



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

Grant Agreement No. 101060806

Work package: WP4 – Substitution: new fibre-based packaging solutions.

Deliverable: D4.4: Evaluation of recycling and compostability analysis.

Dissemination level: Public

Issue date: 30th November 2025

Lead beneficiary: Innovhub

DOCUMENT INFORMATION

HISTORY OF CHANGES		
Version	Publication date	Changes
1.0	26.11.2025	Initial version
2.0	08.01.2026	Second version
3.0	23.01.2026	Final version

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DOCUMENT DISTRIBUTION		
Issue	Date	Distributed to
3.0	23.01.2026	European Commission

VERIFICATION AND APPROVAL		
	Date	Distributed to
Estelle Doineau	23.01.2026	(RE)SET



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I. EXECUTIVE SUMMARY

This document explains the results and outcomes of the recyclability and composting tests carried out during the R3PACK project on the most promising multilayer fibre-based packaging solutions selected for their barrier and sealing performance.

Recyclability tests have been carried out according to the European CEPI Recyclability laboratory test method [2], which enables analysing both recycling process parameters and recycled paper quality parameters. Results have been assessed according to 4evergreen Fibre-based packaging recyclability Evaluation Protocol [3], which describes the assessment of the technical recyclability.

Regarding flexible packaging, the results of three different pilot reels with increased paper ratio and/or bio-based content are presented. First and second reels are considered technically recyclable, while the third reel has some issues regarding the recycled sheets adhesiveness, probably due to a higher fragmentation of the coating layer.

About rigid packaging, six different samples of functionalised cellulosic trays, produced with a semi-industrial equipment by the wet molding process, have been tested. Two types of trays are technically recyclable with a very good score, while sheet adhesion occurs in the other two samples.

Finally, the document presents the results of the home-compostability of one type of trays, with a positive outcome.

Results are deeply discussed together with possible improvements and next steps.

I. INTRODUCTION

The aim of the Work Package 4 is to secure the development of industrial renewable and primarily recyclable, and secondarily compostable/biodegradable fibre-based food packaging solutions by using a novel method of efficient selection and combination of innovative materials and technologies. The main objective is the development of cost-effective, industrially viable, sustainable, and food-safe paper-based packaging alternatives to conventional single-use plastic packaging. Currently there is a significant industry trend of replacing plastic packaging with paper-based alternatives, driven by consumer demand for sustainability, environmental concerns about plastic waste, and regulatory pressure. This requires a big effort for innovation in fibre-based materials with new barrier coatings, to ensure improved packaging performances along with better recyclability and a lower carbon footprint.

Recyclability of fibre-based packaging materials is an essential requirement, from the perspective of the circular economy and European policies, first and foremost the PPWR Packaging and Packaging Waste Regulation [1]. Indeed, one of the most important aspects introduced by the Regulation concerns the obligations and requirements relating to the recyclability of packaging and the promotion of high-quality recycling. This means that packaging must be designed to be recyclable and, once it becomes waste, it must be collected, sorted, and recycled to obtain high-quality secondary raw materials. By January 2030, all packaging placed on the market must be designed to be efficiently recyclable, and recyclability will be expressed through performance classifications A, B, and C. Recyclability performance will be based on design-for-recycling criteria, which must ensure the circularity of the use of secondary raw materials of sufficient quality to replace primary materials. Furthermore, from January 2035 packaging waste will have to be collected, sorted and recycled at an industrial scale, ensuring a certain annual quantity of recycled material at EU level.



Several coating solutions and functionalisation techniques were studied during the R3PACK project and recyclability tests were carried out accordingly during the development of the new fibre-based materials, used as a **tool to assist in decision-making** regarding the choice of materials and the quantity deposited. The multilayer solutions selected for these tests were those that were the most promising in terms of barrier and sealing performance, both for flexible and rigid packaging materials. Regarding flexible packaging, three different pilot reels with increased paper ratio and/or bio-based content have been tested. About rigid packaging, six different samples of functionalised cellulosic trays were tested. Trays were produced with a semi-industrial equipment by the wet molding process, using different types and quantities of additives.

Recyclability tests were carried out by Innovhub at laboratory scale, in accordance with the current European CEPI Recyclability laboratory test method, Part I – Recycling mill with Conventional process [2], which enables analysing both recycling process parameters and recycled paper quality parameters. Results have been assessed according to 4evergreen Fibre-based packaging recyclability Evaluation Protocol, Part 1 [3], which describes the assessment of the technical recyclability.

Additionally, **compostability** of the most promising cellulosic tray, intended for fresh and fatty meals, whose residues could potentially generate problems in the paper collection and recycling loop. 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 ISO 20200 at home composting conditions.

II. METHODOLOGY

III.1. 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 [2]. 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 impurities) of products produced from recycled fibres.

This test method consists of several stages (Figure 1) and defines the parameters of interest for the verification of recyclability as follows:

- **Disintegration at low consistency, 10 minutes disintegration time.** Ease at which the fibres and packaging components can be separated in water, simulating conditions of a recycling mill with conventional process.
- **Filtrate analysis.** Level of dissolved and/or colloidal substances below 12 microns, measured as Evaporation Residue.
- **Coarse screening (5 mm holes).** Determination of the Coarse Reject amount.
- **Fine screening (0.15 mm slots).** Determination of the Fine Reject amount.
- **Visual impurities and sheet adhesion test** on paper sheet produced with the accept pulp after coarse screening (“AC sheets”) and fine screening (“AF sheets”).
- Determination of the content of adhesive particles, **Macro Stickies** (optional).



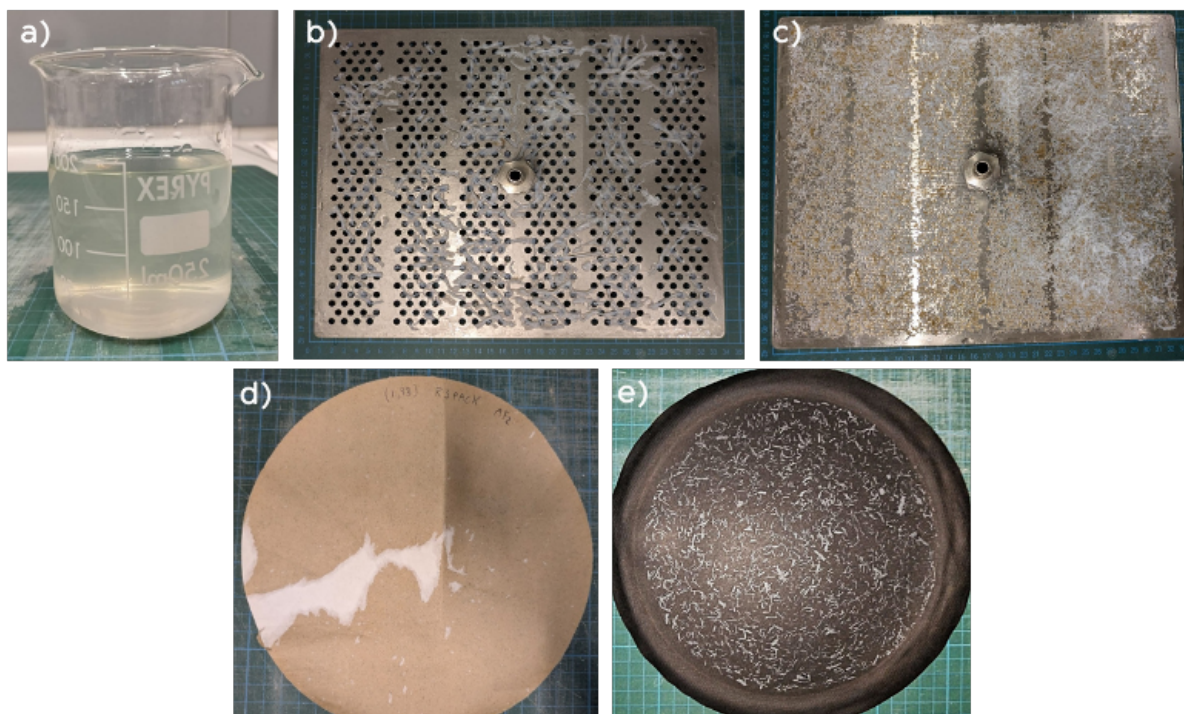


FIGURE 1 : PICTURES OF THE DIFFERENT STAGES OF THE LABORATORY CEPI V3 RECYCLABILITY TEST METHOD WITH A) FILTRATE ANALYSIS, B) COARSE SCREENING, C) FINE SCREENING, D) VISUAL IMPURITIES AND SHEET ADHESION, E) MACRO STICKIES.

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.

This document describes the assessment and score calculation procedures of the technical recyclability of fibre-based packaging products and/or materials. The Evaluation Protocol focuses on the technical recyclability without considering collection, sorting, or the effects of recycling various packaging items together. Possible presence of food residues is not considered.

In order to calculate the recyclability score, several output values of the lab test are considered, in particular the **sum of three components**: screening and mass yield, visual impurities and sheet adhesion. Macro Stickies parameter is not covered in the current version of the Evaluation Protocol, but it will be probably included in a later version.

The Technical Recyclability Scores range from +100 to -100 points (see Figure 2), where the higher numbers indicate better technical recyclability. A negative Technical Recyclability Score means that the assessed product is not technically recyclable in this specific recycling process type (see FIGURE 3).

The recyclability score was calculated by adding up the points and evaluation obtained for each of the following parameters:

- Disintegration time, in minutes;
- Total Screening Yield % (Coarse reject and Fine reject);
- Evaporation residue, in mg/g packaging;
- Sheet adhesion from level 1 to 3 (with level 3 representing the lowest performance);
- Visual impurities from level 1 to 4 (with level 4 representing the lowest performance);

Total Screening Yield Score		Visual Impurities Score			Sheet Adhesion Score			Dissolved and Colloidal Substances Score	
Score	Score interpretation	Level	Score	Score interpretation	Level	Score	Score interpretation	Score	Score interpretation
100 - 90	The packaging creates a high screening yield in a recycling mill with conventional process and is therefore considered 'best in class'.	Level 1	0	Poses no visual quality issues	Level 1	0	Poses no adhesion issues	0 to -3	DCS expected to pose negligible issues in the process of a recycling mill with conventional process and is therefore considered 'best in class'.
		Level 2	-5	Poses minor visual quality issues	Level 2	0	Poses minor adhesion issues		
89 - 70	The packaging creates an acceptable screening yield, but the rejects could already have an impact in a recycling mill with conventional process.	Level 3	-15	Poses noticeable visual quality issues	Level 3	Knockout	Poses significant adhesion issues that can have a significant impact on a recycling mill with conventional process and is therefore not recyclable.	-3 to -55	DCS expected to have minor issues in the process of a recycling mill with conventional process.
69 - 50	The screening yield of the packaging is high for a recycling mill with conventional process, but it is suggested that the packaging should be further optimised for recycling.	Level 4*	-30	Poses significant visual quality issues				-55 to -100	DCS expected to have issues in the process of a recycling mill with conventional process and suggest further optimisation.
49 - 0	This packaging creates a significant amount of rejects which can lead to technical problems in the screening step in a recycling mill with conventional process.							< -100	DCS expected to have major issues in the process of a recycling mill with conventional process. It is recommended to test this product with part III testing in future FBCP process.
< 0	The reject of this packaging is too high for the recycling process in a recycling mill with conventional process and should not be recycled in such a process.								

FIGURE 2: 4EVERGREEN FIBRE-BASED PACKAGING RECYCLABILITY EVALUATION PROTOCOL, PART 1, VERSION 1, ISSUED IN JANUARY 2025, INTERPRETATION OF INDIVIDUAL PARAMETERS.

Technical Recyclability Score for Recycling in mills with conventional process	Description
0 - 100	Technically recyclable in a recycling mill with conventional process.
< 0	Not technically recyclable in a recycling mill with conventional process. Potentially recyclable in mills with other recycling process types.*

FIGURE 3: 4EVERGREEN FIBRE-BASED PACKAGING RECYCLABILITY EVALUATION PROTOCOL, PART 1, VERSION 1, ISSUED IN JANUARY 2025, INTERPRETATION OF TECHNICAL RECYCLABILITY SCORE.

III.2. Compostability

The purpose of this test was to evaluate the quantitative disintegration of the sample in a composting bin at laboratory-scale test, in the presence of a freshly prepared synthetic waste according to the standard ISO 20200:2023 [4].

The composting process was carried out at **home composting conditions** (temperature <30°C), and it was monitored regularly and conducted until the complete disintegration of the sample.

The main process parameters monitored during the test are the following:

- The temperature is detected every day till the end of the test.
- Periodically the composting material is turned and visually inspected (once every two weeks) the bin is weighted and added by water to restore humidity losses due to evaporation.



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- At the beginning and at the end of the test dry weight, volatile solids, pH and total Nitrogen are measured on the composting waste for each reactor.

Additionally, the determination of some heavy metals of possible concern and other hazardous substances was carried out.

Determination of Arsenic (As), Cadmium (Cd), Chrome (Cr), Mercury (Hg), Nickel (Ni), Lead (Pb), Copper (Cu), Selenium (Se), Zinc (Zn) e Molybdenum (Mo): EN 15411:2011.

Determination of Fluorine (F): EPA 5050 and EPA 9056A.



IV. RESULTS AND DISCUSSIONS

IV.1. Flexible paper-based packaging

Three different pilot reels were subsequently produced, with each reel iteration aiming to increase the paper ratio and/or bio-based content.

REEL 1 was produced in January 2024 as a reference, without MFC precoat, to initially test the processability of partially bio-based coatings developed by BIM KEMI. Structure: MG kraft paper / BIM biobased coating / BIM biobased heat-seal coating.
Paper 71% / Biobased 90%

REEL 2 was produced in March 2024 by using a MFC precoated reel sent by Fiberlean as paper substrate, with a BIM petroleum-based heat-seal topcoat. Structure: FIBERLEAN paper / BIM biobased coating / BIM petrobased heat-seal coating.

Paper 77% / Biobased 85%

The paper ratio has been increased from 71% to 77% thanks to the use of 100% cellulose-based MFC material, but also to the petroleum-based heat-seal topcoat, which provides same barriers than BIM biobased coating but with a thinner layer deposited.

REEL 3 was produced in January 2025 by using biobased CNC-based coating as a primer to improve gas barrier properties of the multilayer paper-based solution. Structure: FIBERLEAN paper / CNC-based primer / BIM heat-seal coating.

Paper 80% / Biobased 90%

80% paper ratio has been reached, while displaying a very high biobased content of 90%, as well as very good barrier and sealing performance (detailed in Deliverable 4.5 “Process methodology for producing high-performance fibre-based, high-barrier packaging that can be upscaled for commercialisation”).

Below are detailed images of all the stages of the CEPI recyclability test protocol for each of the three paper reels developed within the R3PACK programme.

COARSE SCREENING:

Somerville type screening with 5 mm holes screen plate. 50 g pulp amount, screening time 5 minutes.

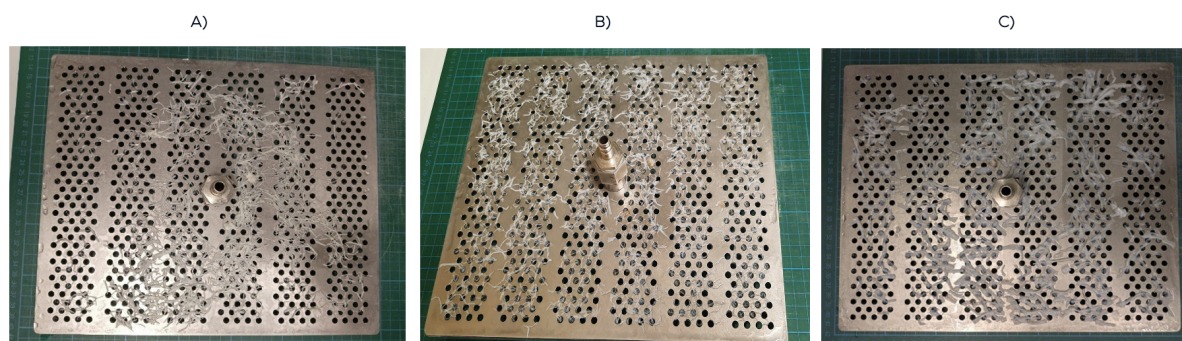


FIGURE 4: PICTURES OF THE COARSE REJECT ON THE SOMERVILLE SCREEN PLATE. A) REEL 1, B) REEL 2, C) REEL 3

The FIGURE 4 show the coarse reject deposited on the Somerville screening plate at the end of the coarse screening step of the test. The coarse reject is mainly made of large coating fragments.



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FINE SCREENING:

Somerville type screening with 0,15 mm slots screen plate. 20 g pulp amount (accept pulp after coarse screening), screening time 20 minutes.

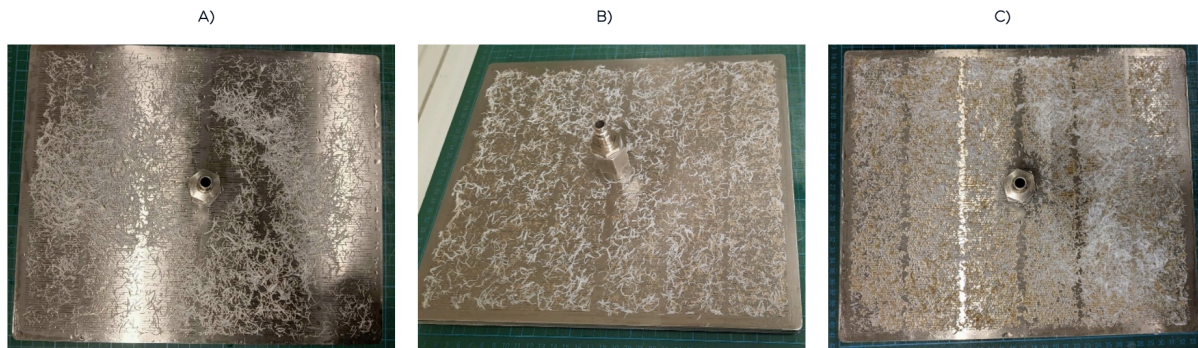


FIGURE 5: PICTURES OF THE FINE REJECT ON THE SOMERVILLE SCREEN PLATE. A) REEL 1, B) REEL 2, c) REEL 3

The FIGURE 5 show the fine reject deposited on the Somerville screening plate at the end of the fine screening step of the test. The fine reject is made of fragmented coating particles.

FILTRATE ANALYSIS:

An amount of the stock pulp is filtered using specific filter paper (12 - 15 μm) and the filtrate is obtained (see FIGURE 6). A known volume of filtrate is used for the determination of the evaporation residue.

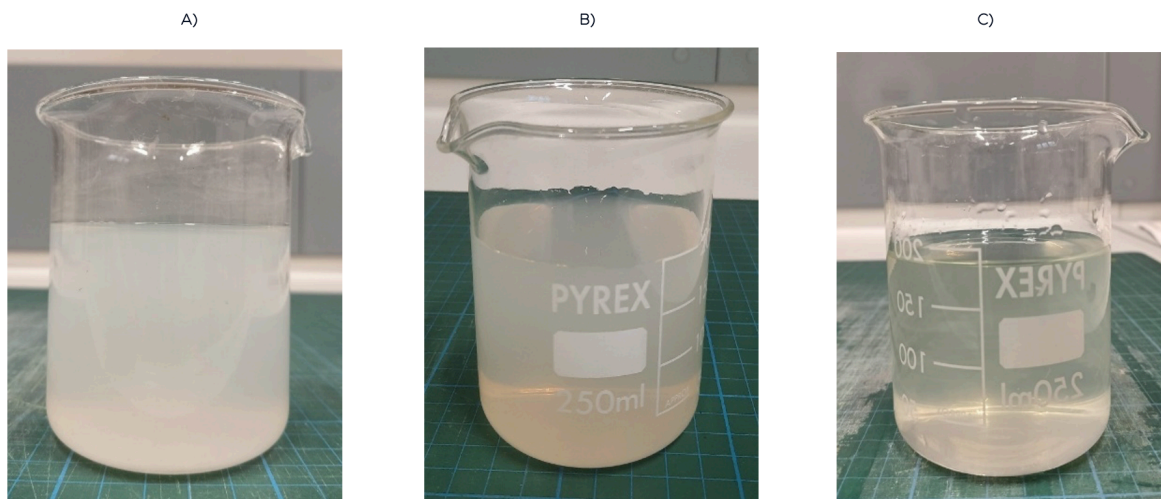


FIGURE 6: PICTURES OF THE FILTRATE. A) REEL 1, B) REEL 2, c) REEL 3

LABORATORY SHEETS:

Paper sheets produced with the accept pulp after the fine screening (“AF sheets”). Visual impurities and sheet adhesion test.



FIGURE 7: PICTURES OF THE AF SHEETS. A) REEL 1, B) REEL 2, C) REEL 3

The FIGURE 7 show the laboratory sheets produced with the recycled pulp at the end of the test. In the case of sample C), the drying support remained attached to the sheet showing partial adhesiveness.

The summary of the results related to the flexible paper-based multilayer packaging solutions developed is presented in **Erreur ! Source du renvoi introuvable.1**.

TABLE 1: SUMMARY OF RECYCLABILITY RESULTS RELATED TO FLEXIBLE PACKAGING

PARAMETERS	REEL 1	REEL 2	REEL 3
Coarse Rejects (%)	4,13	2,49	4,11
Fine Rejects (%)	8,96	12,43	10,31
Evaporation residue (g/kg packaging)	115,4	80,2	72,0
AC sheets - Adhesion test	Present	Present	Present
AC sheets - Visual impurities	Level 3	Level 3	Level 3
AF sheets - Adhesion test	Absent	Absent	Present
AF sheets - Visual impurities	Level 1	Level 1	Level 2
Area Macrostickies < 2000 μm (mm^2/kg)	306.226	430.190	946.367
OPTIONAL			
RECYCLABILITY SCORE	21	33	Not Recyclable*

***Knockout because of sheet adhesion**

REELS 1 and 2 showed a similar recycling behaviour and they **are considered technically recyclable in a recycling mill with conventional process.**

These materials highlighted some good recyclability properties, in particular:

- Low fragmentation of the coating layer and a complete screening of the rejects;
- Good quality of recycled paper with no adhesion and no visual impurities on paper sheets after fine screening.

However, a significant amount of Macrostickies < 2 mm was measured and a non-negligible amount of dissolved and colloidal substances were detected in the residual water. These results are highlighted in yellow/orange in Table 1, because they could have a negative effect on recyclability and they should be improved in future developments.

Although Macrostickies are optional in the test and are currently not part of the 4evergreen recycling score evaluation, maintaining a low level is preferable to minimise the risk of operating issues and web paper breakage during the production of paper made with recycled cellulose fibres.

REEL 3 behaves similarly to REELS 1 and 2, except that it fails the ‘sheet adhesion’ test; this means that laboratory sheets made of recycled fibres had an adhesive and sticky behaviour during the drying stage. REEL 3 is currently considered not technically recyclable because of sheet adhesion, this outcome is highlighted in red in the table of results. This is probably due to a higher fragmentation of the coating layer, and therefore an incomplete screening of small adhesive particles, possibly deriving from the heat seal layer. Indeed, the test showed an adhesion of the paper sheets in correspondence with individual adhesive particles, not in the bulk of fibre or the entire surface of the sheets.

To better understand the substances present in the recovered cellulosic fibres post-recycling and thus improve the paper-based multilayer structure to reduce adhesiveness, further chemical analysis should be performed. IR analyses carried out on the fine reject particles showed that they are mainly made of methacrylate polymers, a component of the heat-seal coating.

As a future development, it is believed that **optimizing the quantity and possibly the formulation of the heat seal coating can solve this issue**, obtaining a lower fragmentation of the coating and a lower adhesion of the polymer particles at the temperature in which the sheet adhesion test is carried out.

IV.2. Rigid packaging - molded cellulose trays

RECYCLABILITY

Recyclability tests were carried out at Innovhub laboratory on functionalised cellulosic trays produced at the Swedish RISE Institute with a semi-industrial equipment. Trays were produced by the wet molding process, in which the wet cellulose pulp is formed into a tray using a mold, then hot-pressed and dried to remove the water from the tray. To obtain better quality cellulose trays, i.e. with a more closed and denser surface as well as barrier performance, RISE adjusted the machine settings (time, temperature, pressure, etc.) and the pulp recipe by testing different types and quantities of additives (biobased, wet strength, etc).

A reference wet moulded tray was first tested, made 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. Polyaminoamide-epichlorohydrin (PAE) is a permanent wet-strength agent that confers water resistance to cellulose pulp in wet conditions, while

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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).

Top coating is a PHBV-based dispersion coating for spray application. PHBV was selected for its highly attractive barrier properties (oil, water) but above all for its bio-based and biodegradable nature in all natural environments (industrial and home composting, soil, fresh water and marine biodegradation).

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, i.e. PAE, MFC or starch material.

Tray E belongs to another production batch, with an AKD content of 5 %, no chitosan and MFC layers and same PHBV-based heat-seal top layer, having undergone a hot-press stage to smooth the surface.

In order to assess the recyclability of the developed cellulosic trays, the same test method and evaluation protocol were used as for the flexible packaging described above.



COARSE SCREENING:

Somerville type screening with 5 mm holes screen plate. 50 g pulp amount, screening time 5 minutes.

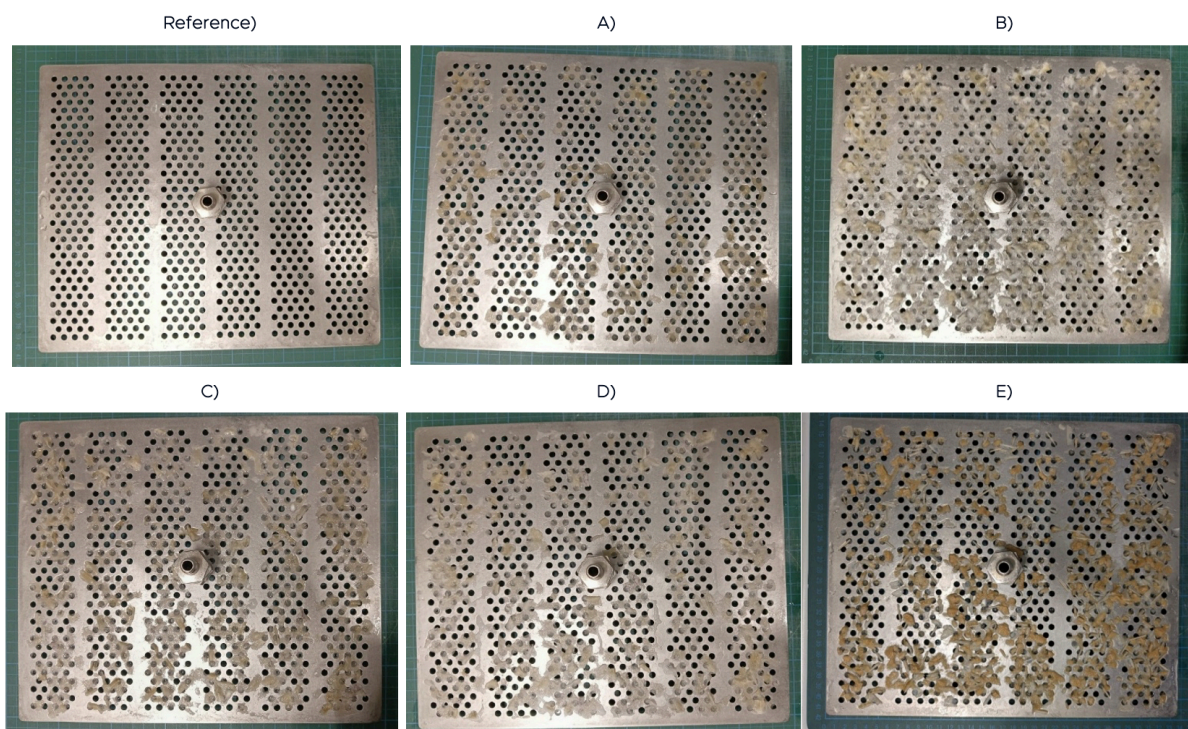


FIGURE 8: PICTURES OF THE COARSE REJECT ON THE SOMERVILLE SCREEN PLATE. REFERENCE), TRAY A), TRAY B), TRAY C), TRAY D), TRAY E).

The FIGURE 8 illustrates the coarse reject deposited on the Somerville screening plate at the end of the coarse screening step of the test. The coarse reject is mainly made of undisintegrated cellulose fragments, with a certain variability among samples: in case of Tray A) the coarse reject was almost non-existent, while Trays B and E have shown the highest amount.

FINE SCREENING:

Somerville type screening with 0,15 mm slots screen plate. 20 g pulp amount (accept pulp after coarse screening), screening time 20 minutes.

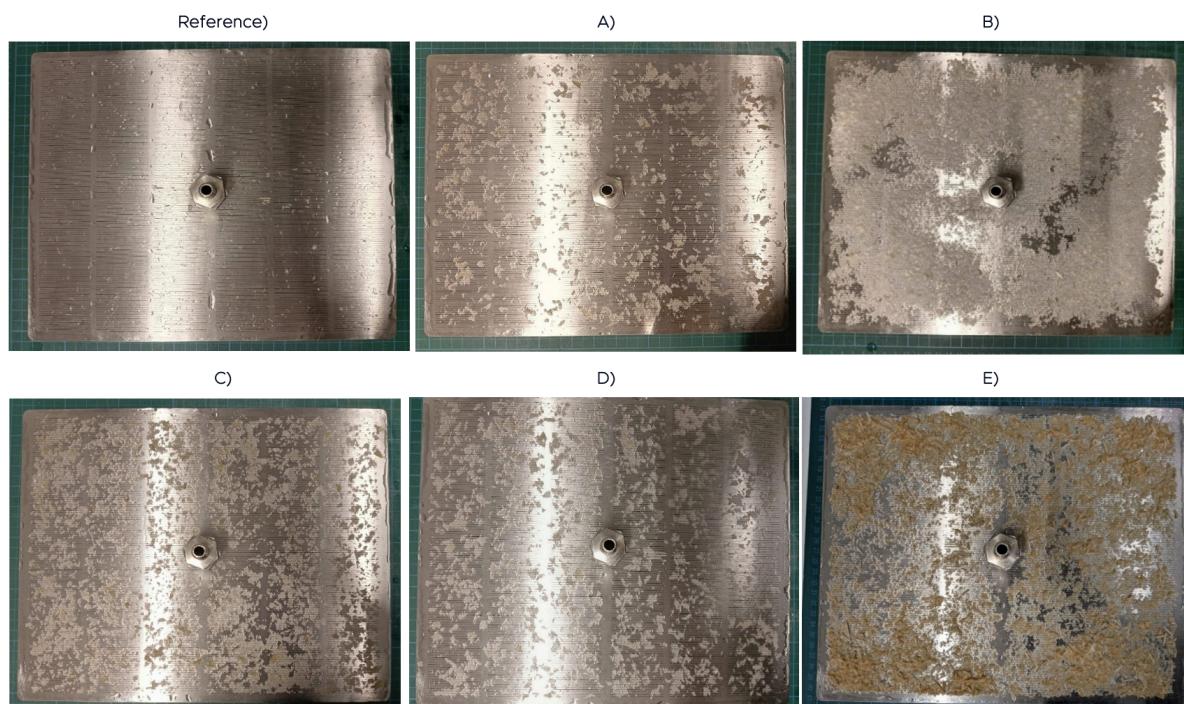


FIGURE 9: PICTURES OF THE FINE REJECT ON THE SOMERVILLE SCREEN PLATE. REFERENCE), TRAY A), TRAY B), TRAY C), TRAY D), TRAY E).

The FIGURE 9 show the fine reject deposited on the Somerville screening plate at the end of the fine screening. The fine reject is mainly made of cellulose fragments. As for the coarse reject, Trays B and E have shown the highest amount, possibly due respectively to the PAE additive in the pulp and the hot-pressing process used onto the PHBV top layer.

FILTRATE ANALYSIS:

An amount of the stock pulp is filtered using specific filter paper (12 - 15 μm) and the filtrate is obtained (see FIGURE 10). A known volume of filtrate is used for the determination of the evaporation residue.

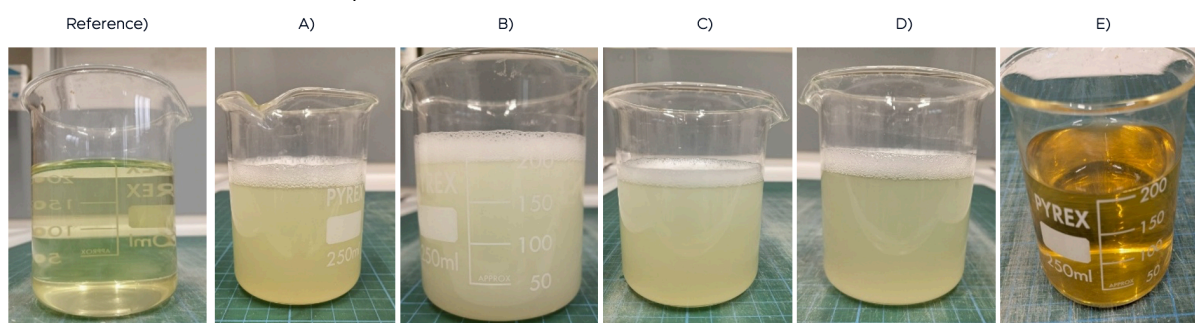


FIGURE 10: PICTURES OF THE FILTRATE. REFERENCE), TRAY A), TRAY B), TRAY C), TRAY D), TRAY E).

LABORATORY SHEETS:

Paper sheets produced with the accept pulp after the fine screening ("AF sheets", see FIGURE 11). Visual impurities and sheet adhesion test.

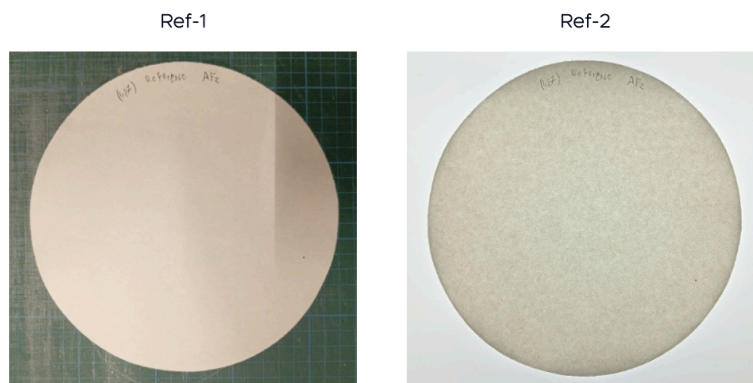


FIGURE 11: PICTURES OF THE AF SHEETS. REF-1 WITH REFLECTED LIGHT, REF-2 WITH TRANSMITTED LIGHT

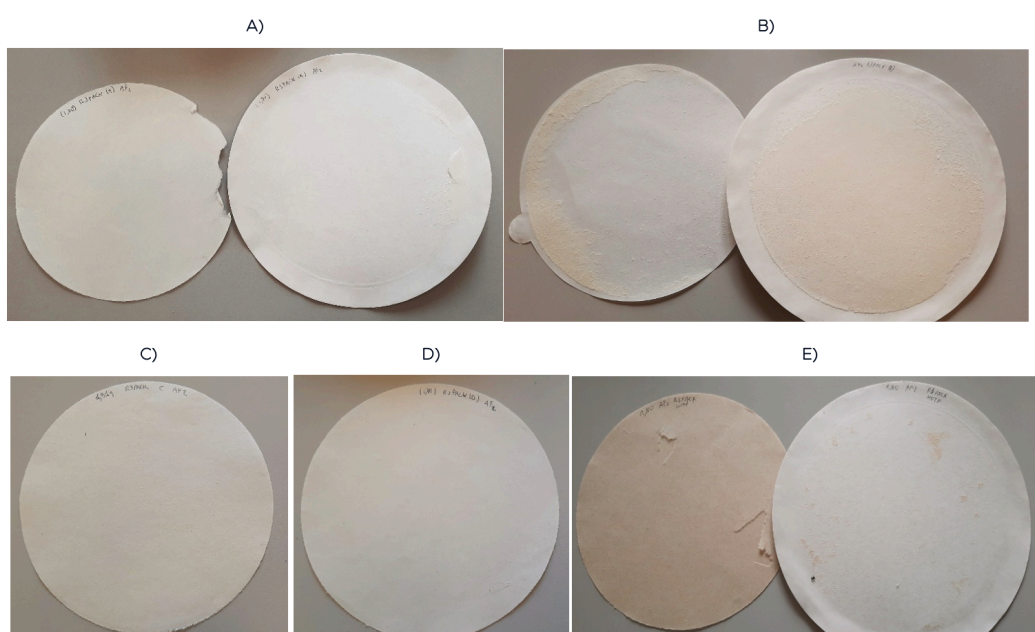


FIGURE 12: PICTURES OF THE AF SHEETS. TRAY A), TRAY B), TRAY C), TRAY D), TRAY E).

The FIGURE 12 illustrates the laboratory sheets produced with the recycled pulp at the end of the test. In the case of sample A), B), E), the sheet appeared to break and/or part of the drying support remained attached to the sheet.

The summary of recyclability results related to the six different cellulose tray packaging solutions developed is reported in **Erreur ! Source du renvoi introuvable.2**.

TABLE 2: SUMMARY OF RECYCLABILITY RESULTS RELATED TO RIGID PACKAGING

PARAMETERS	Reference Tray	Tray A	Tray B	Tray C	Tray D	Tray E
Coarse Rejects (%)	< 0,1	1,50	3,74	1,97	1,74	4,55
Fine Rejects (%)	0,15	1,18	8,09	2,30	1,62	15,75
Evaporation residue (g/kg packaging)	3,4	24,6	50,8	29,9	23,8	14,1
AC sheets - Adhesion test	Partly present	Present	Present	Present	Present	Present
AC sheets - Visual impurities	Level 1	Level 3	Level 3	Level 3	Level 3	Level 3
AF sheets - Adhesion test	Absent	Present	Present	Partly	Partly	Present
AF sheets - Visual impurities	Level 1	Level 1	Level 1	Level 1	Level 1	Level 1
Area Macrostickies < 2000 μm (mm ² /kg) - OPTIONAL	< 500	< 500	< 500	540	< 500	< 500
RECYCLABILITY SCORE	100	Not Recyclable*	Not Recyclable*	91	93	Not Recyclable*

Tray C and D showed a good recycling behaviour and they **are considered technically recyclable in a recycling mill with conventional process, with a very good technical recyclability score above 90/100**.

These materials highlighted some good recyclability properties, in particular:

- Low quantity of Rejects;
- Very low quantity of insoluble adhesive particles in the pulp (Macrostickies).
- Intermediate values for Evaporation residue, acceptable without any specific issues.

Tray B showed a negative impact of PAE additive, even at low dosage, with higher Fine and Coarse rejects rates, which is not the case with 10% MFC additive (Tray C) or 0.5% starch additive (Tray D). It is known that the addition of PAE in the pulp increases inter-fibres bonding, inhibiting the fibre network breakdown and giving wet resistance [5]. On the other hand, PAE can adversely affect paper disintegration and repulping, even at a low concentration of 0,5%.

Tray E showed a very high quantity of total rejects, in particular Fine rejects, possibly due to the hot-pressing step and/or a direct deposition of the molten PHBV layer onto the cellulose fibres, without MFC and chitosan as intermediate layers, thus possibly increasing the penetration of the coating into the fibre network.

The main general outcome of this series of tests is that **Sheet Adhesiveness is the key factor for the recyclability** of this type of trays. Sheet adhesion occurred in Trays A, B, E, meaning that laboratory sheets made of recycled fibres had an adhesive and sticky behaviour during the drying stage. It was not possible to separate the sheets from the drying supports without a visible damage to the sheets themselves. **Adhesiveness is due to some soluble substances from the barrier layers.** Adhesiveness could be impacted by the water-soluble binders composing the PHBV spray-coating formulation, assuming that it passes through the sieves and thus resulted in the formation of sticky fibres. To go further, investigation need to be done on the origin of this sticky behaviour, potentially originating from the heat-seal coating fragmentation, and perhaps also on the presence of MFCs as primer. MFC is an interesting primer to possibly improve interlayer adhesion strength and reduce



delamination issues, as explained in Deliverable D4.5 from the R3PACK project, but can impact final repulpability and so recyclability.

COMPOSTABILITY:

Tray C (Pulp + 1 % AKD + 10 % MFC / 10-15 g/m² chitosan / 10-15 g/m² MFC / 50 g/m² PHBV-based top coating) is almost 100 % biobased and recyclable, and was selected for compostability testing due to its composition, with almost 100% of the materials being theoretically home-compostable.

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).

- **Biodegradability** (ISO 14855) was already taken for granted for Tray C with substances and materials composing the tray packaging being 100% biobased and biodegradable.
- **Ecotoxicity** (OECD 208) was not verified in the laboratory, due to lack of sample quantity for performing this test.
- **Disintegration (ISO 16929 / ISO 20200)**

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.

Three plastic bins, each 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.

The total duration of the test was 84 days (see FIGURE 13). During the test, the odour and visual inspections evidenced a regular aerobic composting process. The visual aspect of the synthetic waste in the bins evolved starting from an initial darkening.

During the first three weeks:

- The growth of white moulds was noted, which increased up to cover major part of the biowaste.
- The specimens did not show any significant modifications, but it started to be dark.

After 34 days of test: The disintegration process quickly increased with a reduction of the dimensions and a fragmentation of the sample specimens.



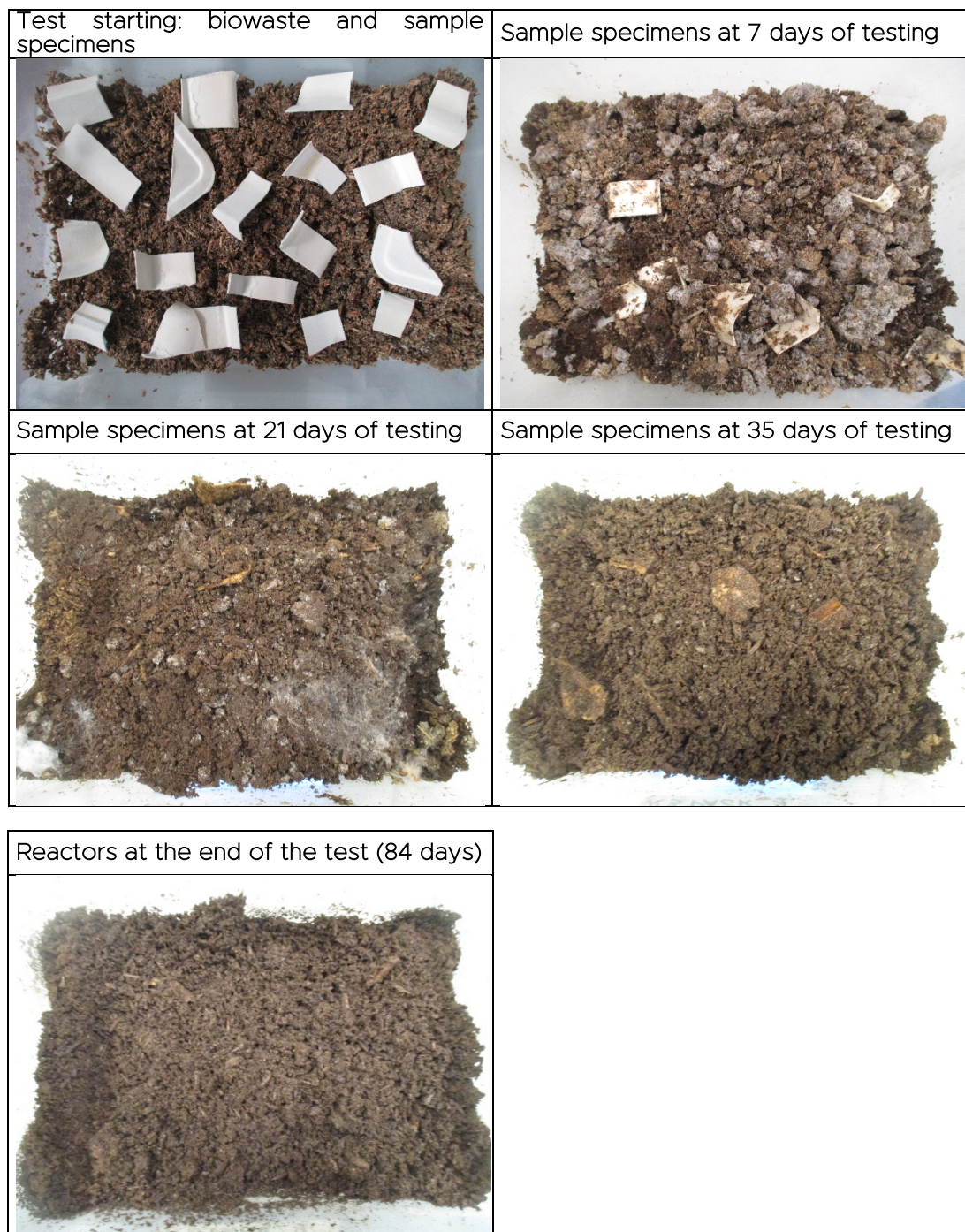


FIGURE 13 PICTURES OF THE SAMPLE DURING THE TEST.

TABLE 3: RESULTS OF THE DISINTEGRATION TEST.

	Weight of sample residues (g) as dry weight				Disintegration (%)	Average disintegration (%)	Disintegration limit required (%)
	Initial	2-5 mm	>5mm	Total			
Reactor 1	15.0	0.0	0.0	0.0	100	100%	≥90
Reactor 2	15.0	0.0	0.0	0.0	100		
Reactor 3	15.0	0.0	0.0	0.0	100		

TABLE 4: VALIDATION OF THE DISINTEGRATION PROCESS (ISO 20200:2023 PARAGRAPH 13)

	YES	NO
The decrease in volatile-solids content is greater than or equal to 30% after 12 weeks?	X	
Is the difference among the degrees of disintegration of the three replicates < than 20%?	X	
Validation of the disintegration process:	X	

⇒ **The complete disintegration was evident at the end of the test, when no residual sample specimens could be observed (refer to TABLE 3, TABLE 4).**

At the end of the test the content of the three reactors was sieved with sieves of 5 mm and 2 mm dimensions: no residual fractions of the sample were recovered from all three reactors. The sieved compost was used for the determination of the quality of the compost in terms of chemical characterization (TABLE 5).

TABLE 5: CHEMICAL CHARACTERIZATION OF THE COMPOST OBTAINED AT THE END OF THE TEST.

	Reactor _1	Reactor _2	Reactor _3
Final reactor total mass (g)	1431	1355	1549
Humidity (%)	0.7	0.7	0.7
Volatile solids (%)	81.1	63.8	78.3
pH	6.7	7.1	7.4
N tot (%)	2.0	2.1	2.1
C/N ratio	19.8	15.1	18.2

TABLE 6: HEAVY METALS AND OTHER TOXIC AND HAZARDOUS SUBSTANCES AND RELATED LIMITS SPECIFIED IN THE STANDARD NORM EN 13432:2000 ANNEX A.1.2/AC:2005, TABLE A.1.

Element	Concentration (mg/kg)	Limits EN 13432:2000 (mg/kg)
Cr	1.6	50
Co	<0.1	38 (a)
Ni	0.7	25
Cu	1.0	50
Zn	14.5	150
As	<0.1	5
Se	<0.1	0.75
Mo	<0.1	1
Cd	<0.1	0.5
Pb	<0.1	50
Hg	<0.05	0.5
F	<50	100



**Funded by
the European Union**

This project has received funding from the European Union's Horizon Europe Research and Innovation programme under Grant Agreement No 101060806. This document reflects the views of the author(s) and does not necessarily reflect the views or policy of the European Commission. Whilst efforts have been made to ensure the accuracy and completeness of this document, the European Commission shall not be liable for any errors or omissions, however

The concentration of heavy metals and other toxic and hazardous substances of the sample Tray C complies with the limits specified by the EN 13432:2000 Annex A.1.2/AC:2005 reported in table A.1.

The test to determine the compostability of cellulose tray C was a success with very positive results. The sample Tray C disintegrated at 100% value within 84 days of test, therefore showing 100% quantitative disintegration value at home composting conditions.



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V. CONCLUSIONS

Recyclability tests at lab scale according to the CEPI recyclability laboratory test method and 4evergreen Fibre-based packaging recyclability Evaluation Protocol have shown overall positive results but also some critical issues for both flexible and rigid fibre-based packaging.

Regarding the three paper-based flexible packaging reels tested in the R3PACK project, the fragmentation of the barrier layers, especially the heat-seal layer, is very important. Low fragmentation of the coating layer enables an efficient and almost complete screening of the rejects from the pulp, with a good quality of recycled paper with no adhesion and no visual impurities after fine screening.

The quantity of dissolved substances in the filtrate is quite high, even if still acceptable for the recycling process.

In REEL 3, the one with the highest paper content of 80% and biobased ratio of 90%, 'sheet adhesion' occurred, probably due to a higher fragmentation of the coating layer, and therefore an incomplete screening of small adhesive particles, possibly deriving from the heat seal layer.

The recommendation for this type of flexible packaging materials is to optimize the type and quantity of soluble and sealing coatings in order to lower the quantity of dissolved substances in the water, reduce the quantity of adhesive particles in the pulp and decrease the level of sheet adhesiveness.

Regarding the six rigid cellulose tray packaging tested in the project, recyclability properties are good in terms of low quantity of rejects and very low quantity of insoluble adhesive particles in the pulp (Macro Stickies).

Sheet adhesion occurred in Trays A, B, E, due to some soluble substances from the barrier layers, assuming that they pass through the sieves and they make the bulk of fibres sticky at drying conditions.

Sheet Adhesiveness is the key factor for the recyclability behavior of this series of trays and it would need further investigation regarding the possible impact of the water-soluble binders composing the PHBV spray-coating formulation and/or the MFC used as a primer.

The recommendation for this type of rigid packaging trays is to optimize the type and quantity of soluble coatings, in particular the PHBV spray-coating formulation, in order to decrease the level of sheet adhesiveness.

Finally, it is important to bear in mind that these results need to be consolidated and repeated for the confirmation of repeatability and reproducibility; furthermore, it is still difficult to clearly differentiate the impact of each material on recyclability due to disparities in multilayer structures, as well as process and application method.



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