa and 8.2 GPa, which would either match or exceed that of the highest performing NFM sample.

However, it is worth considering that the "adjusted" values are purely hypothetical, and are based on the assumption that the flow medium can be removed without damaging the laminate, which seems ambitions considering it is currently not able to be removed at all. Consequently, it may be more useful to draw conclusions from the data which was obtained during the tests and consider the "adjusted" values as an area for further study.

With this in mind, the FVF vs modulus graph shows that the NFM samples significantly outperformed the FM samples; NFM samples had an average fibre volume fraction 15% greater than the average FM fibre volume fraction, and NFM samples had an average modulus 27.4% greater than the average FM samples. As such, this study suggests that the demonstrator component would possess a higher fibre volume fraction and flexural modulus if a flow medium was not used. The exclusion of a flow medium would also reduce the amount of waste material consumed during manufacturing, coinciding with the broader scope of the project, which is to produce bio-based composites for marine applications which have a minimal environmental impact.

NFM Vf vs Modulus

# Conclusion

- All samples achieve a flexural modulus equal to or greater than the 7mm thick polyethylene sample, while remaining up to 45.5% thinner.
- Samples with no flow medium have an average fibre volume fraction 15% higher than the average achieved by the flow medium samples.
- Samples with no flow medium have an average flexural modulus 27% higher than the flow medium samples.
- "Adjusted" figures show that flow medium samples have potential to achieve a higher fibre volume fraction and flexural modulus than non flow medium samples, if the flow medium can be removed without compromising laminate quality.

## **Recommendations For Future Work**

 Devise a manufacturing method which facilitates the removal of flow medium after curing and repeat investigation

# References

- Mahmood, A.S., Summerscales, J. and James, M.N., (2022). <u>Resin-Rich Volumes (RRV) and the performance</u> of fibre-reinforced composites: a review. Journal of Composites Science 6, 53.
- Phillips, S. <u>Characterization of flax fibres for application in the resin infusion process</u>, PhD thesis, McGill University (Canada), 2013.

# RIFT manufacturing method for flax/epoxy composite plate production routes

- 1. Select a glass plate and using a sharp blade remove any cured resin remaining on the surface and then wipe the plate clean with solvent.
- 2. Stick (blue) tape around the edge of the plate where the tacky tape will eventually be placed.
- 3. Apply release agent to the plate and allow dry.
- 4. Record the time, temperature, pressure and relative humidity
- 5. Place the individual lamina onto the centre of the glass plate in the required stacking sequence.
- 6. Place a sheet of peel ply over the laminate
- 7. Place the transport mesh/flow medium on top of the stack such that it is >10 mm inside the laminate edge on both sides and along the length of the laminate.
- 8. Stick (yellow) dam tape around the edges of the laminate
- 9. Cut a 800 mm length of inlet pipe and place at one end of the laminate
- 10. Notch the end of the pipe which will go into the resin pot.
- 11. Cut a 800 mm length of vacuum outlet pipe sufficient to connect to the resin trap and place at other end of the laminate
- 12. Cut bagging film to size such that there is around 200 mm excess in both directions.
- 13. Stick a square border of tacky tape to the edges of the glass plate
- 14. Remove (white protective) tape
- 15. Place inlet and outlet tubes in desired locations at each end of the laminate
- 16. Stick additional layer of vacuum tape over the tubes where they lie over the existing tape
- 17. Drape vacuum bag over the plate and complete the sealing of the bag by systematically pressing the bag to the tape
- 18. Using a permanent marker, mark an arrow indicating the resin flow direction onto the bag.
- 19. Record the time, temperature, relative humidity and pressure.
- 20. Attach a pressure meter to the inlet pipe.
- 21. Attach the outlet pipe to the resin trap and apply a vacuum to the bag, smoothing the bagging film away from the laminate area.
- 22. Identify any leaks in the bag and seek to achieve a vacuum level of ~20 mbar on the pressure gauge.
- 23. Isolate the vacuum (crimp the outlet pipes in a couple of places) and record the rate of pressure increase on the gauge.
- 24. Re-introduce the vacuum and continue to improve the seals of the bag until the rate of pressure drop is 1 mbar/minute or less.
- 25. Clamp the inlet pipe and remove the pressure gauge.
- 26. Calculate the quantity of resin (see below) required to fill the laminate, flow medium and feed pipe.
- 27. Mix the resin and hardener/catalyst/accelerator in the given proportions (4% hardener)
- 28. Fix the resin pot to a support, then insert the notched end of the pipe.
- 29. Unclamp the inlet pipe for just long enough that the resin rises to the clamp position, then reclamp.
- 30. After 30 seconds (to allow air displaced from the pipe to be evacuated from the bag), open the clamp and resin will flow into the bag.
- 31. Once the flow front has reached the outlet pipe, and assuming the plate has filled, clamp the inlet pipe to stop further resin inflow.
- 32. If possible, with Unsaturated Polyester Resin reduce the vacuum level to ~500 mbar absolute and leave the moulding under vacuum until the resin gels.
- 33. Record the time, temperature, pressure and relative humidity
- 34. If required, postcure at the appropriate temperature for the required time in the oven.
- 35. The plate will be post-cured according to the resin manufacturer's recommendations.

# Appendix G: Flow medium and peel ply investigation (principal author Lloyd Vance) Flax/Epoxy Composite Flow Medium Removal Investigation

#### **Previous Work**

Prior to the Flax/Epoxy composite investigation, initial infusions were carried out using 200gsm 2x2 twill weave Flax reinforcement fibres and easy composites IP2 unsaturated polyester resin. The purpose of these infusions was to gain familiarity with the resin infusion under flexible tooling (RIFT) process, especially with flax fibres, and to ascertain what consumable materials may be required in the lay-up.

Following initial infusions, and investigation was carried out to ascertain whether 200gsm 2x2 twill weave Flax reinforcement fibres /IN2 epoxy infusion resin composites require a flow medium to be included in the manufacturing process. This investigation concluded that:

- All samples achieve an effective panel stiffness equal to or greater than the 7mm thick polyethylene sample, while saving mass by being up to 45% thinner.
- Samples with no flow medium have an average fibre volume fraction 15% higher than the average achieved by the flow medium samples
- Samples with no flow medium have an average flexural modulus 27% higher than the flow medium samples
- "Adjusted" figures show that flow medium samples have potential to achieve a higher fibre volume fraction and flexural modulus than no-flow-medium samples, if the flow medium can be removed without compromising laminate quality.

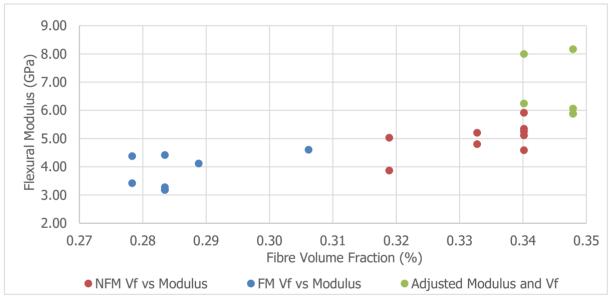


Figure F1: Comparison of flexural moduli of flax/epoxy composite with or without flow medium against achieved fibre volume fraction

This work recommended that a manufacturing method which facilitates the removal of flow medium after curing should be devised and the investigation should be repeated.

As such, an investigation into developing a manufacturing method which facilitates the removal of the flow medium after curing was initiated, with a view to testing samples and comparing to previously obtained results to ascertain whether the proposed method of flow medium removal has an effect on composite mechanical properties.

#### Introduction

This work aims to further optimise the Resin infusion under flexible tooling (RIFT) manufacture of flax fibre/epoxy resin composite laminates by eradicating the issue of peel ply removal post cure. This aims to be done by either defining a process which facilitates the removal of the peel ply and flow medium post cure, or concluding that it is more effective to remove a flow medium and peel ply from the manufacturing process.

#### Plate Manufacture

Mechanism 1 – Loctite Release Agent

The first proposal for flow medium removal was to carry out the standard infusion manufacturing process at University of Plymouth (Appendix K), but with a coat of Loctite release agent applied to the top flax ply of the layup prior to the addition of the standard PP230 Nylon 66 peel ply and flow medium.

The reasoning behind this being that the bonding at the epoxy/peel ply interface would be significantly poorer, which would therefore facilitate its removal after curing. However, there were also concerns with this proposal, as the addition of Loctite may have negatively an affected the mechanical properties of the composite. This is because the Loctite will have permeated the fibres of the uppermost ply, which could therefore affect the interfacial bond between the flax fibres and the epoxy resin system. This could encourage void formation and inhibit effective load transfer between the fibres and matrix, which would ultimately lead to poor composite mechanical properties and premature failure.







Figure F2: Loctite 770-NC label and plates FM3 and FM4.

The plates manufactured can be seen in Figure F2. The surfaces seen above are the "top" of the composites, where the Loctite was applied to and where the peel ply was removed. It is worth noting that even with the presence of a release agent, the peel ply still required a large degree of force to be removed with the aid of pliers, which suggests the peel ply was still bonded to the upper ply with a significant strength.

The absence of surface breaking voids in the peel ply region of the "FM3" plate suggests that the release agent may not have encouraged void formation as much as anticipated. It also suggests that there may not be a significant void volume at the bonding interface between the fibre/matrix on the other side of the ply, indicating that composite mechanical properties may not have been significantly impacted by the presence of the Loctite release agent.

However, the "FM4" plate appears to have significant voids in the upper region of the surface, along with stress whitening of the matrix. This is probably because the peel ply in this region was particularly difficult to remove. Consequently, the voids may be a result of the peel ply removal process, rather than defects resulting from manufacturing.

Mechanism 2 - Glass fibre Peel Ply

This method involved manufacturing the composite plates as per the same manufacturing procedure (Appendix K), with a 2x2 twill weave glass fibre peel ply (GFPP) replacing the previous PP230 Nylon 66 Peel Ply.

The reason behind this being that the lower resin uptake of the glass fibre would produce weaker bonding at the resin/peel ply interface, allowing the peel ply to be removed more easily after laminate curing. This method initially seemed more favourable than the Loctite method described previously due to the absence of substances being introduced to the fibre layup which could negatively impact the mechanical properties of the resulting composite.



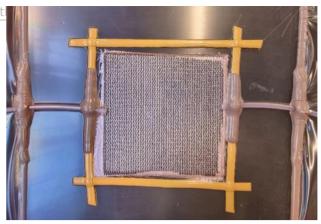


Figure F3: (left) the fibreglass peel ply above the laminate stack before bagging. (right) peel ply and flow medium laid over the fibres under vacuum conditions

Figure F3 (left) shows the thin glass fibre twill waeve pel ply (GFPP) laid on top of the flax fibres, while Figure F3 (right) shows the peel ply and flow medium laid on top of the fibres compressed under vacuum conditions. The GFPP was draped over the edge of the flax fibre layup to ease removal of the ply from the laminate after laminate cure.





Figure F4: The cured laminate after attempting to remove the peel ply at panel scale (left), and detail of a corner (right).

Figure F4 shows the cured laminate, with the results of attempting to remove the peel ply shown in the top left and bottom right regions of the laminate. This shows that the excess GFPP which draped over the edge of the laminate did help initiate a peeling action, however, this was halted approximately 20mm into the laminate. Instead of the glass fibre/resin interface failing and the glass fibre "peeling" off the laminate, the glass fibre itself ripped and resulted in the remaining GFPP being bonded to the laminate, unable to be removed.

This showed that the bonding in the GFPP/resin interface was much stronger than anticipated, resulting in the GFPP not able to be removed, leading to the conclusion that the GFPP is not a suitable peel ply for a flax/epoxy composite. As the peel ply was unable to be removed, this plate was not tested and fibre volume fraction not calculated.

Mechanism 3 -Non Flow Medium/ No Peel Ply

This method involved infusion manufacture of composite plates (Appendix K), with the omission of a peel ply and flow medium.

The reasoning behind this was that previous work has shown the flow medium is not essential, so can be removed. The previous manufacturing undertaken during this work has also shown that despite how well the peel ply and flow medium are bonded to the laminate, the vacuum bag is always removed with ease. As such, without the requirement for a peel ply to separate a flow medium from the laminate, or to stop the bag sticking to the laminate, the peel ply and flow medium can be removed completely.

[D. 3.5.2 LCA for the different biocomposites production routes



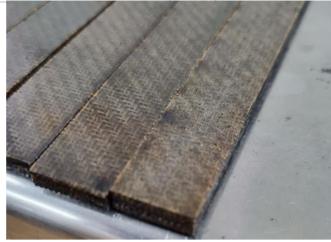


Figure F5: Test samples extracted from the NFM/PP laminate by laser cutting (left) and close-up (right) indicating an extremely low surface breaking void volume.

Figure F5 shows the test samples extracted from the NFM/PP laminate by laser cutting, with a close-up of the laminate surface indicating an extremely low surface breaking void volume. This indicates that, as seen previously, the flax fibres exhibit high resin uptake without a flow medium, and the vacuum consolidation during manufacturing has minimised the air content within the laminate.

However, there appears to be some discolouration on the surface of the samples. This may be a consequence of the high temperatures during laser cutting causing some burning of the matrix, rather than condensation of moisture within the laminate. This is because, as seen in Figure 5, even though there is decolouration, there is no evidence of moisture or water droplets within the laminate. In addition, this plate was manufactured in very similar environmental conditions as the rest of the plates, none of which exhibit moisture within the laminate, so there is also no cause to believe moisture was introduced to this laminate during manufacturing.

## **Testing**

Five 100 x 10 mm samples were taken from the Loctite and GFPP plate by the laser cutter in SMB XXX by Hannah Poulson. However, upon revising the ISO14125-1998 standard, samples from NFM/PP were 135mm x 15mm.

Each sample from the Loctite and GFPP plates were tested in three-point bend configuration with a span-to-depth ratio of 16 using the Instron 5582 machine with a 100 kN load cell (University of Plymouth materials characterisation laboratory SMB001, calibration certificate expired, Figure F6) with the assistance of Katie Shore. The support span was 80mm, matching that of the FEA and analytical calculations, and the crosshead speed was 1mm/min. These testing parameters were selected to remain in accordance with those used during the MATS347 module. However, the NFM/PP samples were tested with a 90mm span, in accordance with the aforementioned ISO standard, and a 1KN load cell.

The fibre volume fraction was calculated using Equation 1. The parameters in Equation 1 are Vf = fibre volume fraction, n = number of layers, AF = Areal weight of fabric,  $\rho f =$  assumed fibre density at 1270 kg/m3 and t = thickness of the sample. For the FM samples, overall Vf (left) and a laminate Vf assuming 1.0 mm flow medium (right) thicknesses were calculated.

$$V_f = n A_F / \rho_f t$$



Figure F6: Instron 5582 universal testing machine

Results Discussion Loctite Release Agent

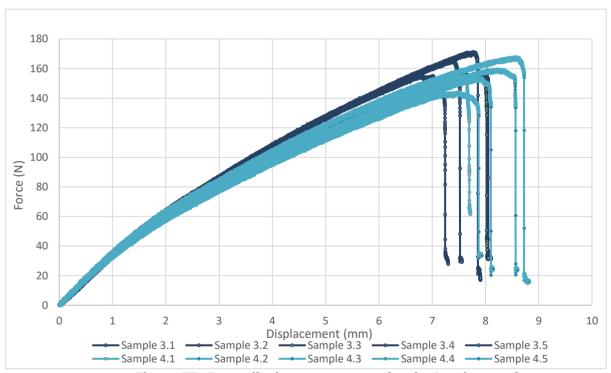


Figure F7: Force displacement traces for the Loctite specimens

SeaBioComp Table F1: Test data for the 8-ply/Loctite samples with flow medium routes (flax fibre density 1270 kg/m³, fabric areal weight 243 gsm, test crosshead speed 1 mm/min)

	Testing Data – Loctite Samples		
Sample	Thickness	Vf	Modulus
	[mm]	[n/a]	[GPa]
3.1	4.20	0.36	5.88
3.2	4.20	0.36	5.88
3.3	4.20	0.36	5.88
3.4	4.30	0.36	5.48
3.5	4.30	0.36	5.48
4.1	4.40	0.35	5.06
4.2	4.20	0.36	5.88
4.3	4.40	0.35	5.11
4.4	4.40	0.35	5.11
4.5	4.40	0.35	5.11

The data in Table F1 shows that all the samples achieved a near identical fibre volume fraction of approximately 35%. However, the calculated flexural modulus values have a slightly larger degree of variance, ranging from 5.11GPa to 5.88GPa, which is likely due to a variance in the slope of each curve. The variance in slope could be due to a 100KN load cell being used within 2% of the system load capacity, which introduces inaccuracies in the data recorded by the Instron machine and therefore potential inaccuracies in the load/displacement data obtained. The variance could also be due to manufacturing defects, but given the similarities in factors such as achieved vacuum consolidation and leak rate, this is relatively unlikely to have an effect large enough to reduce the flexural modulus by 13% compared to the maximum value (5.88GPa).

# Non Flow Medium/Peel Ply

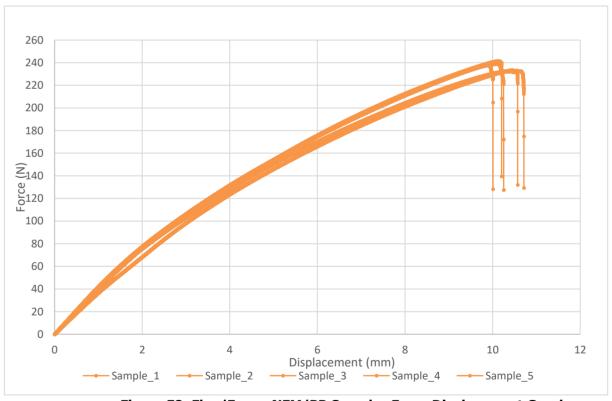


Figure F8: Flax/Epoxy NFM/PP Samples Force Displacement Graph

SeaBioComp—Valable F2: Test data for the No/Flow Medium/Peel Plye samples duction routes (flax fibre density 1270 kg/m³, fabric areal weight 243 gsm, test crosshead speed 1 mm/min)

Testing Data – NFM/PP Samples			
Sample	Modulus	Thickness	FVF
	[GPa]	[mm]	[n/a]
1	4.29	4.30	0.36
2	4.29	4.30	0.36
3	3.76	4.30	0.36
4	4.29	4.30	0.35
5	4.29	4.30	0.35

The data in Table F2 shows that the samples all achieved a fibre volume fraction of approximately 35%, with very little variance. This indicates that, without a flow medium or peel ply, the resin is still effectively impregnating fibres throughout the composite. This in turn is a consequence of the characteristics of cellulose-based fibres, such as their coarse nature and fibre structure allowing for high resin uptake, as discussed in the "Investigation to determine whether flow medium in needed for natural fibre composites" report by Lloyd Vance and John Summerscales.

The calculated flexural modulus of these samples is also very consistent, with all but one sample achieving approximately 4.3GPa. This is due to the similar thickness and slope of of each sample. The consistent thickness is further evidence of the good uptake throughout the laminate. It also shows that a vacuum pressure was exerted uniformly across the surface of the laminate during the infusion and cure cycle. An non-uniform vacuum pressure (caused locally by a small leak in the bag) would cause a varying laminate thickness as vacuum pressure varied across the laminate surface.

# Comparisons

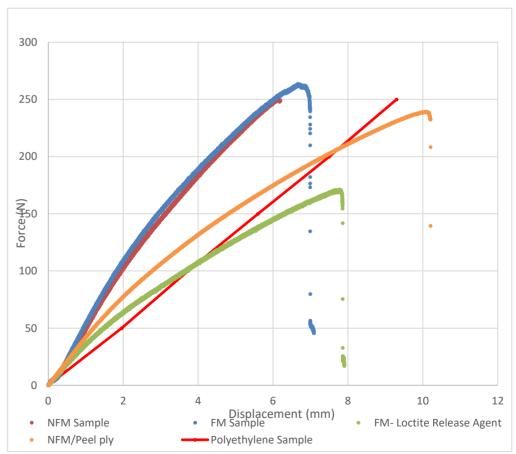


Figure F9: Force displacement curves for the flax/epoxy composites

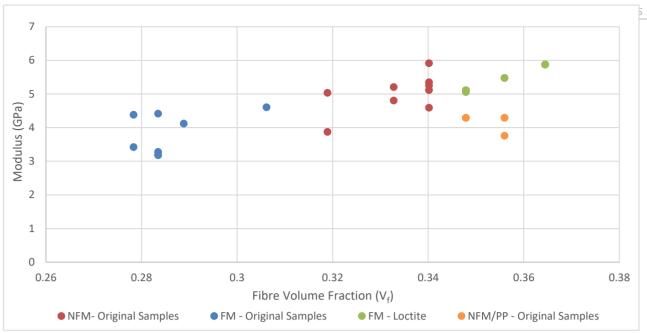


Figure F10: Modulus of the flax/epoxy samples plotted against fibre volume fraction

Figure F10 shows the fibre volume fraction (FVF) and flexural modulus exhibited by each of the composite samples produced so far in this investigation. This clearly shows that the "Loctite" and "NFM/PP" samples achieved a greater FVF than either of the samples produced in previous work. As the number of layers, fabric areal weight, and density of fibres are identical to that of the previous samples, the increased FVF suggests that the removal of the flow medium significantly reduces the thickness of the resulting composite laminate, which has caused the FVF to be greater. This could also mean that there is a reduction in the volume of resin seen at the "upper" surface of the test samples, which could also impact mechanical properties. However, further work would be required to quantify this statement.

The "Fibre Volume Fraction vs Modulus for Flax/Epoxy Samples" graph also shows that the modulus calculated for the NFM/PP samples was lower than that of the Loctite samples. This may be a result of the fact that the the samples extracted from each plate were different dimensions. As mentioned previously, the Loctite samples were 100x10 with an 80mm spane, while the NFM/PP samples were 135x15 with a 90mm span. The flexural modulus calculations for the NFM/PP samples are likely to be the most accurate, as the sample dimensions were in accordance with ISO standards. As such, even though the flexural modulus calculations enable useful qualatative comparrisons between the NFM, FM, and Loctite samples, the quantative values must be discarded due to the invalid test sample dimensions. As the NFM/PP samples (with correct dimensions) exhibited a fibre volume fraction similar to that of the Loctite samples, it is reasonable to assume the two sets of samples also had a similar flexural modulus. Consequently, the data shows that the Loctite and NFM/PP samples achieved a higher flexural modulus than the FM and NFM samples.

Assuming the fibre volume fraction does indicate that the Loctite and NFM/PP samples had a similar flexurla modulus, this suggests that introducing the Loctite release agent to the flax fibre reinfiorcement does not negatively impact the mechanical properties of the composite. However, before this can be fully justified, the Loctite composite testing must be repeated using samples with valid dimensions, as this will allow direct quantatative comparrison with the NFM/PP samples.

The force/Displacement graph above shows the curves given by representative FM/NFM samples from the previous "Investigation to determine whether flow medium in needed for natural fibre composites" report, along with curves from the Loctite sample, NFM/PP sample and the computational predictions from a 7mm thick polyethylene sample. This is not necessarily useful to draw quantitative conclusions.

However, it is useful to give context to the broader scope of the work, allowing for qualitative comparison between each of the composite samples tested so far and the material which they are ultimately being designed to replace. As such, at a purely "skin deep" assessment it can be seen that each of the samples so far achieves a greater stiffness than the current component until approximately 3mm deflection, a value which is unlikely to be achieved during the component's lifetime.

As a result, through carrying out the "Investigation to determine whether flow medium in needed for natural fibre composites" investigation, along with this work, a method of producing a flax fibre/epoxy resin composite which satisfies the stiffness requirement while reducing the amount of consumables used (peel ply and flow medium) during manufacturing has been defined.

# Conclusion

- Removing peel ply and flow medium post cure reduces resin content in laminate, and increases achieved FVF
- Loctite and NFM/PP samples achieved a similar FVF
- Despite indications that the Loctite and NFM/PP samples achieved a similar flexural modulus, further work is required to quantify this
- This investigation, along with previous work, has defined a method of producing a flax fibre/epoxy resin composites which satisfiy the stiffness requirement while reducing the consumables used (peel ply and flow medium) during manufacturing

Manufacturing Details - Flax/Epoxy Flow Medium 3		
Composite Type	Flax fibre/ IN2 Epoxy infusion resin	
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 77.5g, actual weight 240gsm	
Resin Used	Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)	
Manufacturing Method	RIFT as per manufacturing appendix, with additional "Loctite" release agent	
Cure Details	24 hour ambient cure, followed by 24 hours at 60 in oven	
Vacuum Details	vacuum achieved -> 12mbar leak rate -> 0.1mbar/min	
Lab Conditions	temperature 24°c, 68% humidity 23/08/22	
Laminate Details	8-ply, 77.5g fabric used	
Additional Details	correct fabric/resin/catalyst flow medium used	

Manufacturing Details - Flax/Epoxy Flow Medium 4		
Composite Type	Flax fibre/ IN2 Epoxy infusion resin	
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 77.5g, actual weight 240gsm	
Resin Used	Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)	
Manufacturing Method	RIFT as per manufacturing appendix, with additional "Loctite" release agent	
Cure Details	24 hour ambient cure, followed by 24 hours at 60 in oven	
Vacuum Details	vacuum achieved -> 28mbar leak rate -> 0.1mbar/min	
Lab Conditions	temperature 24°c, 68% humidity 23/08/22	
Laminate Details	8-ply, 77.5g fabric used	
Additional Details	correct fabric/resin/catalyst flow medium used	

Manufacturing Details - Flax/Epoxy with GF Peel Ply		
Composite Type	Flax fibre/ IN2 Epoxy infusion resin	
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 77.5g, actual weight 240gsm	
Resin Used	Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)	
Manufacturing Method	RIFT as per manufacturing appendix, with glass fibre peel ply	
Cure Details	24 hour ambient cure, followed by 8 hour cure at 60 in oven	
Vacuum Details	vacuum achieved -> 8.8mbar leak rate -> 0.2mbar/min	
Lab Conditions	temperature 23°c, 49% humidity 25/08/22	
Laminate Details	8-ply, 77.5g fabric used	
Additional Details	correct fabric/resin/catalyst flow medium used	

Manufacturing Details - Flax/Epoxy NFM/PP		
Composite Type	Flax fibre/ IN2 Epoxy infusion resin	
Fabric used	2x2 twill weave "200gsm" flax fabric 8-ply, fabric mass 76.5g, actual areal weight 240gsm	
Resin Used	Easy Composites IN2 Epoxy Infusion resin, AT30 Fast curing hardener (100:30 weight Ratio)	
Manufacturing Method	RIFT as per manufacturing appendix	
Cure Details	24 hour ambient cure, followed by 8 hour cure at 60 in oven	
Vacuum Details	Vacuum achieved -> 7.5 mbar leak rate -> 0.2	
Lab Conditions	temperature 22°c , 45% humidity 31/08/22	
Laminate Details	8-ply, 77.5g fabric used	
Additional Details	correct fabric/resin/catalyst no flow medium used	

# **Appendix H: Flow medium testing data (principal author Lloyd Vance)**

(SeaBioComp\_D3.5.2\_Appendix\_H\_flow\_medium\_testing\_data\_all\_LV.xlsx: University of Plymouth internal document).

The flax/composite samples have now been tested, the excel sheet with the data and resulting graphs has been attached. The samples appear to have a better volume fraction than the samples used in the previous "flow medium investigation", and a modulus similar to the stiffer samples previously tested. The Loctite samples were cut using a laser cutter instead of the diamond cutter, could this have an impact on performance as the samples hadn't been subjected to any potential damage/moisture absorption during cutting?

It is still difficult to remove the peel ply from samples when using Loctite, and the glass fibre peel ply is not able to be removed at all. However, I have found an easy composites flax tutorial video (https://www.easycomposites.co.uk/learning/flax-fibre-in-composites), and they did not use a flow medium or peel ply. As such, it seems sensible to produce plates without any peel ply and see what happens?

Additionally, easy composites offer a bio-based epoxy resin (used by Joe Searle for Maozhou's project) which is marketed to have equivalent properties of a usual epoxy resin, but is comprised of 38% bio-based material. Would this be of interest to the dome project?

[LV e-mail Tue 30/08/2022 15:05]

# Appendix I: Flax-bioepoxy test data (principal author Lloyd Vance)

SeaBioComp\_D3.5.2\_Appendix\_Spreadsheets\_Consolidated.xlsx

Sheets: Appendix I\_Results

# **Appendix J: Flax-epoxy test data comparisons (principal author Lloyd Vance)**

SeaBioComp\_D3.5.2\_Appendix\_Spreadsheets\_Consolidated.xlsx

Sheets: Appendix J\_Non-Flow Medium Data

Appendix J Flow-Medium Data

Appendix J\_Combined

Appendix J\_Word Format Tables

# **Appendix K: Flax-Elium test data comparisons (principal author Lloyd Vance)**

SeaBioComp D3.5.2 Appendix Spreadsheets Consolidated.xlsx

Sheets: Appendix K\_Flax-Elium-1

Appendix K\_Flax-Elium-2 Appendix K\_Flax-Elium-3 Appendix K Comparrisons

# **Appendix L: Composite plate manufacturing procedure for resin infusion Appendix J: Composite plate manufacturing procedure for resin infusion**

- 1. Select a glass plate and using a sharp blade remove any cured resin remaining on the surface and then wipe the plate clean with solvent.
- 2. Stick (white) tape around the edge of the plate where the tacky tape will eventually be placed.
- 3. Apply release agent to the plate and let it dry.
- 4. Record the time, temperature, pressure and relative humidity, and tows/m for the fabric.
- 5. Place the individual lamina onto the centre of the glass plate in the required stacking sequence.
- 6. Place a sheet of peel ply over the laminate
- 7. [only required for complex shapes] Place a sheet of porous release film over the peel ply.
- 8. Place the transport mesh/flow medium on top of the stack such that it is
- >10 mm inside the laminate edge on both sides and along the length of the laminate.
- 9. Cut a 800 mm length of inlet pipe and drill or cut holes at ~25 mm apart for the width of the laminate
- 10. Notch the end of the pipe which will go into the resin pot.
- 11. Wrap the (yellow) flow medium around the drilled inlet pipe.
- 12. Cut a 800 mm length of vacuum outlet pipe sufficient to connect to the resin trap and insert a small rolled piece of flow medium into the pipe end, and wrap any excess peel ply around the mesh.
- 13. Cut bagging film to size such that there is around 200 mm excess in both directions.
- 14. Stick a square border of tacky tape to the edges of the bagging film.
- 15. Remove (white protective) tape locally and adhere the corners of the bagging film to the glass plate.
- 16. Remove the (white) tape locally and stick the centres of each edge of the bagging film to the plate.
- 17. Make a tab of tacky tape around the pipes where they will exit the bag.
- 18. Complete the sealing of the bag to the glass plate.
- 19. Using a permanent marker, write the group name, laminate stacking sequence and an arrow indicating the resin flow direction onto the bag.
- 20. Record the time, temperature, relative humidity and pressure.
- 21. Attach a pressure meter to the inlet pipe.
- 22. Attach the outlet pipe to the resin trap and apply a vacuum to the bag, smoothing the bagging film away from the laminate area.
- 23. Identify any leaks in the bag and seek to achieve a vacuum level of ~20 mbar on the pressure gauge.
- 24. Isolate the vacuum (crimp the outlet pipes in a couple of places) and record the rate of pressure increase on the gauge.
- 25. Re-introduce the vacuum and continue to improve the seals of the bag until the rate of pressure drop is 1 mbar/minute or less.
- 26. Clamp the inlet pipe and remove the pressure gauge.
- 27. Calculate the quantity of resin (see below) required to fill the laminate, flow medium and feed pipe.
- 28. Mix the resin and hardener/catalyst/accelerator in the given proportions.
- 29. Fix the resin pot to a support, then insert the notched end of the pipe.
- 30. Unclamp the inlet pipe for just long enough that the resin rises to the clamp position, then reclamp.
- 31. After 30 seconds (to allow air displaced from the pipe to be evacuated from the bag), open the clamp and resin will flow into the bag.
- 32. It may be appropriate to record the progress of the flow front.
- 33. Once the flow front has reached the outlet pipe, and assuming the plate has filled, clamp the inlet pipe to stop further resin inflow.
- 34. If possible, with Unsaturated Polyester Resin reduce the vacuum level to  $\sim$ 500 mbar absolute and leave the moulding under vacuum until the resin gels.
- 35. Record the time, temperature, pressure and relative humidity
- 36. If required, postcure at the appropriate temperature for the required time in the oven.
- 37. The plate will be post-cured according to the resin manufacturer's recommendations.
- 38. Remove the laminate from the bag and transfer the data written on the bag to the plate.
- 39. The technician will cut the plate into samples for mechanical testing.

Volume of resin in laminate  $\approx (1-Vf) \times length \times thickness e.g. 0.5 \times 20 \times 20 \times 0.2 = 40 cm^3$ 

Volume of resin in flow medium (FM at 690g/m2)=  $(1-Vf) \times I \times b \times t$  e.g.  $0.9 \times 18 \times 22 \times 0.1 = 36$  cm<sup>3</sup>

Volume of resin in 100 cm length of 0.6 cm internal diameter (r = 0.3 cm) pipe =  $\pi r 2l = 28$  cm<sup>3</sup>

Total volume of laminate, resin in FM and pipe = 104 cm3, and assuming density of resin = 1.15 g/cm3,

∴ required resin = 120 g and with a little extra for bottom of feed cup,

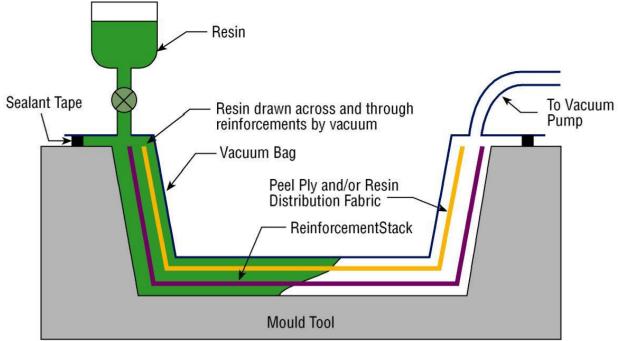
so mix ~170 g of combined (resin + hardener/catalyst)

Cite this document as: John Summerscales and Richard Cullen, Composite plate manufacture

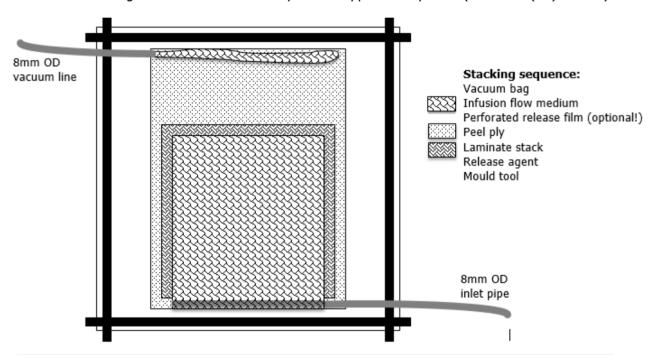
by resin infusion, University of Plymouth module MATS347 Appendix A,

https://dle.plymouth.ac.uk/pluginfile.php/1800770/mod\_folder/content/0/Appendix\_A\_RIFT.doc?forcedownload=1, accessed at <time> on <date>

Appendix G (page 2 of 2)



The above diagram is understood to be by David Cripps of SP Systems (now Gurit (UK) Limited)



# Schematic of the resin infusion process

# Appendix M: Required thickness of flax/polyester composite to achieve equivalent stiffness to rotomoulded polyethylene (principal author Lloyd Vance)

SeaBioComp\_D3.5.2\_Appendix\_Spreadsheets\_Consolidated Sheets: Appendix M Composite Design

# **Appendix N: Energy consumption monitoring equipment**

The telecommunications dome demonstrator component mould tool is heated by circulating oil. The energy consumption is monitored by a New Found Energy Limited 32-amp three phase meter with 5 pin connectors and standard kWh electricity (Albar Associates, Park View House, Worrall Street, Congleton, Cheshire CW12 1DT) Details are in Appendix O.

Ancillary equipment (e.g. vacuum pumps) energy consumption is monitored by Maxcio Vismax PM1 13 A / 3210 W power metering sockets with backlight (Ningbo Cowell Electronics Technology Company Limited, B232 Room, 11 Building, No. 22 689 Lane, Chanxing Road, Jiangbei, China imported by OPBC Limited, The Media Centre, 7 Northumberland Street, Huddersfield, West Yorkshire, HD1 1RL.

**Appendix P: Demonstrator mould tool (Composites Integration Limited)** 

(Confidential to consortium members and the sponsors)

# Appendix Q: J Summerscales, Lactide in In Situ Polymerisation (ISP) during Monomer Infusion under Flexible Tooling (MIFT), abstract submitted to ICCM23, Belfast, July-August 2023.

#### J. Summerscales\*

Materials and Structures (MAST) Research Group, School of Engineering, Computing and Mathematics (SECaM), Reynolds Building, University of Plymouth, Plymouth PL4 8AA, UK \* Corresponding author (J.Summerscales@plymouth.ac.uk)

**Keywords**: Lactide, In Situ Polymerisation (ISP), Monomer Infusion under Flexible Tooling (MIFT)

#### 1 Introduction

The InterReg 2 Seas Mers Zeeën SeaBioComp project sought to develop durable bio-based composites for use in the marine environment. The long-term ecological impact of plastic litter and microplastics in the marine environment is a growing issue that has gained considerable momentum in public perception and global media. Bio-based polymers, or polymers from renewable resources, could be a viable substitute to conventional oil-based polymers for many applications. The change might significantly reduce greenhouse gas emissions and has potential to ease end-of-life issues if the materials are biodegradable.

One of the polymers of interest is poly(lactic acid), or poly(lactide) produced from the dimer. The SeaBioComp project primarily used compression moulding or fused filament additive manufacture of the polymer to produce demonstrator components.

# 2 Large marine composite structures

For large composite structures, the process of choice would be Resin Infusion under Flexible Tooling, also known as SCRIMP, VARTM or a multitude of other abbreviations [1]. However, molten thermoplastic polymers typically have viscosities far in excess of those used for the Liquid Composite Moulding (LCM) processes. Further, the melt temperatures of many thermoplastic systems are higher than the degradation temperature of the lignocellulosic fibres used in biocomposites.

# 3 Infused thermoplastic matrix composites

Van Rijswijk and Bersee [2] reviewed *in situ* polymerisation for thermoplastics and classified the principal systems of potential use for Monomer

Infusion under Flexible Tooling (MIFT). Qing et al [3] further down-selected monomers suitable for biobased composites to be used in the marine environment. The parameters considered were (i) monomer viscosity, (ii) processing temperature, (iii) moisture absorption, (iv) mechanical properties, (v) bio-based availability, (vi) process open window, (vii) cost, and (viii) recyclability. Commercially available acrylic resin was the best fit to the above criteria, but was not available as a bio-based infusion system.

#### 4 Lactide monomer

The in situ polymerisation of lactic acid was deemed inappropriate as the condensation polymerisation would release water that would manifest as voids in the composite. The dimer of lactic acid (lactide) polymerises by ring-opening without releasing water. Lactide is supplied as a white crystalline solid with a melting range of 90-100°C.

The product data sheet for lactide says "preferably store below 35°C. On returning from Covid-19 lockdown, the open package of lactide had gone into solution (deliquescence) in the moist air in the laboratory. A recently delivered package of lactide was labelled "packed under vacuum .. content is moisture sensitive .. use immediately after opening or keep under a nitrogen atmosphere". Our technical team advised that "storing 20 kg under an inert gas is going to be a challenge"! [4].

Louisy et al [5] have reported *in situ* bulk polymerisation of l-lactide after resin transfer moulding (RTM) preparation of glass fabric composites, but results were limited to degree of polymerisation data and optical microscopy to assess composite quality. The SeaBioComp project established that flow and polymerisation of the lactide is best achieved in the temperature range 120-180°C.

1 Realisation of predicted mechanical properties In comparative tests between test samples, flax/ acrylic samples achieved 53%, while flax/PLA samples achieved 37% of properties predicted by rules-ofequation and only considering fibres aligned with the stress, flax/ acrylic samples achieved 104%, while flax/PLA samples achieved 62% of the predicted properties.

# 2 Demonstrator component

The project sought to deliver a 5G telecommunication dome as a demonstrator component. The intention was to use integrally heated infused composite tooling, but despite placing the order with a well- respected supplier, the mould tool proved to be a challenge due to a combination of complex geometry, with consequent flow paths leaving dry spots and delamination during heating cycles.

#### 3 Conclusion

MIFT for lactide remains at around Technology Readiness Level (TRL) 1. While it may be suitable for just-in-time manufacture, storage of material under dry nitrogen presents challenges. The process temperatures are challenging for integrally-heated composite tooling, so oven-cure or metal mould tools may be appropriate. The composites do not achieve predicted mechanical properties, but the experiments conducted here did not use a coupling agent on the natural fibres.

# Funding

This work was conducted within the SeaBioComp project, which has received funding from Interreg 2 Seas Mers Zeeën program 2014–2020 co-funded by the European Regional Development Fund under subsidy contract No. 2S06-006.

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# Appendix R – Appendix X: not used.

# **Appendix Y: Political context**

At the 23 June 2016 UK referendum, a small majority voted to leave the European Union (Brexit).

On 01 March 2019, the InterReg SeaBioComp project formally started.

On 31 December 2019, the outbreak of Covid-19 was first reported.

On 31 January 2020, the UK left the EU and entered a transition period.

On 24 February 2022, Russia escalated the war against Ukraine that began in 2014.

The decision for the UK to leave the EU was the result of a popular vote where the electorate were mislead by "promises" that were not honoured.

A key driver of the implementation of Brexit by UK government was to avoid the implementation of the EU Anti-Tax Avoidance Directive (2016/1164).

That driver permitted the continuation of use of tax havens within the UK economy (which pleased many Conservative party supporters!).

The Covid-19 pandemic was considered to be an unexpected disruption to life in the UK and beyond. Exercise Cygnus was a simulation exercise carried out by the UK Government in 2016 to estimate the impact of a hypothetical influenza pandemic on the United Kingdom.

The "results of three-day simulation exercise ... were 'too terrifying' to be made public" according to The Guardian.

The exercise identified four main learning points and 22 further recommendations, and showed that the pandemic would cause the country's health system to collapse from a lack of resources.

Rumour has it the costs of implementing all the recommendations would have terrified Conservative supporters!

It is not clear that full implementation of improvements was conducted before COVID-19,

A Freedom of Information request and judicial review were needed before the eventual disclosure of the Exercise Cygnus documents.

A full 30 months on from Brexit day, the UK government are claiming to have "got Brexit done", but much remains in transition, especially the Northern Ireland situation.

Many European nationals left the UK, leaving significant gaps in a number of service industries, especially for the supply chain.

Costs and timescales for EU to UK deliveries have risen significantly, with consequent delays to realisation of orders.

The conflict in Ukraine has put pressure on non-renewable energy supplies.

UK energy companies have increased prices in line with changes in supply costs (and reported high corporate profits over the same period)!

The "control" that UK sought to take back from the EU was never the problem.

The wealthy, the multi-national corporations and the neoliberal economy, who control the popular media, have been sowing dissatisfaction in the general population for years.

Keep the general population fighting one another and they will not notice who is promoting the fight to distract from their immoral and unethical practices!

... and then the Conservative Party decided Liz Truss should be Prime Minister for 44 days, and Kwasi Kwarteng was appointed Chancellor of the Exchequer for 38 days! There may now (too late for the SeaBioComp project) be some stability, but no-one in government is prepared to acknowledge that Brexit was a serious error of judgement.

# **Appendix Z: Product Data Sheets and Materials Safety Data Sheets**

Z1A: Product Data Sheet for Easy Composites IB2 infusion epoxy resin

**Z1B:** Materials Safety Data Sheet for <u>IB2 infusion epoxy resin</u>

**Z2A: Product Data Sheet for Easy Composites IN2 infusion epoxy resin** 

**Z2B:** Materials Safety Data Sheet for IN2 infusion epoxy resin

**Z3A:** Product Data Sheet for Easy Composites IP2 infusion polyester resin

**Z3B:** Materials Safety Data Sheet for <u>IP2 infusion polyester resin</u>

**Z4A:** Product Data Sheet for Arkema <u>Elium 188 XO</u> **Z4B:** Materials Safety Data Sheet for <u>Elium 188 XO</u>

ELIUM® Supplier contact information: Paul C Welter/Business Development Manager EMEA

<u>paul.welter@arkema.com</u> M: +33 6 08 68 85 58

Arkema Srl Group HQ 420 rue d'Estienne d'Orves 92700 Colombes Cedex, France. https://www.arkema.com/global/en/

**Z5A: Product Data Sheet for Total Corbion Lumilact L lactide monomer** 

**Z5B:** Materials Safety Data Sheet for <u>Lumilact L lactide monomer</u>
Appendix Z5B and Appendix Z6B are identical documents.

**Z6A:** Product Data Sheet for Corbion <u>Puralact®B L-lactide monomer</u> **Z6B:** Materials Safety Data Sheet for <u>Puralact®B L-lactide monomer</u>

**Z6C:** Packaging label for Corbion Puralact®B L-lactide monomer

Appendix Z5B and Appendix Z6B are identical documents.

Puralact®B L-lactide monomer supplier contact information.

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