D3.8 GUIDELINES ON DESIGN FOR RECYCLING

WORK PACKAGE 3

Associated Task(s):
T3.7 Design for sorting and recycling guidelines

Lead Partner: UM
Partners involved: Fraunhofer, Polysecure, Siegwerk, Amcor-G, Suez
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QUALITY REVIEW

Quality check	Date	Status	Comments
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ABBREVIATIONS

Abbreviation	
CFP	CIRCULAR FoodPack
DfR	Design for Recyclability
FAQ	Frequently asked questions
PE	Polyethylene
TBS	Tracer Based Sorting

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PUBLISHABLE EXECUTIVE SUMMARY

Based on semi-industrial trials with tracer-based sorting and de-inking of laminated printed PE-based flexible packaging, the project has distilled Design for Recycling guidance for this specific circularity route. This guidance is complimentary to existing guidelines on (printing on) PE flexibles.

Furthermore, we have added some Frequently Asked Questions (FAQ) and key learnings from the trials.

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1. INTRODUCTION

The EU-funded project CIRCULAR FoodPack comprises different loops, demonstrating the circular use of plastic packaging material. One of those loops (so-called Loop 3) aims for the circularity of PE-based mono-material food-grade packaging items, running two cycles of packaging production, sorting, and recycling. First, PE-based mono-material laminates and pouches are produced by AMCOR and NESTLE, respectively. During the lamination process, tracers provided by Polysecure are integrated into the food packaging items as part of the printing ink. This artificially produced household flexible packaging waste material is then mixed with the real household waste from Poitiers, France, provided by SUEZ. Tracer-Based-Sorting (TBS), a process developed by Polysecure, serves as the sorting protocol and takes place at the Steinert Technical Center in Pulheim, Cologne, Germany. After TBS, the materials are de-inked/delaminated and recycled into new films, which is evaluated in other deliverables.

In this deliverable, CIRCULAR FoodPack aims to provide a practical guideline to support industry towards the future implementation of new technologies like TBS, de-inking and delamination in accordance with recyclability.

2. FRAMEWORK OF DELIVERABLE AND THE GUIDELINE

In terms of **obtaining effective recyclability** with flexible LDPE-based TBS-marked food packaging items, there are two main blocks in the value chain to evaluate:

1. **Sortability**: will the packaging item effectively be sorted to its intended stream?

2. **Recyclability**: assuming sortability, will the packaging item be recycled without the tracer disturbing the quality of recycling (of the intended stream)?

If the answer to both questions is yes, the packaging item can be considered recyclable.

For the CIRCULAR FoodPack guidelines to be practical, it should be aligned with existing DfR guidelines in the market for flexible LDPE, the most prevalent of which are RecyClass[1] and CEFLEX[2]. Both widespread guidelines include a section on inks already. The tracer, which is added to accomplish TBS, is added to the ink system (in the concept of CIRCULAR FoodPack). Therefore, when it comes to the aspect of (not disturbing) recyclability, ***we defer to the inks section of existing guidelines***.

The DfR guideline focuses instead on that aspect, which is not yet worked out, the sortability. Tracers are not yet part of current packaging design. Rather, they are a potential way forward for better circularity of food-grade LDPE-based flexibles. Therefore, the **DfR guideline for TBS provides guidance for proper packaging design to ensure that the packaging would be effectively sorted out to its intended stream, *assuming the infrastructure for TBS is effectively in place*.** The guideline itself is based on the project results and will only make statements on aspects which have been

[1] https://recyclass.eu/recyclability/design-for-recycling-guidelines/
[2] https://guidelines.ceflex.eu/guidelines/

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extensively tested and validated in the project. Additionally, a FAQ is added, wherein we offer experience-based assumptions on circumstances which were not explicitly tested, or only in an explorative way.

Finally, this deliverable will also offer key learnings regarding the de-inking and delamination steps of the recycling process within CIRCULAR FoodPack.

Summarizing, this deliverable contains three result sections, discussed in Sections 3, 4, and 5:

3. DfR guideline for TBS of LDPE-based food packaging

4. FAQ beyond the boundaries of the above DfR guideline

5. Key learnings de-inking and delamination of LDPE-based food packaging

The DfR guideline is based on the project results from Loop3, food packaging with the following specifications:

- Multilayer PE/PE packaging with top layer of 25 mm MDOPE
- Tracers added in the ink system
- Reverse printing on top layer
- Lamination by 2-component PU adhesives, solvent free, 2gsm typically
- Best performing tracer formulation available at time of testing (2023)
- Detection technology used (in state-of-the-art NIR sorting machine): hyperspectral imaging camera, wavelength 970-1700 nm, wavelength resolution 3 nm, spatial resolution 5 mm.

Please note: the Tracer-Based-Sorting technology as developed in the project and described here in this deliverable is compatible to sorting technologies used in Materials Recovery Facilities nowadays. As long as the NIR sorting line is equipped with a suitable HSI detection technology, e.g. Steinert technology, Pulheim, Germany, TBS can be implemented on the line with a feasible upgrade.

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3. DFR GUIDELINE FOR TBS OF LDPE-BASED FOOD PACKAGING

Table 1: DFR guideline for TBS of LDPE-based food packaging

Item	Guidance
Type of printing	Tracers can be used for surface print and reverse print (requires top polymer layer and any print layers on top to be transparent in the relevant NIR range[3])
Printing techniques	Gravure printing has been verified in the project. Others: see FAQ.
Minimum size of individual printed area	Dot with 10 mm diameter or square with 10 mm edge length
Minimum tracer coverage of whole packaging	Stripe of minimum 10 mm width around the object, or multiple patches evenly distributed over the surface. The number and/or size of patches per packaging item depend on size and geometry and should be chosen such that from all directions at least one dot (better two) of the above minimum size is visible. This condition should also hold under commonly foreseeable levels of soiling, flattening, folding, crumpling and fragmentation of the packaging item during usage and waste phases. General recommendation: Use at least double the minimum tracer area, or two patches, to be visible from all sides. Avoid surfaces which are likely to be folded inwards.
Usable colors/pigments for tracer incorporation	White (titanium dioxide) Yellow (organic) on top of white background color Others: see FAQ
Minimum area concentration of tracer	20 µg/cm² in white or in yellow on white background.
Maximum area concentration of tracer	50 µg/cm². The maximum concentration is determined by the maximum ink viscosity and may vary slightly depending on ink types and process parameters.
Overprinting or lacquering	With transparent[3] print/lacquer only.
Mixing and application procedure	Tracer should be mixed into ready-to-print ink. Additional dilution steps after mixing must be avoided or must be taken into account in the tracer concentration calculation. No variable dilution adjustment after tracer addition. Mixed tracer-ink has no shelf-life due to sedimentation of the tracer particles. Stir immediately before use and keep stirred continuously during usage.
Mixing equipment	Use fast stirrer, no grinder. Grinding damages the tracer particles and reduces fluorescence efficiency.

[3] 900 nm – 1700 nm. Transparency in the visible range is not required.

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Dosage calculation	The tracer area concentration ρ_{tr} relates to the dry weight of the ink $\rho_{ink,dry}$. Tracer does not evaporate with the solvent. Thus, the tracer dosage in g per kg ready-to-print (visco) ink c is given by $c\left[\frac{g}{kg}\right] = \frac{\rho_{tr}\left[\frac{\mu g}{cm^2}\right]}{\rho_{ink,visco}\left[\frac{g}{m^2}\right]} \cdot 10 = \frac{\rho_{tr}\left[\frac{\mu g}{cm^2}\right]}{\rho_{ink,dry}\left[\frac{g}{m^2}\right]} \cdot s \cdot 10$ where s is the solids content of the ready-to-print ink and $\rho_{ink,visco}$ is the application weight. Note that the dry weight or application weight must be calculated with respect to a full coverage print.
Quality control	Correct tracer concentration should be verified in advance by measuring fluorescence intensity on test prints made with the same ink, printing machine and cylinder intended for use.
Restrictions on use of tracers	Do not use tracers in combination with carbon black pigments or metallic inks. Areas overprinted with or containing a share of carbon black must be considered as non-traced.

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4. FAQ BEYOND THE BOUNDARIES OF THE ABOVE DFR GUIDELINE

Table 2: FAQ beyond the boundaries of the guideline

FAQ	Answer	Remarks
Will the TBS work for top layers thicker than 25 µm, or BOPE rather than MDOPE?	It is expected to do so, assuming the top layer remains transparent in the NIR range.	
Which printing techniques can be used?	Gravure printing has been verified in the project. Flexo and offset have been successfully tested before, but not evaluated to the same extent. Inkjet printing is not usable.	
Can other ink colors than white and yellow be used to incorporate the tracers?	Yes, as long as absorption in the NIR range is not too high. Many pigments, even darker ones in the visible range, have high transparency in the NIR range and are therefore usable. Smaller degrees of absorption can be compensated by increasing the tracer concentration. Backing the tracer layer with a white layer can increase intensity significantly. Chemical compatibility of tracers is expected to be given with all pigments.	Characterization of the optical properties of the ink is recommended beforehand. Individual adjustment of the tracer concentration necessary.
Do I need to apply an extra printing step for the tracer color?	No, the tracer can be incorporated in one of the existing layers of the design print, as long as the above guidelines are fulfilled.	
Can the tracers be incorporated in a transparent layer, e.g. overprint varnish?	Yes. However, a white backing layer – either print or white-colored polymer – is recommended to ensure sufficient tracer signals.	Incorporating tracers in the outermost layer is not ideal. A protective layer on top of the tracer layer is recommended to prevent abrasion.
Will the tracer alter the optical properties of the ink?	Not to be expected under usual conditions.	For very high-gloss surface finish, pre-tests are recommended.
I want to use a print layout with finer structures than the minimum tracer area stated above. Is this possible?	Small features will probably not be detectable but will have otherwise no negative effect. Consider all layout parts with small features as	

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	non-traced. Make sure to include enough areas compliant with the guidelines.	
I want to use a print layout with halftone printing/mixed colors. What should I consider?	To maintain the minimum tracer area concentration as stated in the guidelines, increase the tracer-in-ink concentration accordingly (if possible) to compensate for lower ink coverage. If carbon black or carbon-black-containing pigments are mixed in, the respective areas should be considered as not detectable.	Note that the tracer concentration does not necessarily scale linearly with ink coverage, because for printing cylinders with smaller cell sizes the transfer of tracer particles may be significantly reduced. Individual testing and adjustment necessary.
Can I embed the tracers in the PE matrix of the film (i.e. as sort of a masterbatch)?	Technically, this is a feasible and proven process. Preferred usage e.g. in applications where the tracer should remain in the material during recycling to enable later recognition. We do not recommend it for packaging recycling applications as it goes against the intended use of TBS to sort out packaging efficiently in dedicated streams and remove the tracers subsequently in the pre-treatment/recycling process.	

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5. KEY LEARNINGS DE-INKING AND DELAMINATION

Printing ink is a crucial component of flexible plastic packaging, offering several functional benefits. They provide essential information, such as details about the product's composition, the presence of allergens, and nutritional content. Additionally, inks enhance the visual appeal of packaging, making it more appealing to consumers for marketing purposes. While inks are an essential component of flexible plastic packaging, they pose a significant challenge in plastic recycling. When printed plastic films are processed together, the resulting recyclates often suffer from contamination, leading to low-quality materials. This limits their usability, making them suitable only for downcycled products rather than high-value applications. Furthermore, during reprocessing, residual ink can decompose and release gases, leading to the formation of rancid odors and a decline in the physical properties of the recycled material. Scientific proof for this was recently delivered by a Maastricht and Ghent University joint paper[4].

Key learnings number one and two remains thus:

1. ***Closed loop recycling of printed flexible packaging requires 'advanced mechanical recycling', which includes technologies such as delamination/de-inking or dissolution recycling.***
2. ***Based on these advanced washing solutions that delaminate, combined with tracing technologies also studied in the CFP project, even certain types of multilayer structures can be recycled in closed loop.***

Furthermore, what we have seen is that in case of uncertainties about the quality, converters/brand owners may 'hide' the recyclate in between other layers and/or add new barrier concepts. This leads to a third learning:

3. ***The quality of the recycling process should be high in the first case. If the first recycling cycles produce materials that have quality problems, including barrier/color/smell/safety issues, there will be a trend to cover these recycled content layers with more complex structures which will complicate the next recycling cycle (including processes such as delamination/de-inking become more complicated on such structures).***

Related to the recycling process itself, within the CFP project, de-inking and delamination process optimization was carried out at lab scale prior to large-scale processing. What we have seen is that depending on the application purpose, the type of inks and polymers used in flexible plastic packaging varies. This complicates the selection of a universal medium for efficient de-inking and delamination, especially given the heterogeneity of post-consumer plastic packaging. For example, water-based de-inking media proved ineffective for de-inking and delaminating PE-based monomaterial multilayers laminated with conventional adhesives. To address this issue, the project incorporated a delamination primer into the multilayer flexible packaging, which enabled effective de-inking and delamination. Key learnings number 4 and 5 are thus:

[4] 10.1016/j.jhazmat.2024.134375

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4. ***Water based delamination/de-inking procedures (typically 85°C, with 2-5% NaOH and detergents), have their limitations and fail in delamination and de-inking of all common samples in flexible packaging. These water based delamination/de-inking procedures are typically used in direct printing / surface printing where the wash media can easily get to the ink components. Although these direct printing / surface printing are applicable in a wide window of products, they are not applicable to any types of products that require pasteurisation or sterilisation by steam (the ambient ready meal, wet pet food, etc. market)***
5. ***Debondable adhesives or delamination primers do allow delamination and de-inking in such aqueous medium, yet experiments have shown that those water-based solutions still do not allow deep cleaning of the polymers (meaning enhancing diffusion of components from the core of the plastic to the medium). Solvent-based delamination and de-inking processes were able to clean the polymers deeper and even repair defects from the water-based process (e.g. staining problems).***

Yet, we were limited in this project to focus on water-based delamination and de-inking, given the fact that for upscaling only water-based infrastructure was available. Thus:

There is too little (pilot) infrastructure for solvent-based washing/delamination and de-inking.

Related to optimisation of both water or solvent-based delamination and de-inking processes; in addition to medium selection, the process conditions such as particle size, temperature, solid/liquid (S/L) ratio, reagent concentration, and contact time are critical to the efficiency of de-inking and delamination, significantly affecting the quality of the recovered material. For example, it was observed that increasing temperature and particle size led to curling of the flexible plastic packaging, which hindered effective contact between the material and the liquid medium. In fact, higher temperatures also accelerate de-inking and delamination of the material. As a result, the process conditions were optimized to achieve a high de-inking rate while avoiding material curling. Regarding reagent concentration, low NaOH concentrations (<2%) proved ineffective for de-inking and delamination at large scale. This issue was addressed by increasing the NaOH concentration in the process. The solid-to-liquid (S/L) ratio is also a critical factor in improving the efficiency of the de-inking and delamination process. A high S/L ratio can lead to oversaturation of the medium, which decreases its de-inking effectiveness. On the other hand, a higher S/L ratio increases friction between the materials, thereby enhancing delamination. Therefore, the S/L ratio was optimized to improve the quality of the recovered material. Contact time with the liquid medium is another parameter that requires optimization based on the composition of the input material. For instance, the delamination of triplex multilayer flexible plastic packaging takes longer than that of duplex material. Therefore, ensuring sufficient contact time with the liquid medium is crucial for achieving an efficient process.

6. ***The CFP project has delivered evidence on which parameters are crucial for proper delamination and de-inking, including practical aspects that need to be mitigated such as curling of the film. Somehow surprisingly, we have also found that some parameters like residence time and temperature should not per se be 'the highest and longest', therefore,***

all parameters have an optimum somewhere in between, which requires good knowledge of the process to apply it properly.

A large portion of the attention in this project went to the problem of staining/bleeding. Indeed, during the de-inking process, the reversible adsorption and desorption of colorants occur. Depending on the colorant's affinity for the plastic packaging, its concentration, and the S/L ratio used, re-adsorption of the colorants onto the packaging can take place, leading to color staining on the material. Experimentally, we have found that after de-inking and delamination, LDPE flakes could still contain some ink (slight quantity) which led to contamination of the equipment (densifier and extruder) due to the release of ink substances present in the PE matrix. Also, a sufficient degassing is really necessary during extrusion as VOC compounds with high odor smelling can be released during extrusion.

Therefore, effectively stabilizing or removing colorants from the liquid medium is essential to prevent colour staining on the recovered material. Within the CFP project, a de-inking process was developed using scavengers in the liquid medium. These scavengers dissolve in the liquid and stabilize the dissolved colorants, thereby reducing their concentration in the medium. This helps to prevent the re-adsorption of colorants, thus avoiding colour staining.

7. ***A good de-inking process should have mitigation of staining/bleeding issues. In CFP several solutions (adding scavengers or better reactor design) were proposed for the first time.***

Finally, one should be aware that advanced chemical wash systems such as delamination and de-inking are not standalone solutions. Post-consumer flexible plastic packaging waste also contains various physical impurities coming from the use phase, such as dust, grease, and food remnants. To prevent these contaminants from saturating the medium and to enhance the de-inking capability of the medium, a dry-cleaning step (e.g. such as wind sifting) and pre-washing the waste material is crucial. Within the project, it was observed that if coffee grains were not effectively removed before the de-inking process, the efficiency of de-inking decreased, negatively impacting the quality of the recovered material. Additionally, it was observed that extracts from these grains could absorb to the films and cause fuming in the later recycling and production process. Similarly, it was noted that post-washing the material after the de-inking process is also essential to remove any remaining colorants and reagents from the surface of material.

8. ***Delamination and de-inking is one block that can truly contribute to closed loop recycling of flexible packaging, probably best with some more 'chemistry' in the medium such as solvents/scavengers/etc., but it is not on standalone miracle solution. It should be well placed in a whole recycling chain, starting from collection, then going to sorting (preferentially with tracing), then to pre-washing to remove most of the impurities, and only then to such a de-inking. Even after the core de-inking a post-wash is necessary and eventually other solvent-based processes or extrusion/depolymerization steps may follow.***

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6. CONCLUSION

From its semi-industrial trials (Loop 3), CIRCULAR FoodPack has distilled the first ever Design Guidance for TBS of PE-based flexible printed food packaging. This Design Guidance is complimentary to existing design for recycling guidelines in industry. The Guidance is further complimented by some FAQ addressing very practical questions related to the printing with tracers.

Furthermore, via this deliverable, CIRCULAR FoodPack discloses novel key learnings on recycling with de-inking and delamination to industry and academia alike, for them to benefit from the project's trials.

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