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R3PACK - REDUCE, REUSE, RETHINK PACKAGING TOWARDS NOVEL FIBRE-BASED PACKAGING AND REUSE SCHEMES

Grant Agreement No. 101060806

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TABLE OF ABBREVIATIONS

AKD: Alkyl ketene dimer

B2B: Business to Business

CNC: Cellulose nanocrystals

DoE: Design of Experiment

FFS: Form-Fill-Seal

HCS: Heat conductive sealing

H-O-M: Handle-O-Meter

LCA: Life cycle assessment

MAP: Modified atmosphere packaging

MFC: Microfibrillated cellulose

OPPmet: Metallized oriented polypropylene

OTR: Oxygen transmission rate

PAE: Polyaminoamide-epichlorohydrin

PHA: Polyhydroxyalkanoate

PHBV: Poly(3-hydroxybutyrate-co-3-hydroxyvalerate)

PEF: Polyethylene furanoate

PPWR: Packaging and Packaging Waste Regulation

PVD: Physical vapor deposition

PVOH: Polyvinyl alcohol

SiOX: Silicon oxide

USS: Ultrasonic sealing

WP: Work Package

WVTR: Water vapor transmission rate

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I. EXECUTIVE SUMMARY

Building on the advancements of D4.2, this deliverable D4.5 continues to explore the development of sustainable innovative packaging solutions tailored to the diverse and complex requirements of the food industry as part of the R3PACK project. This document details the iterative and multi-route methodology process followed as part of work package 4 dedicated to substitution, focused on developing cost-effective, industrially viable, sustainable, and food-safe paper-based packaging alternatives to conventional plastic, while detailing the overall workflow and decision paths followed within the project. In fact, addressing this challenge was made even more complex by the dynamic context in which the program unfolded, marked by ongoing regulatory shifts, technological progress, and evolving market expectations, all of which have shaped and redirected the project's priorities overtime.

WP4 has therefore been divided into two main paths, "short-term" and "long-term", to simultaneously:

- Address the machinability on industrial packaging lines of more mature paper-based solutions that already approach the barrier and sealing requirements of food manufacturers, despite still containing a significant amount of plastic and petro-based materials, and still not covering all product categories (short-term);
- Maintain long range efforts in substituting plastic by functionalized paper, for flexible and rigid formats, with a range of solutions extending to the production of semi-industrial functional reels and trays, while maintaining the objective of a high paper ratio and increased bio-based content, as well as recyclability in accordance with European standards, and even compostability and/or biodegradability (long-term).

The deliverable concludes with a synthesis of the achievements to date, the main constraints and limitations associated to introducing cellulose-based food packaging on retailers' store shelves, next steps required to enable the deployment of fibre-based materials. It also provides recommendations and perspectives for overcoming the remaining barriers to large-scale market implementation.

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II. R3PACK PROJECT – WP4 CONTEXT AND METHODOLOGY

II.1. CONTEXT AND INITIAL METHODOLOGY

A) WP4 CONTEXT

The R3PACK project, funded by the European Commission under Grant Agreement 101060806, has been dedicated since 2022 to addressing the environmental challenges posed by single-use plastic packaging through innovative solutions. By focusing on reducing, reusing, and rethinking food packaging, the project aims to develop sustainable, fibre-based alternatives and implement reuse schemes that are both economically viable and environmentally sustainable.

With a consortium of 24 organizations across seven countries as illustrated in Erreur ! Source du renvoi introuvable., including stakeholders from packaging manufacturers to retailers and experts in material sciences, R3PACK integrates cross-sectoral expertise to translate research output into commercially viable and industrially scalable applications.

Figure 1: Partners of the R3PACK project

The aim of the Work Package 4 is to enable the development of industrial renewable and primarily recyclable and secondarily compostable/biodegradable paper-based food packaging solutions by using a systematic and efficient methodology for identifying, selecting, and combining advanced and promising technologies. The partners involved in this Work Package are detailed **Figure 2.**

WP4 INVOLVED PARTNERS	LOGO	PARTNER CONTRIBUTION
BIM KEMI	BIM	Surface analysis, preparation, formulation, deposition,
BIOEXTRAX	Bioextrax	PHA provider, PHA preparation
FIBERLEAN	FiberLean Technologies	MFC provider, surface preparation
FRAUNHOFER	Fraunhofer IVV	Deposition, machinability evaluation, shelf-life analysis
GASCOGNE	Gascogne	Paper provider, Deposition, cost optimisation
GUILLIN/THIOLAT	GUILLIN EMBALLAGES THIOLAT BY GUILLIN	Molded fiber and cardboard provider, deposition, cost optimization
INNOVHUB	INNOVHUB STAZIONI SPERIMENTALI PER L'INDUSTRIA innovazione e ricerca	Recyclability and compostability evaluation
POLIMI	POLITECNICO MILANO 1863	Financial modeling, cost optimisation
RISE	RI. SE	Surface analysis and preparation, formulation, deposition, evaluation
(RE)SET	(RE)SET	Work Package management

Figure 2 : Partners involved in WP4 and their contribution

This deliverable D4.5 highlights progress made in developing cutting-edge solutions such as microfibrillated cellulose (MFC) coatings, polyhydroxyalkanoates (PHA) processing, starch-based formulations, and advanced coating deposition techniques. These innovations are crucial for enhancing the performance of cellulose-based packaging, particularly in terms of barrier and sealing performance, while ensuring scalability, cost-efficiency, and minimal environmental impact, thereby meeting the stringent requirements of the food industry. Moreover, a key emphasis of this work has been placed on enhancing barrier and sealing properties to avoid food waste resulting from insufficiently guaranteed product shelf life.

This work has been conducted across a broad variety of food products, each of which requires specific machinability parameters and barrier properties as shown in **Figure 3**, with extensive involvement from industrial partners to support the transition towards practical implementation. It must be mentioned that some of the technical industrial trials carried out in the Substitution pillar involved products provided voluntarily by manufacturers that were not beneficiaries of the R3PACK consortium. These manufacturers joined the experimentation through the two beneficiary retailers, Carrefour and Système U, to expand the range of categories assessed—particularly for sweet biscuits, savoury snacks and pastry dough. Their participation did not involve any financial contribution from the project, and all packaging

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development work on their side was carried out independently.

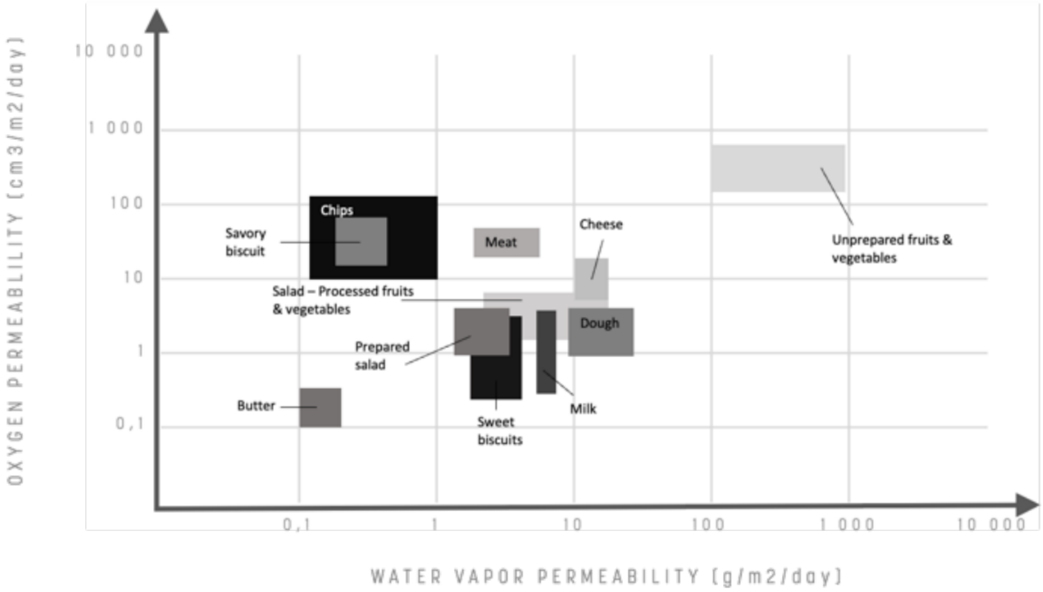

Figure 3 : Graph with logarithmic scales representing the food products included in the programme according to their targeted barriers to oxygen (OTR at 23 °C, 50 % RH) and water vapor (WVTR at 38 °C, 90 % RH).

Through this work, R3PACK paved the way for transformative change, fostering innovation in packaging materials while contributing to a more sustainable future for the food industry.

B) WP4 INITIAL METHODOLOGY

The methodology initially implemented for the WP4 R3PACK program is a multi-step and iterative process focused on developing cost-effective, industrially viable, sustainable, and food-safe paper-based packaging alternatives to conventional plastic. The key steps are presented below and are also summarized in **Figure 4.**

1. **Substrate Selection**: Paper, paperboard, and 3D-molded cellulosic fibre were identified as suitable substrates for the development of both flexible and rigid packaging solutions.
2. **Development of Multi-Layer Barrier Solutions**: Bio-based coating formulations (such as PHA, MFC, and other renewable materials) were selected, tested, and optimized for deposition on cellulosic substrates using various techniques (e.g., extrusion, lamination, spray-coating, vacuum deposition, etc.).
3. **End-of-Life Compliance**: Materials and processes will be evaluated through standardized recycling and composting tests to ensure compliance with end-of-life sustainability requirements.
4. **Shelf-Life Modelling**: Mathematical modelling, conducted in collaboration with Fraunhofer IVV, will be used to estimate the barrier properties needed to ensure food product shelf-life. Results are reported in **D4.3 *"Final Assessment Report on Shelf Life."***, and are therefore not further detailed in this deliverable.
5. **Industrial Line Testing**: Reels and trays will be produced and tested on industrial lines to assess scalability, including barrier performance, machinability, and end-of-life behaviour, supported by field trials.
6. **Sustainability Modelling**: A Life Cycle Assessment (LCA) will guide the selection of the most sustainable solutions, with findings to be presented in **D6.3 *"Comparative Analysis: LCA of SUBSTITUTION Packagings."***

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7. **Field Demonstration**: Demonstrations will be carried out in-shops to evaluate performance under real-world conditions.
8. **Economic Modelling**: A cost analysis will be conducted and detailed in ***D7.2 "R3PACK Business Plan."***

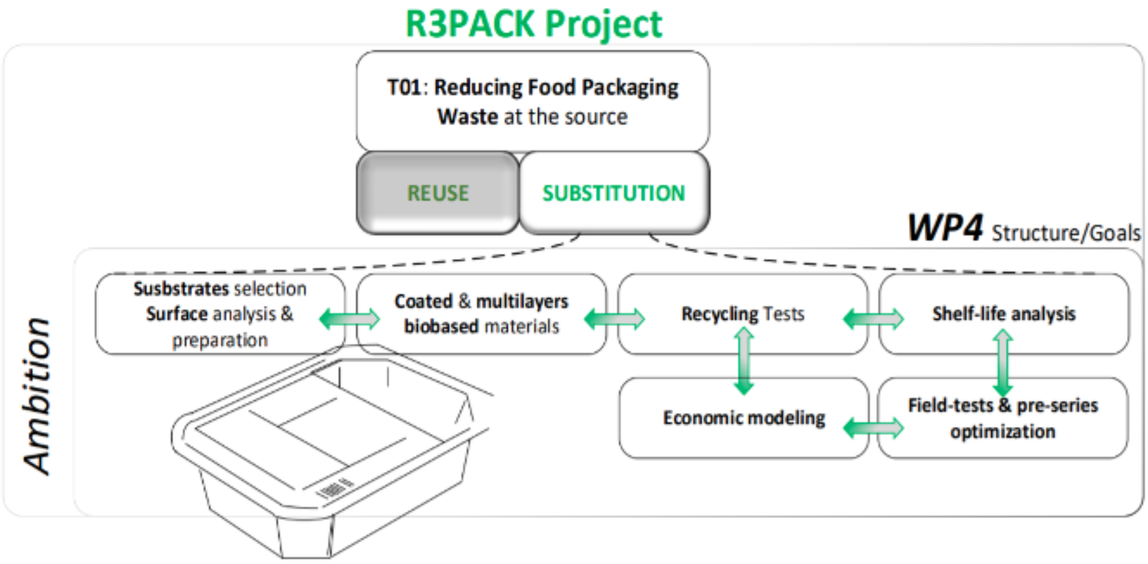

Figure 4 : Summarized workflow of the Work Package 4 (WP4)

To properly carry out this substitution work package, several obstacles, detailed later in the report, have been addressed and overcome:

- **The technical feasibility** of the production of a multi-layer barrier paper-based solution, including the availability and maturity of materials, their barrier properties, recyclability, and compliance with food contact regulations.
- **The industrial feasibility** concerning the transfer from the laboratory to packaging lines, ensuring that the developed materials can be processed on existing industrial equipment, without significant modifications and without compromising the benefits demonstrated at laboratory/pilot scale. This includes compatibility with forming, sealing, cutting, and filling processes, as well as maintaining production speed and efficiency.
- **The economic viability,** ensuring that the final packaging remains cost-competitive compared to conventional plastic-based packaging systems. This includes raw material costs, processing expenses and any potential investments required for industrial equipment adaptation while also considering consumers' willingness to accept potential price increases.
- **The environmental performance,** particularly improving the end-of-life outcomes and recyclability of the packaging. Even though paper is a renewable and biodegradable material, extra layers such as polymers, coatings, or thin metals are often added to protect the quality of food products. These layers improve performance, but they can reduce the quality of recycled fibres and so the whole recyclability of the packaging, as well as increase its total environmental impact. Accordingly, the priority is to design a multi-layer structure that provides the required technical performance while remaining recyclable, with the full life cycle of packaging always considered.
- The solution must align with current and emerging EU and national **regulations** related to sustainable packaging waste practices.
- **Consumers acceptance,** ensuring that the new paper-based packaging solutions are well-received by consumers, in terms of price, functionality and appearance,

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including attributes such as transparency, which is essential for assessing food freshness of the packaged product.

Addressing these challenges has been further complicated by the dynamic context in which the program unfolds, marked by ongoing regulatory shifts, technological progress, and evolving market expectations, all of which have significantly influenced the project's direction and priorities over time.

II.2. EVOLUTION OF THE CONTEXT THROUGHOUT THE PROGRAM

The first year of the project followed a highly iterative methodology, as detailed below, necessitated by the innovative nature of the materials and technologies[2,4] involved. A **first workshop enabled the prioritization of 6 innovative materials** (MFC, silica, PHA[3], chitosan, lignin, natural wax) **out of 11** (MFC, CNC, silica, PHA, graphene, chitosan, PEF, lignin, montmorillonite, keratin, natural wax) supported the definition of initial layer combinations to achieve functional requirements. Some materials were used to provide barrier and/or mechanical reinforcement to the cellulosic substrates as an additive in cellulose pulp, or as a surface treatment (coating, vacuum deposition, lamination etc.), creating a large number of possible combinations to investigate. This iterative approach yielded valuable insights into the advantages and constraints of each material, their compatibility and their potential synergies when combined in multiple layers, while also confirming the most appropriate deposition methods for the material and the cellulosic substrate. These insights required ongoing adaptations, with multiple iterative loops integrated into the development process to refine performance. All this work resulted in a first deliverable ***D4.2 : "Decision-matrix based on materials, barrier combination and application performance evaluation"*** published in September 2023, which is complemented by additional work in this new public deliverable D4.5.

It is important to mention that during the R3PACK project, the publication of the new European **Packaging and Packaging Waste Regulation**[1] **(PPWR)** had a significant impact on the project's direction and priorities. This external factor introduced stricter rules concerning reusability, recyclability and minimum paper content necessary to ensure commercialization of the developed solution. As a result, it reinforced the urgency to develop solutions that are not only technically, economically commercially viable but also compliant with legal requirements regarding resource origin of the material (biobased, recycled), as well as the end-of-life solutions (recyclability, composting and/or biodegradation in specific environments). This also required a revision of the initial material-selection criteria, which delayed the project and prevented certain targets from being achieved.

Taking this context into account, continuous risk-mitigation efforts were carried out by (RE)SET throughout the project to ensure material availability and reorient development choices based on the results obtained and external factors, while maintaining the in-store demonstration of paper-based packaging solutions as a final objective. Considering the required development timelines, this work package WP4 has undergone a major change in methodology with the introduction of a new path, referred to as **"short-term" path,** aimed at anticipating the demonstration phase and responding to the urgent need to bring cellulose-based substrate packaging to the market. This new strategy aimed to accelerate the development of more mature paper-based packaging solutions:

- available in sufficient quantities.
- meeting the specifications in terms of barrier properties, shelf life and airtightness,

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- suitable for food contact and currently recyclable
- sourced by (RE)SET as part of the project
- that can be tested quickly on industrial packaging lines.

WP4 has therefore been divided into two main paths, "short-term" and "long-term", to simultaneously:

- **Address the machinability of commercially available paper-based solutions with respect to recyclability, barrier properties and processability (short-term);**
- **Maintain efforts to substitute plastic by paper functionalized with biobased materials by targeting a recyclable and potentially compostable and/or biodegradable end-of-life in natural environments (long-term).**

These two approaches are detailed below, including the methodology used and the obstacles and limitations observed for each one.

III. TWO PATHS FOR SUBSTITUTION: LONG-TERM & SHORT-TERM

III.1. LONG-TERM PATH

As mentioned below, the long-term development of the solutions was divided into several phases during the 3 years of work, by moving progressively from the lab-scale (A4 sheets, non-optimized trays) to pilot trials (reels, semi-industrial trays) as shown in **Figure 5**.

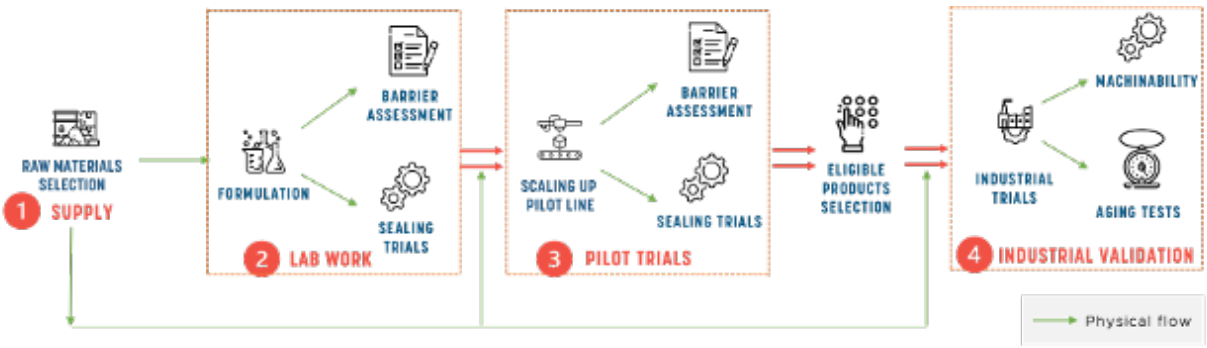

Figure 5 : Long-term path development strategy for WP4 substitution in R3PACK project.

The paper-based packaging solutions developed by the scientific experts involved in this project are the result of successive iterations, decisions and strategic arbitrations with the aim of developing food packaging solutions that meet the specifications of industrial stakeholders, i.e. ensuring effective preservation of the food product and maintaining packaging robustness throughout its use, while maximising biobased content and paper ratio as well as ensuring in accordance with PPWR regulation, and compostability.

Many parameters come into play when developing a recyclable, 90 % paper-based highly biobased food packaging solution:

- **The choice of materials**: availability of raw materials, resource used, biobased content, food contact approval, barrier properties, ease of processing.
- **The choice of the paper-based substrate:** grammage (flexible or semi-rigid paper), rigidity/deformability and format (reels versus tray).

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- **The multi-layer combination**: defining order of layers, compatibility of the materials and industrial feasibility to provide the required functionalities (barriers, sealing etc.) to the cellulosic substrate.
- **The choice of deposition technologies**: compatibility with the selected materials and substrate, equipment availability, and scalability potential.
- **Machinability on packaging lines**: sealing (temperature, pressure, time), friction, bending, mechanical strength, machine speed.

A) SELECTION OF MATERIALS

As described above, six different innovative materials were selected and tested, to which other, more industrially mature materials were added in order to develop realistic and scalable solutions. In total, more than 11 materials were tested for functionalising paper/cellulosic pulp, with a focus on natural and/or bio-based characteristics, while also considering end-of-life performance (recycling, composting), food safety and non-toxicity:

- **Biobased or petrobased dispersion coatings from BIM KEMI[2]**
- **Carnauba wax**
- **Cellulose nanocrystals (CNC)**
- **Chitosan**
- **Lignin**
- **Microfibrillated cellulose (MFC) from Fiberlean**
- **Polyhydroxyalkanoates (PHAs)[3], especially the poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) from different suppliers (Bioextrax, TianAn-ENMAT, etc.)**
- **Polyvinyl alcohol (PVOH)**
- **Silica/Silicon oxide (SiOx) from Fraunhofer**
- **Starch-based primer from BIM KEMI[4]**

It should be noted that considerable work was carried out on formulation, and various application techniques were investigated, such as lamination, dispersion coating and vacuum vapor phase deposition. This iterative work, involving than 100 multi-layer structures, enabled the clear identification of the properties provided by each material (barriers, sealing), as well as the selection of those that offer advantages in terms of processability, material availability, compatibility and upscaling capacity. All this work is available in the previous public deliverable ***D4.2. « Decision-matrix based on materials, barrier combination and application performance evaluation »***, as mentioned above.

To address as many food products as possible, two types of packaging were targeted:

- **Flexible packaging,** corresponding in the project to paper with a grammage of 50-80 g/m^2 **(part B) below)**;
- **Rigid packaging** with the case study of trays **(see part C) page 23).**

The development process covered material selection, functionalisation technologies, and the pilot scale production to the production of functional multi-layer cellulosic packaging.

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B) FLEXIBLE PACKAGING: MAIN DECISIONS AND CONCLUSIONS

LABORATORY WORK

- Work on coating formulation, deposition on paper substrates and characterisation of barrier properties

Initial **formulation work** lasting approximately six months was carried out by BIM KEMI using carnauba wax, chitosan and PHBV with the aim of achieving enhanced performance through the combination of materials with complementary barrier properties. Many parameters adjusted through numerous iterations, including the ratio of each constituent in the formulation, cross-linking and drying conditions, and the thickness of the deposited layer.
Main results and conclusions of this initial work:

- The addition of carnauba wax impaired formulations stability and was therefore discontinued.
- PHBV or chitosan used alone in dispersion coating demonstrated promising grease results and/or water barriers and were selected for use by RISE institute for the rigid tray packaging work.
- For flexible packaging, the decision was made to use more mature dispersion-coating products, at a semi-industrial level of development, either from BIM KEMI or external suppliers (inorganic pigment primer, starch-based primer, partially biobased heat-seals).

Based on these preliminary results and the refined material selection, BIM KEMI, Gascogne and Fraunhofer IVV conducted approximately twelve months of laboratory testing **of multi-layer combinations using these barrier coatings**. Performance was evaluated using standard tests: OTR, WVTR, KIT and Cobb1800, and sealing tests.
Main conclusions:

- Commercial PHA-based coating showed promising grease barrier properties and hydrophobicity, but insufficient water vapor barrier performance, i.e. WVTR (38 °C, 90 % RH) = 150 g/(m^2.d) against 50 g/(m^2.d) for a BIM KEMI's partially biobased heat-seal topcoat. Moreover, this PHA-based coating displays a narrow sealability window complicating packaging-line processing.
- Applying a primer or a heat-seal topcoat in a double-coating when possible (i.e. 2x5 g/m^2 instead of 1x10 g/m^2) appears to significantly improve barrier properties and reduced pinhole formation, areas prone to gas or liquid leakage.

To boost barriers, the use of MFCs and SiOx materials was also investigated and implemented into multi-layer structures. The addition of MFCs is supposed to improve the barriers to grease and oxygen, while the application of a very thin transparent inorganic SiOx layer by vacuum deposition is known to boost gas barriers (water vapor and oxygen).

Main conclusions on MFC material:
To assess the impact of MFCs on the final barrier properties, identical multi-layer structures consisting of a primer and a topcoat of BIM KEMI were tested on three different paper substrates, including one substrate pre-coated with MFCs. Results showed that the presence of MFCs resulted in excellent oxygen barrier performance with an OTR (23 °C, 50 % RH) = 2 cc/m^2/d, compared to 30 or even > 300 for the other substrates (a pre-coated paper and a standard kraft paper).

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- The MFCs precoating needs to be perfectly protected from water to provide an effective barrier action.
  Their presence has resulted in higher heat-seal strength of the final structure, highlighting good interlayer adhesion.

Main conclusions on SiOx material:
Applying Siox to MFC pre-coated paper without a primer coating did not improve the barrier properties due to the roughness of the surface. Moreover, improvement strongly depended on the primer previously applied on the paper substrate (one of the primers tested showed no improvement), as well as the paper substrate itself. Considering these learnings, vacuum deposition by gas phase (PVD process) of SiOx confirmed its ability to boost gas barriers (oxygen, water vapor):

- WVTR (38 °C, 90 % RH) reduced by a factor of 2.7 to 3.7, reaching values between 50 and 70 g/(m^2.d) ;
- OTR (23 °C, 50 % RH) reduced by a factor of 3 or even 100, reaching values between 0.7 and 2.0 cc/(m^2.d) ;

The food products involved in the R3PACK project and targeted for substitution were plotted on a graph (see **Figure 6**) according to their oxygen (23 °C, 50 % RH) and water vapor (38 °C, 90 % RH) barrier needs. Food products requiring flexible packaging, such as cheese and grated carrots, could be considered for the pilot reels produced from these multi-layer solutions developed in the laboratory phase. Pilot production is expected to yield more uniform coating application, enabling further performance improvements and the potential to target other food products such as margarine pastry dough. However, it must be emphasized that most of these food products require very high barriers (water vapor, oxygen, grease, water, aroma) and perfect sealing, which are challenging to achieve with paper-based materials and depend heavily on optimised industrial packaging conditions.

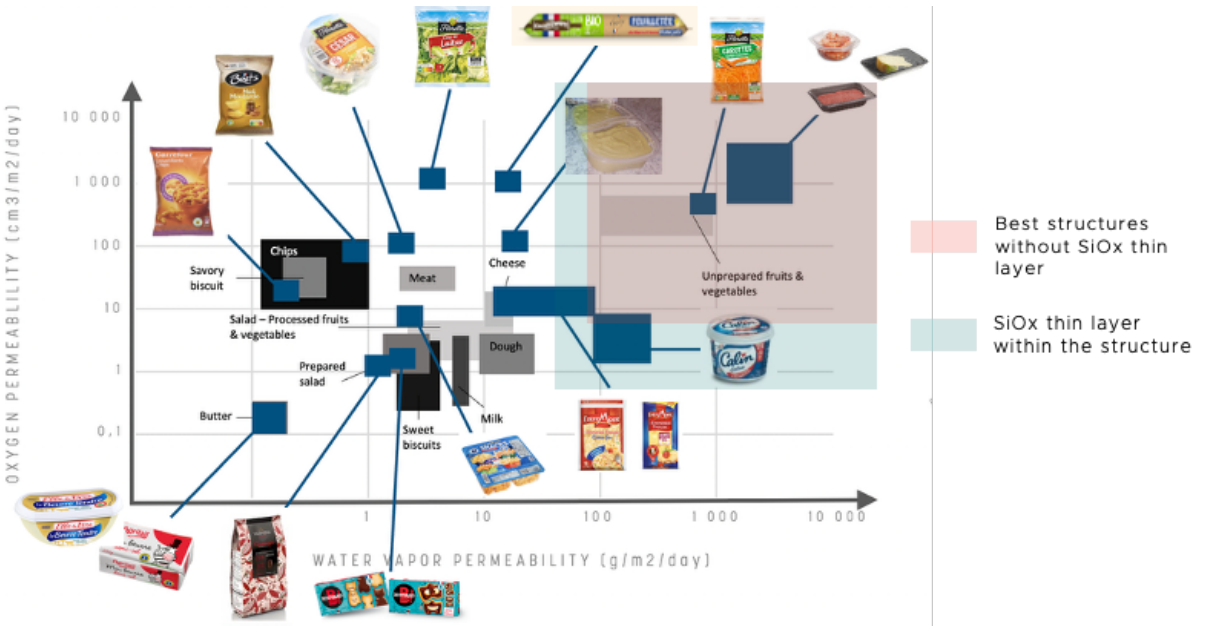

Figure 6 : Graph with logarithmic scales representing the food products targeted by the programme according to their barriers to oxygen (OTR at 23 °C, 50 % RH) and water vapor (WVTR at 38 °C, 90 % RH). The red area corresponds to multi-layer solutions developed in the laboratory phase without the use of SiOx deposition by PVD; the green area corresponds to multi-layer solutions developed in the laboratory phase including a SiOx thin layer.

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- **Sealing properties**

Barrier optimisation was carried out in parallel with regular assessments of sealing performance of the paper-based solutions with Fraunhofer, as an arbitration tool. Seam strength was systematically measured on several samples by varying temperature, while keeping pressure and time constant (as determined through a Design of Experiment (DoE)). A sealing force >3 N/15 mm and preferably >4 N/15 mm was required to validate a paper-based multi-layer structure at the laboratory scale, while ensuring compatibility with sealing conditions used on industrial equipment. Tests to verify the quality of the seal and highlight potential defects were carried out on the solutions developed, e.g. microscopy images of the triple point sealing area, the use of a dye for watertightness, etc. It should be noted that the required sealing force depends on the weight of the food product to be packaged, with 4 N/mm not being sufficient for larger volumes.

PILOT TRIALS

- **Barrier and sealing properties of produced reels**

Three different pilot reels were subsequently produced, with each iteration designed to increase the paper ratio and/or bio-based content (see **Table 1**). These pilot-scale production tests revealed issues related to the deposition of certain coatings, as well as the sealing of solutions, which in some cases resulted in watertightness issues. It was decided not to use SiOx vacuum deposition by PVD at this stage, as this deposition technology requires a highly smooth surface to obtain a uniform and high-quality Siox layer. In this specific case, a coating surface with cellulose microfibrils and/or cellulose nanocrystals induces roughness and therefore does not significantly improve gas-barrier performance.

Table 1 : Specifications of pilot reels produced by Gascogne Flexible in the R3PACK project, i.e. barrier properties, sealing force and parameters, paper ratio and biobased content.

	[THICKNESS] PAPER RATIO / BIOBASED CONTENT	HYDROPHOBICITY	WATER VAPOR BARRIER	OXYGEN BARRIER	GREASE BARRIER	SEALING FORCE
TESTS	-	COBB 1800	WVTR *(38 °C, 90 % RH)*	OTR *(23 °C, 50% RH)*	KIT	SEAM STRENGTH
UNITS	-	g/m^2	$g/(m^2.day)$	$cc/(m^2.day)$	-	N/15 mm
REEL 1	*[77 microns]* Paper **71 % /** Biobased **90 %**	< 1	190	> 200	12	7.0-7.5 [HCS: 150-180 °C] 4.0-5.0 [USS: 150-800 N]
REEL 2 (presence of MFC)	*[90 microns]* Paper **77 % /** Biobased **85 %**	< 1	30	near 0	12	*To be determined*
REEL 3 (presence of CNC and MFC)	*[93 microns]* Paper **80 % /** Biobased **90 %**	< 1	10	< 1	12	6.5-7.0 [HCS : 150-185 °C] 4.8 [USS : 800-1000 N]

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REEL 1 was produced in January 2024 as a reference (see **Figure 7**) to initially test the processability of partially biobased coatings developed by BIM KEMI on the pilot coater, in order to conserve the limited quantity of Fiberlean MFC precoated paper available. The main comments and conclusions on REEL 1 are listed below:

- Primer coating was successfully deposited without any problems with runnability and no pinholes (10 L of coating used).
- The partially biobased heat-sealing topcoat exhibited a 'sticky' behaviour onto the pilot coater equipment (fouling of cylinder).
- Significant thickness of each biobased coating layer to achieve sufficient barriers, while impacting the paper ratio, which only reaches 71% for this solution.

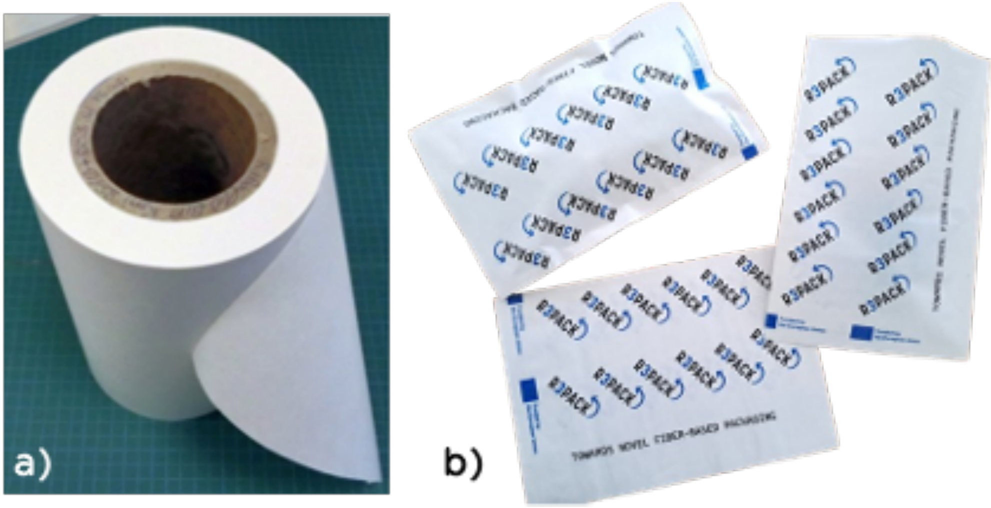

Figure 7 : a) REEL 1 picture, b) paper-based pouches manually formed from the first reel and printed with the R3PACK logos.

A decision was therefore made for future pilot reels to ensure watertightness and sealing performance of the paper-based packaging solution by prioritising a standard petroleum-based heat-sealing topcoat, while continuing efforts to improve the paper ratio and preserve the bio-based content of the remaining barrier layers.

REEL 2 was produced in March 2024 (see **Figure 8b**) using an MFC precoated reel as the paper substrate, with a water-based petroleum-derived heat-seal from BIM KEMI to ensure the packaging integrity as explained above. FiberLean MFC was applied at the wet end of the pilot paper machine using FiberLean's horizonal jet applicator technology (see **Figure 8a**). The MFC was applied immediately after the headbox onto the draining base sheet, ensuring consistent coverage. It should be noted that the MFC pre-coated paper substrate, prior to the application of barrier layers, was produced using a pilot paper machine; therefore its quality could be significantly improved with industrial production conditions. Regarding regulatory clearance, FiberLean MFC has full coverage for food contact in Europe (BfR) and USA (FDA).

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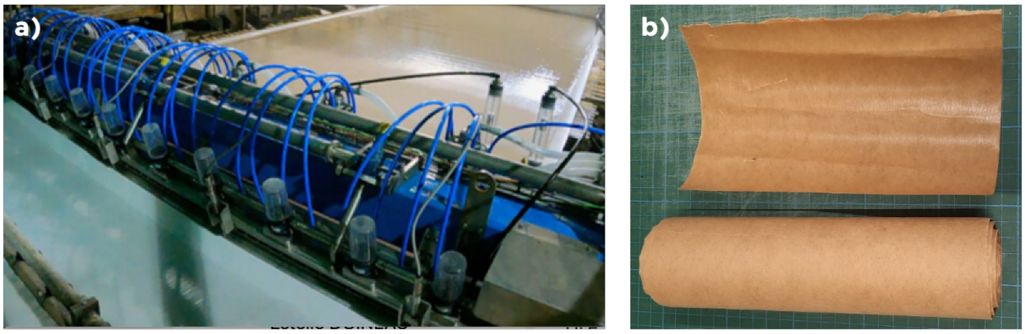

Figure 8 : a) Fiberlean's horizontal jet MFCs applicator technology, b) REEL 2 picture

The main comments and conclusions on REEL 2 are listed below:

- Gas barrier performance have been significantly improved (see **Table 1**) with a WVTR (38 °C, 90 % RH) = 30 g/(m^2.d) and an OTR (23 °C, 50 % RH) close to 0, exceeding the results obtained in the laboratory phase on A4 samples.
- Runnability of all coatings was confirmed to be successful.
- The paper ratio increased from 71 % to 77 %, owing to the use of 100 % cellulose-based MFC material and the petroleum-based heat-seal topcoat, which provides barrier performance comparable to partially bio-based coatings while requiring a thinner deposition layer.

Despite these improvements, the water vapor barrier of REEL 2 remains insufficient for most food products, requiring WVTR values below 10, or even below 1. Consequently, a third reel was produced with the aim of improving this water vapor barrier and achieving a paper ratio close to 80 % to comply with the PPWR by 2040. It was therefore decided for REEL 3 to replace the starch-based primer with a CNC-based primer, known for its excellent oxygen barrier performance.

REEL 3 was produced in January 2025 by using CNC-based material to improve gas barrier properties of the multi-layer paper-based solution, while retaining the same heat-seal petroleum-based coating from BIM KEMI as a topcoat. The main comments and conclusions on REEL 3 are listed below:

- High viscosity of the CNC-based coating, which makes it difficult to apply in a thin layer (minimum 6 g/m^2 deposited).
- 80 % paper ratio reached and 90% biobased content.
- Completion trials with PVOH material onto CNC-based layer have been carried out to achieve the desired oxygen barrier without success (poor compatibility between both coatings). Therefore, a double layer of CNC-based coating was applied instead of one.

- **Machinability**

To assess the machinability of the pilot multi-layer paper-based reels on industrial packaging machines, various tests were carried out by Fraunhofer, followed by recommendations on the settings to be used depending on the paper reel. Both heat-conductive sealing (HCS) and ultrasonic sealing (USS) have been tested.

The paper reals were analysed based on several key performance criteria:

- **Seam-strength** measurement, using heat conductive (HCS) and/or ultrasonic (USS) sealing technologies, in accordance with German standard DIN 55529, to determine the reliability of the sealed seams.
- **Friction** behaviour, evaluated on both conventional and high-speed equipment following the German standard DIN EN ISO 8295.

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- **Bending stiffness** using H-O-M method (Handle-O-Meter ASTM D 2923 – 01).
- **Tightness testing** on tubular or sealed-rim pouches with different heat-seal conditions (temperature profile, pressure, tool etc.) using vacuum decay test (Contura S400) to verify seal integrity, and rhodamine dye to detect potential leakage.

The machinability results obtained on REELS 1 and 3 are presented in **Table** 2, as REEL 2 did not undergo the full testing programme. In addition, more mature paper-based packaging solutions were also tested at Fraunhofer IVV and are presented in the "short-term" section, enabling comparison between the long-term R&D solutions with more mature solutions.

Table 2 : Specifications of pilot reels produced by Gascogne Flexible in the R3PACK project regarding machinability, i.e. sealing force, working range, friction and bending stiffness.

	PAPER RATIO / BIOBASED CONTENT	WORKING RANGE	SEALING FORCE	FRICTION	BENDING STIFFNESS
TESTS	-	HCS / USS	SEAM STRENGTH	DIN EN ISO 8295 µ (COF)	H-O-M method
UNITS	-	°C / N	N/15 mm	(v = 100 mm/min)	N / 200 m / µm
MATURE PAPER-BASED SOLUTIONS **(= reference)**	Paper > **80%** / Biobased **0%**	Non-metallized paper: HCS: **130 - 180°C** Metallized paper: HCS: **120 - 160°C** USS : no data	HCS: **5.5** ± **0,5** USS: **3.0** ± **0,0**	**0.25** ± **0,05**	(substrate vs sword) **0.08** (coating vs sword) **0.09**
REEL 1 **(biobased heat-seal)**	Paper **71%** / Biobased **90%**	HCS: **150 – 180°C** USS: **150 – 800N**	HCS: **7.2** ± **0,3** USS: **4.5** ± **0,5**	**> 1** **extremely high** (slider jumps during testing)	slight increase measured compared to commercial papers
REEL 3 **(CNC, MFC biobased coatings)**	Paper **80%** / Biobased **90%**	HCS: **150 – 185°C** USS: **800 – 1000N**	HCS: **6.8** ± **0,3** USS: **4.8** ± **0,0**	**0.6**	(substrate vs sword) **0.21** (coating vs sword) **0.25**

Here are a few comments and an analysis of the results obtained concerning the machinability of the pilot paper reels developed, with a view to running them on a packaging line:

- The biobased heat-seal topcoat used for REEL 1 showed pronounced stickiness, resulting in a very high coefficient of friction.
- The heat-seal topcoat used for REEL 3 seems proved more suitable and did not display the sticky behaviour, therefore generated a more appropriate friction behaviour than that observed in REEL 1.
- A predominantly fiber tear out was observed on REEL 3 during seam strength tests, showing a good cohesion of the multilayer structure.
- Significant thickness variations were detected in the paper substrate as it was carried out on a pilot paper machine (see **Figure** 9). However, such variations hinder uniform heat distribution within the paper structure during sealing and may therefore lead to packaging leakage issues. This limitation can nonetheless be mitigated through the production of higher quality, industrial-grade paper.
- The bending stiffness measured for REEL 3, was substantially higher, indicating that the combination of the CNC coating with the MFC pre-coating imparts additional rigidity to the substrate.

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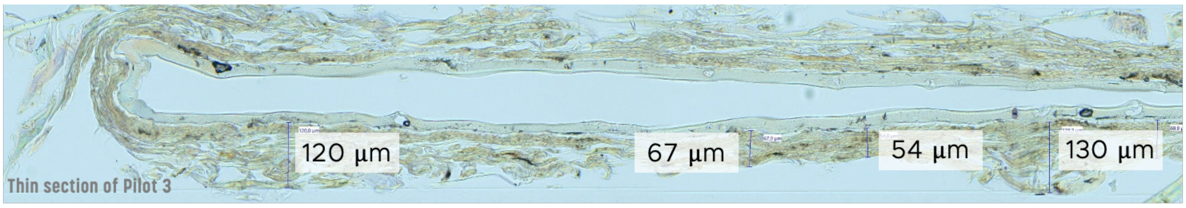

Figure 9 : Cross-sectional image of multi-layer barrier paper 3 showing non-uniform thickness of the paper substrate alone, ranging from around 50 to 130 microns.

Finally, some tightness trials were conducted on REEL 1 to better understand the behaviour of the mulit-layer structure during the sealing phase. The seam was tight, although there remained a risk of barrier damage during (severe) folding operations (tending to produce small defects close to the layer step, without fully penetrating the width of the seam). Recommendations and lessons learned from these trials are listed below to ensure proper sealing of pouches manufactured with this multi-layer paper (REEL 1):

- A sealing tool with a flexible (silicone) pad (and therefore only one-sided heating appears necessary to ensure tight sealing over layer steps, such as in tubular bags (see **Figure 10**). Note: The sealing time will be longer due to the single-sided heating.
- Depending on the type, quality and adhesion of the barrier layer, as well as the intensity of the folding, the coating may be damaged at the fold line, which can lead to leakage.
- Leakage through the seam or folded edge may be significantly greater than through the barrier coating itself. Note: Rhodamine testing (detection limit 20 µm) did not detect any barrier leakage through the coating.

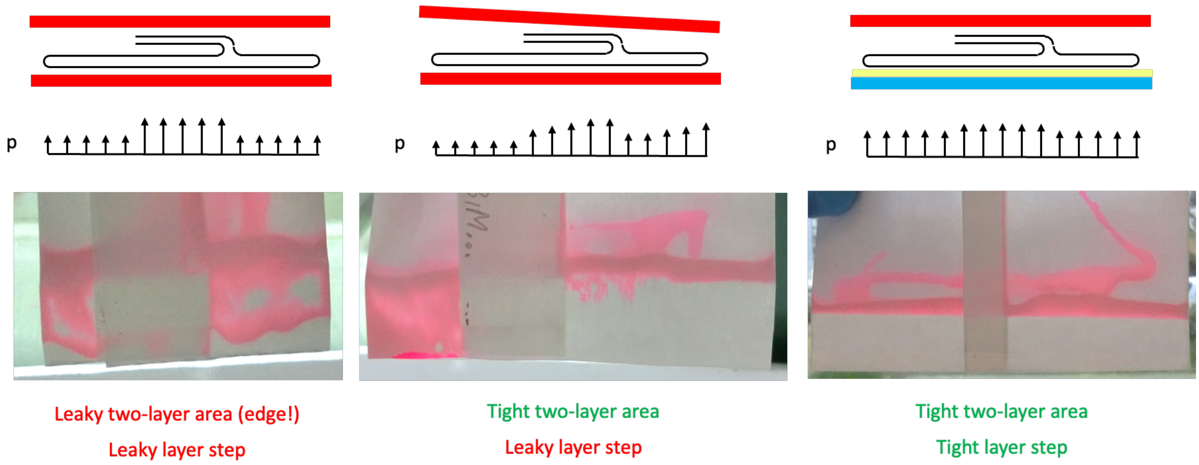

Figure 10 : Different sealing conditions (temperature profile, pressure profile, tool material) to find the adapted settings for the tested multi-layer structure (here REEL 1). The red bars correspond to flat profile heat-sealing jaws. The blue/ yellow bar corresponds to a flexible 'cold' (unheated) sealing jaw with a silicone pad.

- **End-of-life: recyclability**

Recyclability tests were carried out by Innovhub at laboratory scale, according to the European CEPI Recyclability laboratory test method, Part I – Recycling mill with Conventional process, Version 3, issued in February 2025[5]. This method enables analysing both process parameters (coarse reject, fine reject, dissolved and colloidal substances and sticky particles with a diameter smaller than 2 mm) and quality parameters (sheet formation and interfering materials like adhesiveness and visual

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impurities) of paper sheets produced from recycled fibres. This laboratory test method is composed of several stages:

- Disintegration of 10 minutes
- Filtrate analysis to obtain evaporation residue parameter
- Coarse screening (5 mm holes)
- Fine screening (0.15 mm slots)
- Visual impurities and sheet adhesion test on paper sheet produced with accept pulp after coarse screening ("AC sheets") and fine screening ("AF sheets")
- Macro stickies (optional)

Results obtained with the CEPI recyclability laboratory test method were assessed according to 4evergreen Fibre-based packaging recyclability Evaluation Protocol, Part 1, Version 1, issued in January 2025. All the results are detailed in the public deliverable **D4.4** *« Evaluation of recycling and compostability analysis ».*

The main results and conclusions are:

- REELS 1 and 2 are considered as recyclable, with a warning about the presence of a non-negligeable amount of macrostickies < 2 mm (optional in the test) that may pose a risk of paper breakage during the production of paper made with recycled cellulose fibres.
- REEL 3 behaves similarly to REELS 1 and 2 in most of the parameters tested, except that it fails the 'sheet adhesion' test due to the adhesiveness and sticky behaviour of recovered fibers.

The multi-layer paper structures developed are therefore very interesting and mostly recyclable, although the quantity of coatings applied needs to be optimised to eliminate sticky fibres and thus meet the 'sheet adhesion' criterion.

LONG-TERM PATH ON FLEXIBLE PACKAGING: PERSPECTIVES

The main challenge in the substitution of flexible packaging lies in achieving the barrier properties required by certain food products (notably water vapor and oxygen barriers, for foods such as chips or snacks), while ensuring machinability on packaging lines, and recyclability with a high paper ratio and high-quality fibre recovery.

Three high-quality pilot reels have been developed for paper-based flexible packaging applications, with the last reel (number 3) comprising 80 % paper and 90 % bio-based content, while delivering barrier properties suitable for products such as dough, cheese and grated carrots (see **Figure 11**). Furthermore, machinability trials on this REEL 3 have shown very promising results, with a sealing force of approximately 7.0 N/15 mm across a wide temperature range (150 °C-185 °C), and an appropriate coefficient of friction. More recent results from laboratory tests have shown that it is possible to achieve barrier performance levels close to more challenging food products such meat, butter-based pastry dough and even sweet biscuits, by using vacuum deposition of aluminium called "metallisation" and so considerably increasing moisture barrier (purple area on **Figure 11**).

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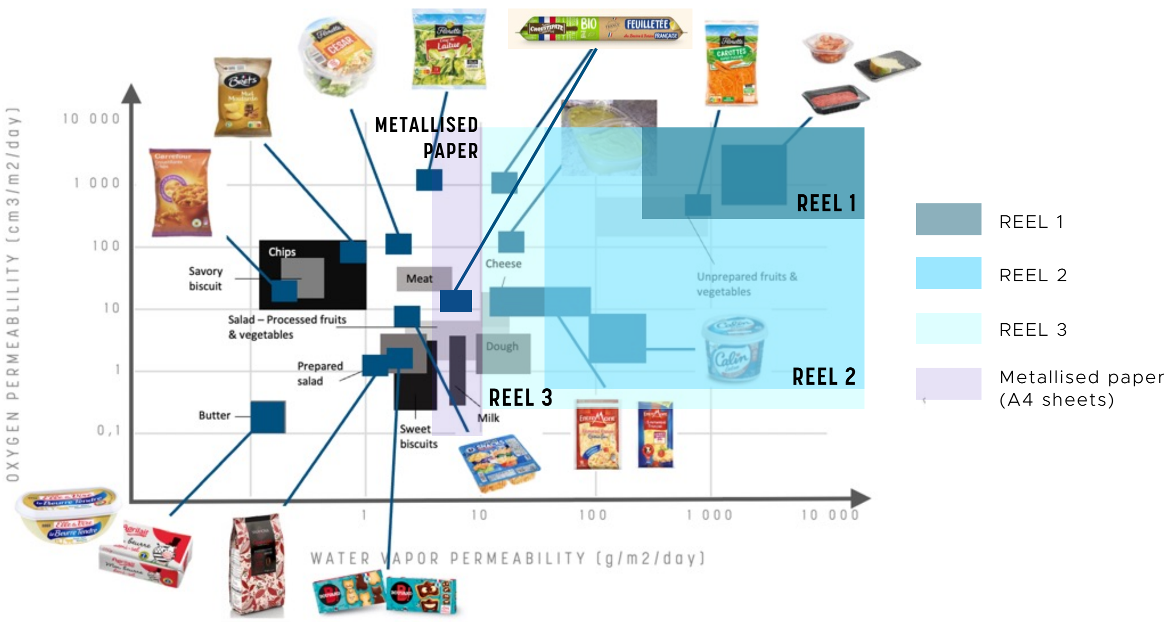

Figure 11: Graph with logarithmic scales representing the food products targeted by the programme according to their barriers to oxygen (OTR at 23 °C, 50 % RH) and water vapor (WVTR at 38 °C, 90 % RH). The blue area corresponds to multi-layer solutions developed, while the purple area coresponds to recent results with metallised papers.

Further iterations based on REEL 3 are needed to find a compromise between barrier performance, machinability and recyclability:

- **Investigate the adhesiveness observed** on paper handsheets after recyclability tests to identify the material or combination of materials responsible, enabling the optimisation or replacement of the barrier coating layer to remove the adhesiveness of recovered fibres.
- **Produce pre-coated MFC paper on an industrial paper machine by Fiberlean** (instead of pilot paper machine used for this project) to achieve a uniform thickness, facilitating smoother passage through the packaging line (reduced friction) and improved sealing quality.
- Adjust the MFC and CNC barrier layers **to reduce bending stiffness** and ease the passage through packaging lines.
- **Improving the water vapour barrier** to target products such as chocolate, biscuits, salad or even chips and snacks.

C) RIGID PACKAGING: MAIN DECISIONS AND CONCLUSIONS

LABORATORY WORK

- **Barrier properties**

Initial barrier development was caried out by RISE institute, exploring various approaches:

1) PHBV material deposition using different strategies on 2D substrates (dispersion coating, lamination or both) to achieve strong adhesion and barrier performance. As with flexible packaging development, the paper substrate was precoated with MFCs to study the impact of MFCs on the properties of the multi-layer solution. Moreover, an optimization of PHBV 5cm wide film laminates was investigated with an extruder at RISE (see **Figure 12**), adjusting PHBV grades

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(Bioextrax, TianAn), additives (clay, plasticizer) and processing conditions (temperatures, pressure etc.). These results are detailed in the previous public deliverable **D4.2 *« Decision-matrix based on materials, barrier combination and application performance evaluation ».*** The main results and conclusions are:

- The presence of MFCs prevents delamination by providing better adhesion with the barrier coating layers, and improving oxygen and grease barriers, as seen previously on flexible packaging.
- Combining MFC, PHBV coating and laminate yielded very promising adhesion between layers and barrier performance, achieving WVTR (23 °C, 50 % RH) = 4 g/(m^2.d) and OTR (23 °C, 50 % RH) = 2 cc/(m^2.d).

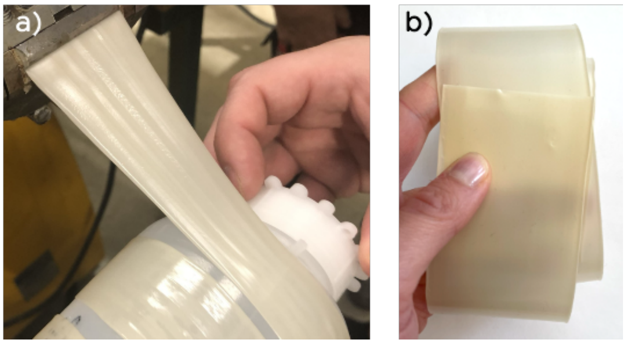

Figure 12 : a) extrusion process of a continuous PHBV-based film on the LabTech Scientific LTE20 extruder at RISE, b) different formulated PHBV-based films (around 70 microns thick) obtained by this extrusion.

2) Spray-coating on industrial trays to optimise coating formulation (stabilizer, binder etc.), select the most promising materials and multi-layer structures (see **Figure 13).** Commercially available trays (320x170x50 mm) from the partner Guillin were used to perform the spray-coating trials. The pilot spray coating technique used highlighted several technical challenges regarding layer uniformity and quantity, formulation stability and reproducibility of the test. Nonetheless, excellent water and oil/grease barriers were achieved after several iterations, with water Cobb60 = 0 g/m^2 and KIT = 12, using MFC as pre- coating and top-coatings from BIM-KEMI.

Figure 13 : Industrial trays from Guillin with different barrier layers spray coated at RISE.

- **Sealing properties (on 2D substrate)**

Seam strength tests (DIN 55529, ASTM F88/F88M-21) were carried out by Fraunhofer IVV on paper substrates functionalized by PHBV barrier layer(s), i.e. dispersed PHBV coating or laminated PHBV film 300 microns. The aim of the study was to identify the

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most appropriate grade of PHBV, and the application technology used. Three different structures were analyzed:

- Paper / MFC / PHBV dispersed coating (calendared)
- Paper / PHBV 11.5 % HV film (calendared)
- Paper / PHBV 1.5 % HV film (calendared)

A sealing force of 5.7 N/15mm was obtained with dispersion coating at 140 °C, comparable to established "short-term" paper-based solutions for the same application. However, industrial seal times are closer to 1 second or even 0.5 seconds, meaning that the 2-second time used here still needs to be reduced to achieve acceptable line speeds. The laminated papers with different grades of PHBV showed different sealing behaviour: sealing force of 15-18 N/15 mm for 1.5 % HV compared to 20-30 N/15 mm for 11.5 % HV, being higher but with significant fluctuations due to non-uniform thickness. Finally, PHBV grade with 1.5 % HV seems more temperature-stable with a processability window of 160-200 °C.

Once the barrier and sealing multi-layer structure was optimized (relevant materials selection, layer order confirmation with no delamination between layers), work on the pulp recipe was conducted using semi-pilot wet moulding equipment at RISE, presented below.

PILOT TRIALS: PRODUCTION OF MOLDED CELLULOSIC TRAYS WITH SEMI-INDUSTRIAL EQUIPMENT

The wet moulding process is composed of several stages[6], which are presented in **Figure 14**. In simple terms, cellulose pulp is formed into a tray using a mould, then hot-pressed and dried in sequential steps to remove the water from the tray.

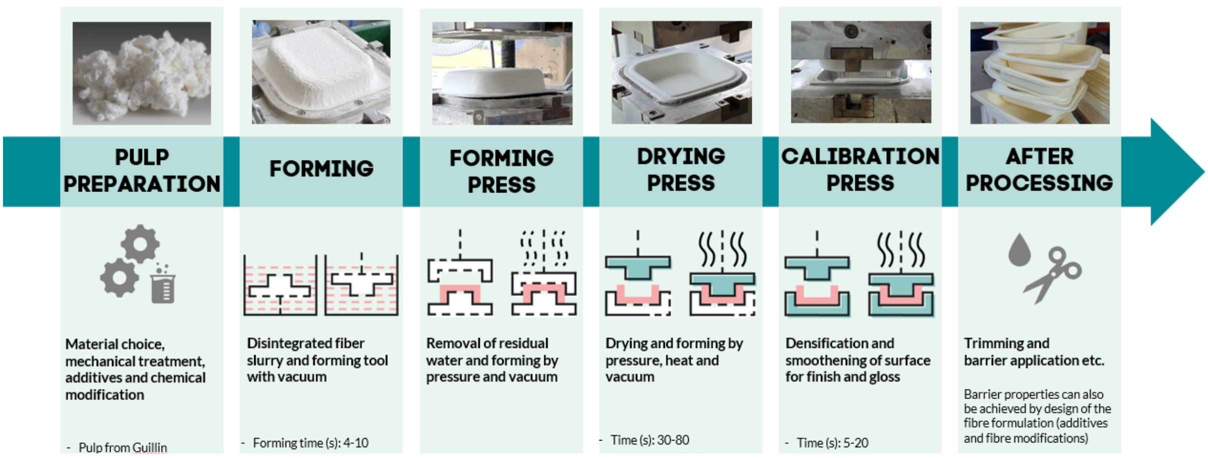

Figure 14 : The different stages of wet moulding pilot process at RISE to form cellulose trays.

- **Barrier properties**

To obtain higher quality virgin cellulose trays, i.e. with a more closed and denser surface and certain enhanced barrier properties, RISE adjusted the machine settings (time, temperature, pressure, etc.) and the pulp recipe by testing several different types and quantities of additives (biobased, wet strength, etc). **The aim here was to find the right process conditions, and a compromise between cellulose tray quality**

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and dosage level of additives, while taking recyclability considerations into account.

Here are the main conclusions of this work:

- Key impact of the size of PHBV particles in achieving a high-quality spray-coating formulation, with a preference for small particle sizes.
- Pre-coating with a cationic polyelectrolyte coating (chitosan) improved oxygen barrier performance and adhesion between layers.
- Significant differences in terms of liquid water barrier and surface roughness, enabling the selection of the best pulp recipe for spray-coating cellulose trays.
- Improved uniformity and barrier properties of the multi-layer structure with hot-pressing after PHBV spray-coating deposition (see **Figure 15** and **Figure 16**).
- Formulation of PHBV spray-coating from Bioextrax to ease the spray-coating process (binder, thickener).
- Better adhesion between layers with MFC added in the pulp recipe.

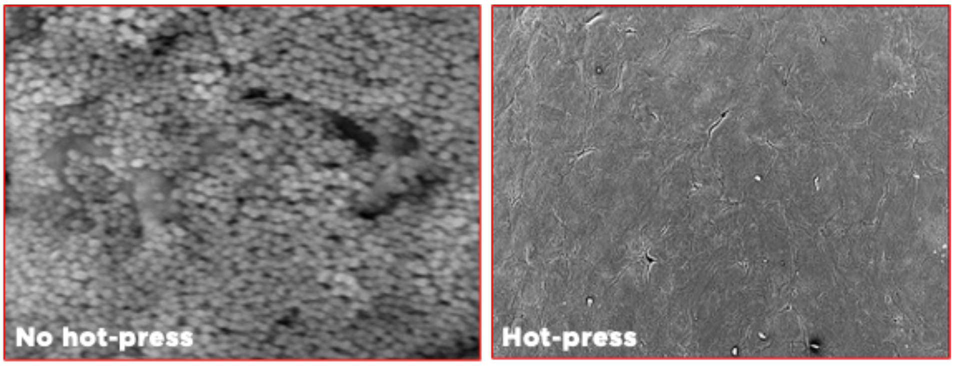

Figure 15 : SEM images of PHBV surface spray-coated onto paper substrates, followed or not by a hot-pressing step.

Figure 16 : Pictures of water droplets behaviour onto PHBV surface spray-coated paper substrates followed or not by a hot-pressing step.

All this preliminary work, both in the laboratory phase for selecting materials, determining the order of layers and spray-coating technology, and in the pilot phase for the trays' pulp recipe, has yielded remarkable and highly promising results in terms of water, oxygen and oil/grease barrier properties (see **Table 3**). Furthermore, the trays produced have a paper content of over 90 % and are entirely bio-based and theoretically home compostable.

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Table 3 : Specifications of pilot trays produced by RISE in the R3PACK project, i.e. barrier properties, cellulose ratio and biobased content.

	CELLULOSE RATIO / BIOBASED CONTENT	HYDROPHOBICITY	WATER VAPOR BARRIER	OXYGEN BARRIER	GREASE BARRIER
TESTS (Units)	-	COBB 1800 (g/m²) Contact angle (°)	WVTR (g/(m².d))	OTR (cc/(m².d))	KIT (-)
PILOT TRAY	Cellulose **90-93 % /** Biobased **100 %**	Cobb1800 = 3 g/m² Contact angle > 80 °	6 *(23 °C, 50 % RH)* 20 *(38 °C, 90 % RH)*	2 to 15 *(23 °C, 50 % RH)*	12

- **End-of-life: recyclability**

Recyclability tests were carried out by the Innovhub laboratory on barrier cellulosic trays produced at RISE, in accordance with the CEPI standard version 3 published in February 2025[5]. Please refer to the description of the entire test in the section **'Long-term path' > 'Flexible packaging' > 'Pilot trials' > 'End-of-life : recyclability'** for details of the different stages of the test and the recyclability score awarded.

A reference wet moulded tray was first tested, composed of 550-600 g/m² of pulp (same grammage for all trays) with 1 % AKD as a sizing agent, and without any barrier layer. Various functionalised cellulose trays were then tested for recyclability to identify potential impacts and recycling disruptors linked to processing or materials used in the production of these trays. Here are the details of the composition of these trays with the order of barrier layers, which will subsequently be named by their corresponding letter (Reference, A, B, C, D, E):

- **Reference tray**: Pulp + 1 % AKD
- **Tray A**: (Pulp + 1 % AKD) / 10-15 g/m² chitosan / 10-15 g/m² MFC / 40 g/m² top-coating (mainly PHBV)
- **Tray B**: (Pulp + 1 % AKD + 0.5 % PAE) / 10-15 g/m² chitosan / 10-15 g/m² MFC / 60 g/m² top-coating (mainly PHBV)
- **Tray C**: (Pulp + 1 % AKD + 10 % MFC) / 10-15 g/m² chitosan / 10-15 g/m² MFC / 50 g/m² top-coating (mainly PHBV)
- **Tray D**: (Pulp + 1% AKD + 0.5% starch) / 10-15 g/m² chitosan / 10-15 g/m² MFC / 60 g/m² top-coating (mainly PHBV)
- **Tray E**: (Pulp + 5 % AKD) / hot-pressed 25 g/m² top-coating (mainly PHBV) => produced in a previous wet-moulding batch and exhibited poor water barrier performance.

Trays A to D were produced using the same process as the reference tray and feature the same multi-layer structure consisting of chitosan, MFC and a spray-coated heat-seal top layer mainly based on PHBV. However, they differ in terms of additives incorporated into the pulp in addition to 1 % AKD, a standard additive used in commercial moulded cellulose products. Polyaminoamide-epichlorohydrin (PAE) is a permanent wet-strength agent that confers water resistance to cellulose pulp in wet conditions, while biobased additives such as MFCs or starch provide barriers against oxygen and grease/oil, as well as densifying the cellulosic material and making it slightly stiffer and less porous (highly dependent on the additive and its content). Tray E belongs to another production batch, with an AKD content of 5 %, and same top layer mainly based of PHBV, which has undergone a hot-press stage to smooth the surface.

Results obtained with the CEPI recyclability laboratory test method were assessed according to 4evergreen Fibre-based packaging recyclability Evaluation Protocol,

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Part 1, Version 1, issued in January 2025. All the results are detailed in the public deliverable **D4.4** *« Evaluation of recycling and compostability analysis ».*

The main results and conclusions are:

- Very good recyclability scores above 90/100 for trays C and D.
- Negative impact of the PAE additive at very low addition level
- Adhesiveness with recovered fibres sticky likely impacted by components composing the PHBV spray-coating formulation
- Use of MFC as a primer to reduce delamination issues, with a compromise on repulping of the fibre-based solution (stronger inter-fibre cohesion)

Tray C structure detailed in **Figure 17** is almost 100 % biobased and considered recyclable and has been selected for compostability testing presented in the following section.

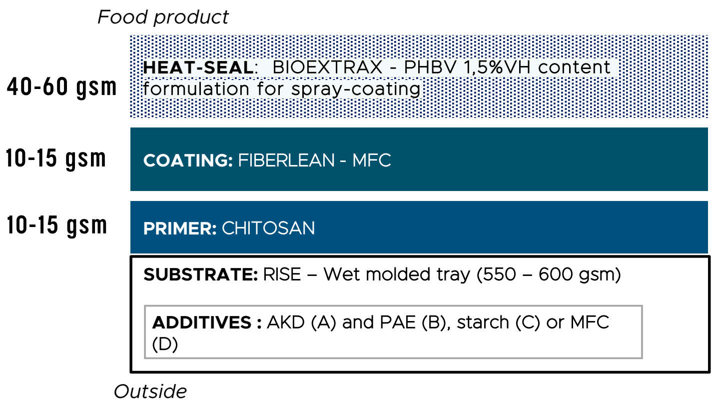

Figure 17: Multi-layer structure of barrier cellulosic tray C developed by RISE.

- **End-of-life: compostability**

The EN 13432 standard collects the requirements that a packaging must have to be considered compostable (home or industrial). In general, 4 consecutive steps are required: chemical characterization, biodegradability (ISO 14855), disintegration (ISO 16929 / ISO 20200) and ecotoxicity (OECD 208). The main purpose of the test was to evaluate the quantitative disintegration of the sample at laboratory-scale test, in a composting bin in the presence of a freshly prepared synthetic waste according to the standard. The composting process was carried out in home composting conditions (temperature approx. 25°C), and it was monitored regularly and conducted until the complete disintegration of the sample.

Plastic bins containing about 1 kg of synthetic waste were used for the composting process. In each composting bin, pieces of trays cut at about 25mm x 25mm size were included, corresponding to about 1% weight of sample respect to the biowaste. The main process parameters were monitored during the test:

- The temperature was detected every day till the end of the test.
- Periodically the composting material was mixed and visually inspected (once every two weeks) the bin was weighted and added by water to restore humidity losses due to evaporation.
- At the beginning and at the end of the test dry weight, volatile solids, pH and N tot were measured on the composting waste for each reactor.

All the results are detailed in the public deliverable **D4.4** *« Evaluation of recycling and compostability analysis ».* The test at home composting conditions was a success with a complete disintegration of the Tray C evidenced after 75 days (see **Figure 18**), when no residual sample specimens could be observed

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Test starting: biowaste and fragmented tray

After 75 days: complete disintegration with no residual specimens

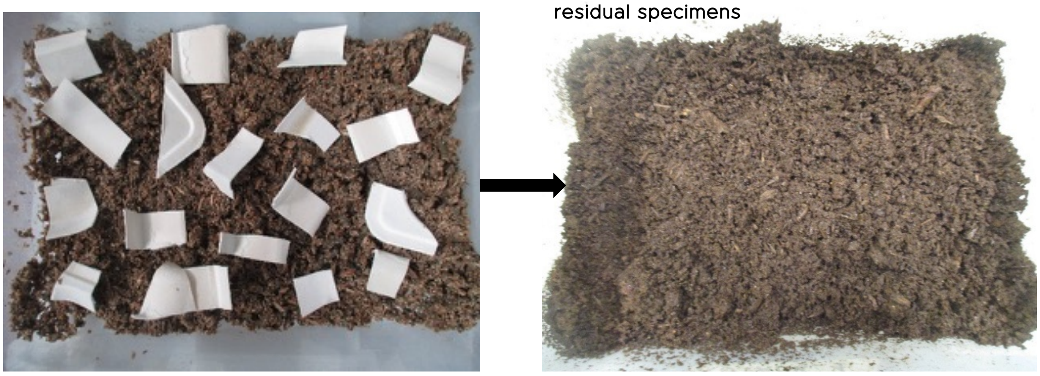

Figure 18: Pictures of the cellulosic tray packaging specimens during the test

LONG-TERM PATH ON RIGID PACKAGING: PERSPECTIVES

The main challenge in the substitution of rigid packaging lies in achieving the barrier properties on a three-dimensional moulded cellulose object using various barrier coating technologies, while ensuring that the packaging is recyclable.

Different pilot trays have been developed with tray C showing remarkable and very promising results in terms of water, oxygen and oil/grease barrier properties suitable for products such as economat goods or fruit/vegetable purée. This multi-layer barrier cellulosic tray is composed of more than 90 % cellulose and almost 100 % bio-based content, is recyclable and biodegradable in home-compost conditions.

Further iterations are still needed to improve the rigid packaging solution:

- Produce a large quantity of trays with uniform deposition of the different layers, while deliberately changing the amount of water-soluble binder in the formulation to validate the hypothesis, as well as the type of binder.
- Select lids and perform sealing and leak tests on trays.
- Reduce the number of drying steps, known to be very energy-intensive and which can impact the environmental footprint.

D) LONG-TERM PATH: LIMITATIONS TO SCALING UP THE DEVELOPMENT OF CELLULOSE BASED PACKAGING SOLUTIONS

WP4 contributors within the R3PACK project have tested in lab and pilot phases more than 10 different materials and explored more than 100 multi-layer barrier structures to functionalise paper/cellulose pulp, by focusing as much as possible on natural and/or bio-based criteria, while considering end-of-life issues (recycling, composting) and food safety.

Scaling up solutions developed in a **laboratory phase** is a lengthy process involving many iterations, setbacks and strategy changes. Listed below are several obstacles and issues highlighted during this long-term work approach:

- Wide range of targeted food products within the R3PACK project.
- Some food products presenting significant technical challenges (high gas barrier properties, modified atmosphere packaging, transparency etc.).

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- Complex intrinsic properties of cellulosic substrates that can complicate the functionalisation step: high porosity, swelling and shrinkage due to water absorption/desorption, thermal insulation, natural affinity with water and oil, etc.
- 3D packaging format, such as rigid moulded cellulose trays, requiring specific functionalisation technologies such as spray coating.
- Incompatibility between materials and/or functionalisation technologies during the preparation of the paper-based multilayer structure.
- A compromise must be found between the paper ratio and barrier properties with the publication of the European Packaging and Packaging Waste Regulation. This point is important because the PPWR regulations will require a minimum paper ratio of 70 % by 2030 and 80 % by 2040.
- in general, more natural/bio-based products needs to be applied to achieve properties equivalent to conventional petroleum-based coatings, complicating the development of recyclable and more sustainable (bio-based, compostable, etc.) paper-based packaging solutions.
- Machinability of packaging solution impacted by a very narrow processability window, a too rigid substrate, a sticky heat seal, etc.

The **pilot scale-up** was successfully completed with three reels produced for flexible packaging (including one with an 80 % paper ratio and 90 % bio-based content) and a wide variety of cellulosic trays for rigid packaging (including one with a paper ratio above 90 % and almost 100 % bio-based), see **Figure 19**.
However, the transition to pilot equipment has occasionally highlighted difficulties in processing materials:

- Some innovative products were not immediately available in large quantities.
- A coating material proved difficult to process on the coating line: too high viscosity, inhomogeneous deposited layer, sticky behaviour after drying etc.
- A narrow processability window or a need for a specific sealing profile requiring adaptations and greater precision in parameters control on the packaging line.
- High friction or excessive bending stiffness of the functionalised paper for a use on the production line: potential machine downtime, significant reduction in production rates etc.

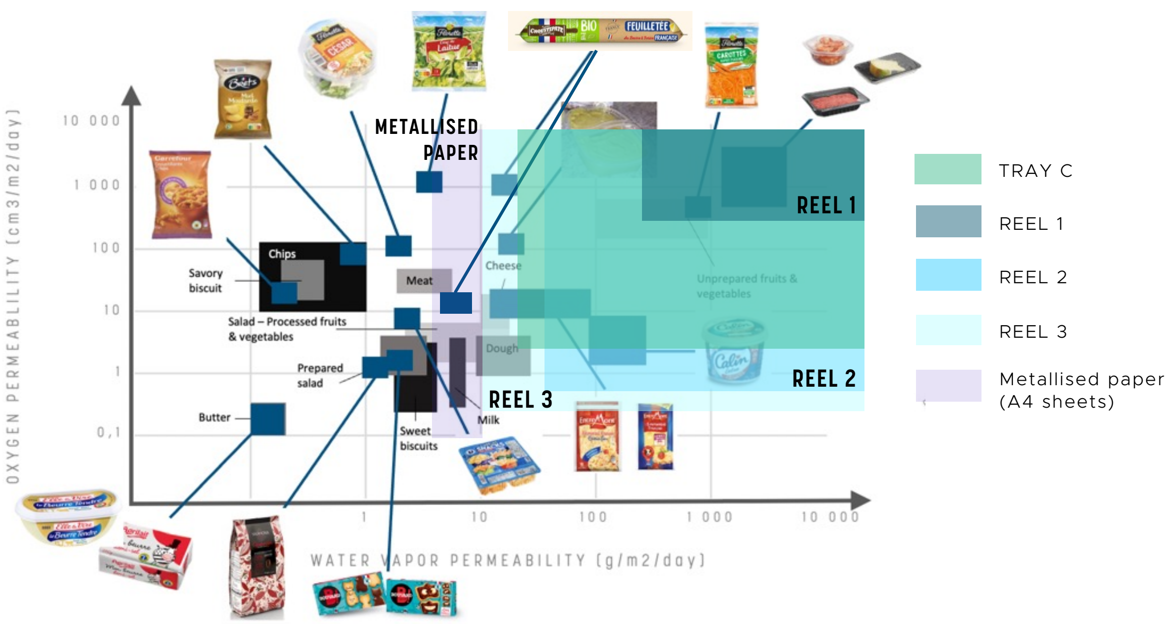

Figure 19 : Graph with logarithmic scales representing the food products targeted by the programme according to their barriers to oxygen (OTR at 23 °C, 50 % RH) and water vapor (WVTR at 38 °C, 90 % RH). The coloured area corresponds to multi-layer flexible and rigid solutions developed in the pilot phase, or under development...

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This hard R&D work in the laboratory and pilot phases has yielded very positive results and lessons learned, demonstrating the ability of bio-based materials to achieve very interesting barrier and sealing performance that can even compete with more standard petroleum-based materials. However, additional time is required to become familiar with these polymers and coatings, which are sometimes more complex and less well understood by converters, and require more precise configuration of processing equipment. The bio-based products tested in this project are not all at the same stage of industrialisation (food safety, scaling, quantity of available material) and still need to be improved in terms of the consistency of the quality of the material produced, as well as the cost, which is still too high compared to petroleum-based solutions offering identical performance (chitosan, CNC-based primers, MFC from Fiberlean, PHBV from Bioextrax, biobased coating from BIM KEMI). For the moment, validation of technical feasibility is well underway and promising, but there remains a significant challenge in terms of competitiveness and economic viability.

As explained in the beginning of this deliverable, a “short-term” path was established in this project to anticipate the demonstration phase and address the urgent need to bring cellulose-based substrate packaging to the market. This approach aims to accelerate the industrialisation of paper-based packaging solutions by sourcing more mature solutions in terms of performance and certification (recyclability, food contact) and testing them rapidly on industrial packaging lines. The results of this work are detailed in the following section.

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III.2. SHORT-TERM PATH

In parallel with the long-term development of innovative and recyclable paper-based packaging solutions, a “short-term” strategy was implemented **to accelerate time-to-market and de-risk the use of paper-based solutions on already existing industrial lines**. This approach is illustrated in **Figure 20**, which details the key stages: sourcing of relevant paper-based solutions adapted to the food product specifications, industrial validation through packaging line testing and quality control, trade negotiation between industrial and retailer, design and communication, with the ultimate goal of in-store demonstration.

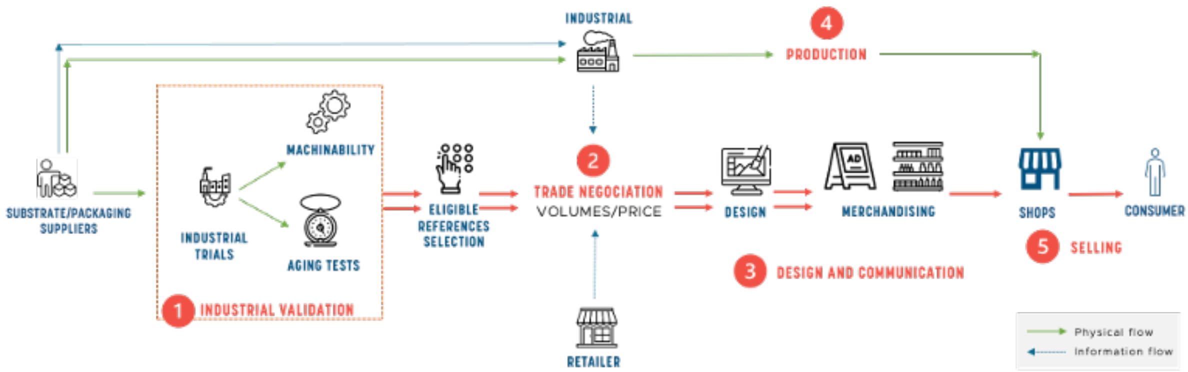

Figure 20 : Short-term path development strategy for WP4 substitution in R3PACK project

A) SOURCING OF MATURE CELLULOSIC PACKAGING SOLUTIONS

The selection of relevant mature cellulosic packaging solutions has been possible thanks to a continuous sourcing phase with a **qualification of companies (more than 2000 in 3 years)** and their solutions, as well as regular meetings between (RE)SET and the industrial partners to fully understand the constraints of their own product and the specifications needed for the packaging. Several criteria were considered such as food contact approval and recyclability, and others depending on the food product to address substrate thickness, barrier properties, transparency (salad, grated cheese), airtightness, rigidity etc.

The recyclability and paper ratio were among the main criteria, with an ambitious initial goal for paper ratio being 90 % or more. Achieving this target is particularly difficult for some food products, particularly for those packaged in flexible materials that require very high gas and/or liquid barriers, as well as specific mechanical properties. In this specific case, the multi-layer paper-based solution consists of thicker ‘plastic’ barrier layers to guarantee the shelf life and integrity of food products, but with thin paper substrate thicknesses to ensure smooth passage through the packaging line. In such cases, it is important to parallelise with a long-term research work to reduce the plastic barrier layers, to find alternative materials and functionalisation techniques for cellulosic substrates to achieve the same performance while increasing paper ratio and bio-based content. In addition, sourcing and development of paper-based packaging solutions in R3PACK project have aligned with the PPWR regulations published in December 2024, which require packaging solutions composed mainly of paper/cellulose to have a paper ratio over 70 % by 2030, and over 80 % by 2040, while keeping in mind the need to carry out standardized recyclability tests to validate the practical recyclability of the solution in paper mills as well as the desire to promote the rate of cellulose fibres recovered after recycling.

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To capitalise on the research done by (RE)SET for all the manufacturers involved in the program, the **development of a comprehensive database of innovative packaging solutions** has been a key initiative of the short-term strategy. This exclusive database, accessible only to industrial partners due to confidentiality reasons, currently contains more than 200 flexible packaging solutions and more than 30 rigid packaging solutions. These solutions encompass a broad range of materials and designs, carefully selected to meet the diverse needs of the food packaging manufacturers.

All these technical constraints made it possible to come closer to existing plastic packaging solutions, however iterative machinability trials were required on packaging lines that are mostly used for plastic solutions.

B) INDUSTRIAL VALIDATION THROUGH PACKAGING LINE TESTING AND QUALITY CONTROL: MAIN RESULTS AND RECOMMENDATIONS

Based on this sourcing and the selection of the most relevant alternative solutions, around **200 solutions** have undergone testing at lab-scale and approximately **30 have passed through the industrial packaging lines** with various food products in collaboration with manufacturers involved in this project. It is also important to note that the level of learning varied depending on the food product category. Indeed, in practice most of the industrial tests were carried out with flexible packaging, as rigid solutions often require higher investment as moulds or dedicated machines that were too high for manufacturers, who therefore did not consider developing such solutions. Moreover for some products[6], economic constraints (such as minimum order quantities) made it difficult to launch tests with a few samples sent by the solution provider.

However, for certain products, the trials provided valuable insights into technical feasibility and necessary adjustments for future implementation. This testing phase is crucial in determining the feasibility of these packaging options in real-world conditions. The evaluation process assesses essential properties such as:

- **Barrier performance:** especially resistance to oxygen and water vapor; ensuring that the packaging maintains product freshness and extends shelf life.
- **Shelf life:** by performing aging tests throughout the supposed shelf life of the product (weight loss, appearance/smell/colour/taste of the food product).
- **Machinability** of these materials on existing production lines to avoid significant process changes.

It has been proven that technical solutions in terms of barrier performance exist for substitution to paper-based packaging, as several aging tests were successfully achieved for different food categories (savoury and sweet biscuits, margarine dough) with no changes in the shelf life of the product.

However, the machinability was a major issue for most food manufacturers, so it was decided to organise a **collaborative approach with the support of Fraunhofer IVV Institute**. Working groups with manufacturers were created based on food products categories (barrier properties, grammage) and packaging typology (flexible, rigid) to align critical technical points and conditions for the pilot machinability trials to be carried out at Fraunhofer IVV to assess a pre-feasibility study. It must be mentioned that only food products packaged in flexible packaging were targeted here, with testing on the

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industrial lines being carried out primarily on flexible packaging for the reasons explained above.

This **pre-feasibility study on industrial lines**, making the bridge between long-term and short-term paths, consisted of testing several mature paper-based solutions on Fraunhofer IVV laboratory and pilot equipment to establish recommendations and best practices for the processability of these papers on industrial packaging lines. **Three different commercial paper** materials were selected to represent a broad range of product categories and applications. The tests carried out are those listed below and have been detailed previously in the section ***III.1. LONG-TERM PATH*** *> FLEXIBLE PACKAGING > Machinability:*

- Seam-strength measurement using heat conductive sealing (HCS) and/or ultrasonic (USS) sealing technologies.
- Friction behaviour using conventional and high-speed equipment.
- Bending stiffness using H-O-M method.
- Tightness on tubular or sealed-rim pouches with different heat-seal conditions (temperature profile, pressure, tool etc.) using vacuum decay test to verify seal integrity, and rhodamine dye to detect potential leakage.

The tests carried out on the three mature paper-based solutions are summarised in **Table 4** below with similar values for the whole papers, but with a distinction between metallised and non-metallised paper.

Table 4 : Specifications of mature paper-based solutions used in the pre-feasibility study conducted by Fraunhofer IVV regarding machinability, i.e. sealing force, working range, friction and bending stiffness.

	PAPER RATIO / BIOBASED CONTENT	WORKING RANGE	SEALING FORCE	FRICTION	BENDING STIFFNESS
TESTS	-	HCS	SEAM STRENGTH	DIN EN ISO 8295 µ (COF)	H-O-M method
UNITS	-	°C / N	N/15 mm	(v = 100 mm/min)	N / 200 m / µm
MATURE PAPER-BASED SOLUTIONS	Paper > **80% /** Biobased **0%**	HCS: **130-180** (non-metallized) HCS: **120-160** (metallized) USS : no data	HCS: **5.0-6.0** USS: **3.0**	**0.2-0.3**	**0.08** (substrate vs sword) **0.09** (coating vs sword)

One example of the obtained results for the sealing behaviour test with the measurement of seam strength is shown **Figure 21**. The black curve illustrates how the strength of the seal varies as temperature increases up to an optimal point. The green-shaded area represents the effective working range, where seam strength is sufficient and stable. The red cross area marks the onset of fiber burn-out and/or seal degradation (above ~180°C in this case), leading to reduced performance and potential package failure. **These data help define the safe operating window for sealing paper-based packaging to avoid any delamination or barrier performance damage.**

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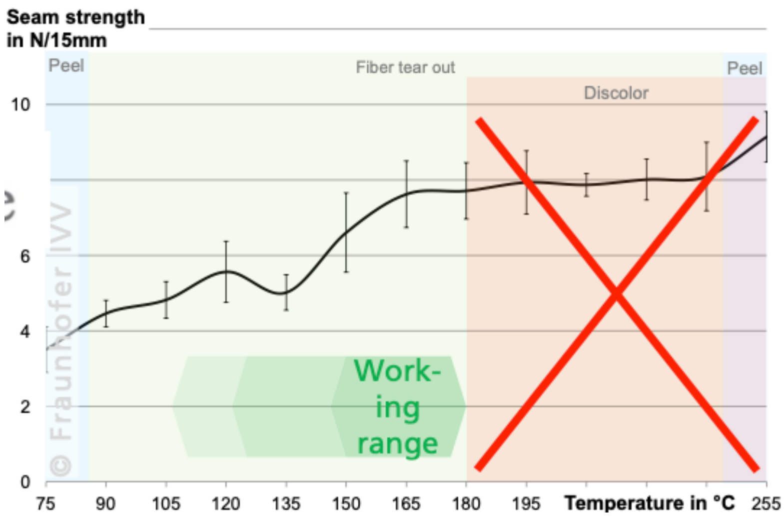

Figure 21 : Seam strength (N/15 mm) of paper-based packaging versus sealing temperature (°C)

Indeed, beyond the 180°C limit, the seal enters a degradation zone characterized by visible shrinkage (a), delamination (b), and discoloration (c) along the seam edge (**Figure 22**). The delamination observed between polyolefin laminate and the metallized layer also results in reduced mechanical strength and an increased risk of package failure. Therefore, maintaining the sealing process within the defined, green-shaded zone is essential to preserve the integrity of the packaging and the shelf life of the food product.

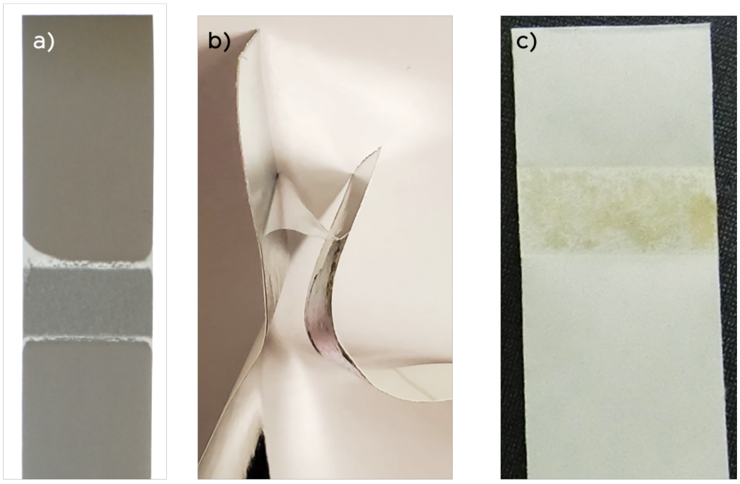

Figure 22 : Damages of barrier paper during sealing step by using temperatures above 180 °C. a) shrinkage, b) delamination, c) discoloration.

The rhodamine dye test highlighted weaknesses by changing the sealing technology and parameters as well as the format of the packaging (sealed-rim bag, or tube bag for instance), and is detailed in the section **Machinability.**

Below are the main factors that can affect the sealing phase of paper materials:

- Layering steps to produce tubular (area with 3-ply) or sealed-rim pouch (area with 2-ply), as illustrated in **Figure 23**, can have a strong impact on the tightness.
- Having structured tool surfaces will increase the tightness and the strength and decrease the barrier damage (detailed in **Figure 10** for the long-term path).

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- Being careful about the packaging elongation while internal machine transport can decrease the barrier damage.
- Varying preheating of the packaging material downtime versus steady-state machine operation.

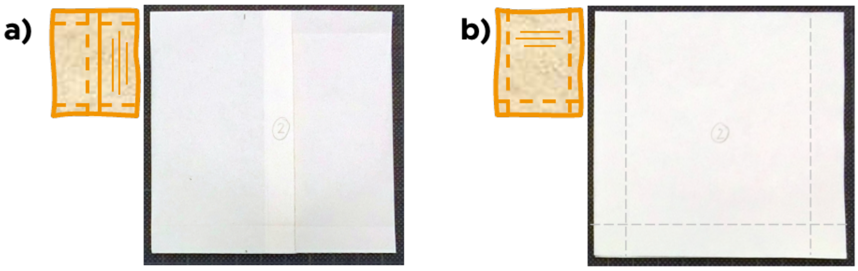

Figure 23 : a) tubular pouch and b) sealed-rim pouch prepared to verify the tightness of paper-based solutions.

In view of the factors that can significantly impact the integrity of the final packaging and the passage through the production line, here are some recommendations:

- Ensure **sufficient conditioning** of the paper-based packaging material: at least 24 hours in a constant production environment.
- **Define the safe operating window for sealing** the paper-based solution to avoid any delamination or barrier performance damage, especially if this paper contains a thin layer of metallisation.
- Have sealing equipment that allows **to control temperature, time and pressure parameters**.
- **Check the real sealing parameters:** Temperature of the sealing bar contact surfaces and the effective pressure between the sealing bars which can be different than the supply pressure at some manometers.
- **The sealing tool should be adapted** to the paper-based multi-layer structure as well as the geometry of the packaging to minimize leakage.

This pre-feasibility study conducted by Fraunhofer IVV on the machinability of mature paper-based reels has enabled industrial partners to obtain **practical recommendations** based on the type of barrier paper they use, allowing them to optimise their internal processes. Following this, the manufacturers continued to carry out individually their own tests under the defined conditions, based on their specific packaging formats and lines.

Certain partners have successfully validated the transition to industrial packaging line of paper-based solutions, and some have invested in equipment to adapt to the requirements imposed by paper:

- The installation of a specific tool called “col de cygne” directly on existing lines was necessary for chips product.
- A manufacturer also invested in sealing jaws with a specific geometry to prevent damage to the paper during the heat-sealing phase.
- An interaction between certain equipment on the packaging line and the metallisation in the barrier paper required the modules on the line to be rearranged to allow the metallised paper solution to pass through the line.

To take this short-term approach and key learnings further, proceed to deliverable ***D5.2 entitled “Comparative analysis for the scenarios from phase 1 to phase 3”***, which deals with the demonstration phase.

The technical validation of the paper solution has been approved for certain food products, but not for all. However, beyond the technical aspect, other challenges

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must be validated for the marketing of a new food packaging solution and thus be able to find it on the shelves of a retailer.

C) COMMERCIALIZATION OF PAPER-BASED PACKAGING SOLUTIONS AND IN-STORE DEMONSTRATION: LIMITATIONS AND CONSTRAINTS

This section outlines the main limitations and constraints associated with commercialization of food packaging solutions in retailers' stores, focusing on technical challenges, while also mentioning other constraints such as economic factors, consumer acceptance and regulatory.

TECHNICAL CHALLENGES

The primary challenge is to develop solutions that work across a wide range of products while considering the specific needs of each individual industrial production line. Moreover, a major constraint faced by manufacturers is the need to limit capital expenditure (CapEx) related to new machinery investments. This means that packaging solutions must be compatible with existing production lines, requiring designs that can be seamlessly integrated without significant modifications to the machinery.
To address this, packaging solutions must have adapted mechanical properties that prevent issues such as breakage or tearing during the production process. For example, ensuring the right tension in paper and film materials is essential to avoid damage while maintaining machinability.

Production speed is another factor closely tied to the tension in the film and the sealing time. Paper-based flexible materials, being more rigid and less flexible than plastic films, often lead to more frequent breakages when operating at the same speed as other materials. This can present challenges in maintaining consistent production rates and product quality, especially when working with packaging that requires higher rigidity for specific applications, such as for certain food types.
The same issue is observed for more rigid packaging where for yoghurt for instance form-filled-sealed (FFS) lines allow high production speed, but switching to pre-formed cardboard or moulded cellulose alternatives needs lines adaptation and investments.

Sealing is another critical area, requiring specialized tooling to handle wear and tear over time, as well as correct settings for seal profile, width, and the interaction between the sealing bars and the film. The parameters for sealing (time, pressure, temperature) must also be carefully controlled, particularly when working with paper-based materials, partly due to the insulating properties of paper.

An analysis of the packaging requirements for various food products, focusing on **barrier properties** and **machinability complexity** in both flexible and rigid packaging formats has been produced. The goal is to prioritize products based on their packaging challenges, to guide packaging material selection and processing improvements. The analysis is based on key parameters for each product:

⇨ **Barrier Criteria:**
- WVTR (Water Vapor Transmission Rate)
- OTR (Oxygen Transmission Rate)
- Water Cobb (water absorption)
- Oil Cobb (oil absorption)
- Modified Atmosphere Packaging (MAP) requirement
- Shelf-life demand

⇨ **Machinability Criteria:**

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- Sealability (cold, hot, presence of a cardboard box)
- Basis weight (grammage of material)
- Conditioning (ambient, fresh/humid, hot/humid)

Each criterion is scored on a scale, with higher values indicating greater complexity or stricter requirements as detailed in **Appendix 1** and **Appendix 2**. The results for flexible packaging products are detailed and presented in **Figure 24** below**.**

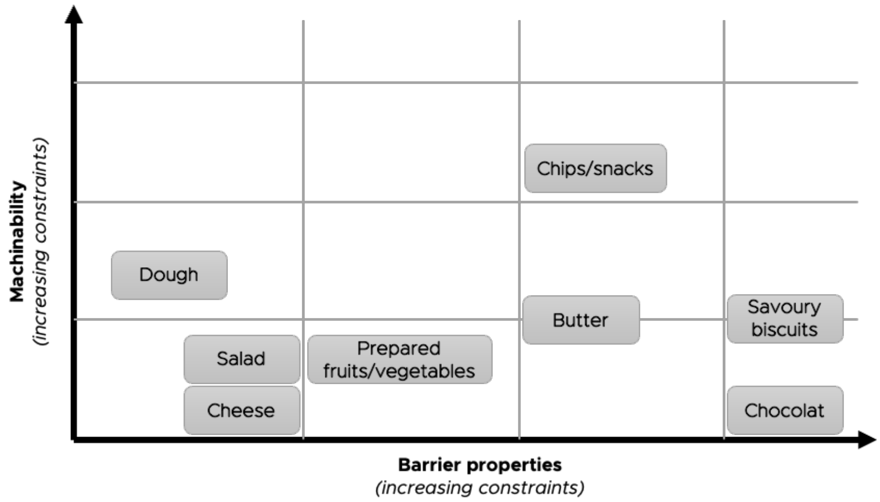

Figure 24 : Matrix of complexity for flexible packaging food products regarding machinability and barrier properties criteria

The main conclusions for flexible packaging are as follows:

- **Chips/snacks** pose the greatest machinability challenge, due to low grammage, hot/humid conditioning, and complex sealing. As shown in Figure 25, grease leakages are often observed on the sealed area due to the complexity of the product.
- **Chocolate** has the lowest machinability complexity, mainly due to cold seal requirements.
- **Products like butter and biscuits** have intermediate challenges linked to material memory and grammage.

Figure 25 : Grease leakage on the sealing area observed after conditioning chips in paper-based flexible packaging

Overall, the analysis highlights significant differences in packaging requirements across product categories, emphasizing the need for tailored material and process strategies. Prioritization matrices assist in focusing resources on the most complex products, improving overall packaging efficiency and product shelf-life.

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The same work has been conducted for rigid packaging however only barrier criteria were defined as the machinability is generally the same for such packaging.

The main conclusions for rigid packaging are as follows:

- **Snacks** have the highest packaging complexity, indicating critical barrier requirements, especially for oxygen to maintain crispiness
- **Yogurt and cream** follow closely, reflecting challenges related to moisture sensitivity
- **Prepared salads, butter and purée have moderate complexity**
- **In-shop products** present the lowest complexity among the listed items indicating fewer demanding requirements for barrier properties

However, the main complexity in rigid packaging is not merely a matter of achieving barrier properties but primarily involves addressing the economic and production issues associated with packaging.

OTHER CHALLENGES: ECONOMIC, SOCIETAL & REGULATORY

In addition to the significant technical challenges, the R3PACK project identified three main challenges in the substitution of plastic with cellulosic fibre-based packaging: economic, societal, and regulatory.

Economically, fibre-based packaging remains significantly more expensive than plastic, with unit costs two to five times higher. For example, switching from plastic to paper sachets for instance increases the cost per unit from €0.04 to €0.12, partly due to slower production speeds (–20%). This translates into a 45% decrease in distributor margins and a notable reduction in overall profitability. Although current costs are unfavourable, increasing environmental taxes on plastics (e.g. eco-contribution, PPWR obligations) will progressively improve the competitiveness of fibre-based solutions. Investments in process optimisation and material innovation are therefore essential to achieve long-term cost parity.

From a **societal** perspective, consumer acceptance is a decisive success factor. While environmental awareness is high, functional expectations (transparency[7,8], protection, usability) remain critical. Paper packaging is less accepted for highly perishable products such as fresh salad, where visibility of contents drives purchasing decisions. Conversely, for dry goods and snacks, consumer acceptance is significantly stronger.

Finally, **regulatory** uncertainty under the evolving Packaging and Packaging Waste Regulation (PPWR) represents a major barrier to industrial uptake. Unclear implementation timelines and heterogeneous national interpretations discourage investment and delay deployment. Manufacturers require harmonised, predictable EU-level rules to de-risk innovation and accelerate adoption.

In summary, addressing these intertwined economic, societal, and regulatory barriers is essential to enable the successful market transition toward sustainable, fibre-based packaging solutions across Europe. All these aspects are described in greater detail in deliverable ***D5.2 entitled "Comparative analysis for the scenarios from phase 1 to phase 3"***, which deals with the demonstration phases.

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IV. CONCLUSIONS & PERSPECTIVES

This deliverable presents the methodological framework developed within R3PACK work package 4 for designing and producing high-performance, fibre-based packaging with advanced barrier properties, suitable for large-scale industrial application. The work has established a coherent workflow integrating material formulation, process optimisation and functional characterisation to ensure performance, recyclability, sustainability and economic feasibility.

The project successfully identified optimal combinations of fibres, additives and coating systems providing mechanical strength, sealing and protection against moisture and oxygen. A major focus has been the surface functionalisation through thin-layer deposition, biobased coatings and vacuum treatments to enhance barrier properties without compromising recyclability sometimes going as far as compostability. Pilot-scale trials confirmed the technical feasibility of continuous processing (extrusion, coating and lamination) using existing industrial equipment. Functional tests demonstrated competitive barrier and mechanical performances compared with conventional plastic food packaging. Nonetheless, several technical challenges remain, notably the uniformity of barrier layers at large scale, the stability of coatings under mechanical and thermal stress, and the balance between high barrier performance and recyclability, which often conflict. The cost-performance optimisation of biobased formulations also requires further work. The project recommends adopting a multi-criteria evaluation framework combining environmental, technical and economic indicators for future developments.

To conclude, WP4 substitution pillar has delivered a robust methodological process towards the industrial implementation of fibre-based high-barrier packaging. While the technical feasibility of paper-based packaging has been demonstrated, economic viability remains a major obstacle. To move forward, it is essential to optimise the economic model surrounding paper packaging and continue developing processes specifically tailored to paper materials. Plastic remains the cheaper option today, primarily because its production processes have been refined and optimised over several decades. Bridging this gap will require continued innovation, investment, and time to unlock the full potential of paper as a viable, scalable, and sustainable alternative to plastic. It is therefore recommended to support and accompany the development of innovations focused on high-speed, efficient industrial machinery adapted to paper-based packaging. These innovations are essential to move from laboratory or pilot-scale solutions to full industrial deployment. By enabling high production rates while maintaining product quality and sustainability, such equipment will play a key role in making paper-based alternatives more economically viable and competitive compared to conventional packaging.

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APPENDIX

Appendix 1 - Table of Barrier criteria scale

Criteria	Description	Scale Explanation
WVTR	Water Vapor Transmission Rate	+ = Low permeability (less critical) ++ = Medium permeability +++ = High permeability (very critical)
OTR	Oxygen Transmission Rate	+ = Low oxygen barrier required ++ = Medium oxygen barrier +++ = High oxygen barrier required
Water Cobb	Water absorption (considering condensation effects)	+ = Low absorption, minor condensation ++ = Moderate absorption, potential condensation +++ = High absorption, product exudes moisture internally
Oil Cobb	Oil absorption	+ = Low oil barrier needed ++ = Medium oil barrier +++ = High oil barrier required
Modified Atmosphere	Requirement for controlled atmosphere packaging (MAP), including effects of sachet perforations	+ = Low MAP requirement ++ = Medium MAP complexity +++ = High MAP complexity, including perforation control
Shelf-life	Expected product shelf-life	+ = Short shelf-life (< 1 month) ++ = Medium shelf-life (~3 months) +++ = Long shelf-life (>7 months)

Product	WVTR	OTR	Water Cobb	Oil Cobb	Modified Atmosphere	Shelf-life	Product	Total
Salad	9	1	9	1	1	1	Salad	22
Chips/ snacks	9	3	1	9	1	9	Chips/ snacks	32
Chocolat	9	9	1	9	1	9	Chocolat	38
Dough	3	1	3	9	1	1	Dough	18
Cheese	3	3	3	9	1	3	Cheese	22
Butter	9	9	1	9	1	3	Butter	32
Savoury biscuits	9	9	1	9	1	9	Savoury biscuits	38
Prepared Fruits and vegetables	1	1	9	1	9	3	Prepared Fruits and vegetables	24

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Appendix 2 - Table of Machinability criteria scale

Criteria	Description	Scale Explanation
Sealability	Ability to seal packaging material under different conditions	+ = Cold sealing ++ = Conventional thermal sealing +++ = Mechanical/shape constraints on sealing zones
Grammage (Basis Weight)	Material weight per square meter (g/m²)	+ = High grammage (> 80 g/m²) ++ = Medium grammage (~60 g/m²) +++ = Low grammage (< 60 g/m²)
Conditioning	Environmental conditions during packaging	+ = Ambient conditions ++ = Fresh and humid conditions +++ = Hot and humid conditions

Product	Scellability	Basis Weight	Conditioning	Total
Salad	3	3	3	9
Chips/ snacks	3	9	9	21
Chocolat	1	1	1	3
Dough	9	3	3	15
Cheese	3	1	3	7
Butter	9	1	3	13
Savoury biscuits	3	9	1	13
Prepared Fruits and vegetables	3	3	3	9

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