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# Upcycling of Contaminated Post-Industrial Polypropylene Waste: A Design from Recycling Case Study

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**This paper presents a detailed case study on the effective upcycling of a post-industrial plastic waste stream to a renewed compound, fit for re-use in a new application. The material investigated was a PET-contaminated recycled PP, destined for a high-impact, medium-stiffness application. After two research trials and one large-scale industrial trial, an upcycling formulation was determined to bring the recyclate to the required level of the new application, thus closing this specific material loop within the case company. The used methodology adheres to the *Design from Recycling* principle, in which industrially available (mechanically) recycled polymer materials are matched to potential new products. The design strategy starts from either the properties of an available recycled polymer (and then defines the product) or from the functional boundary conditions of the product (and then selects the material and/or an optional material upcycling step). The *Design from Recycling* principles can elegantly be combined with those of *Design for Recycling*, within the framework of a Circular Economy.**

## INTRODUCTION

Recycling of polymers is an ever-expanding field of industrial interest, especially given the ambitious targets set in the recent Circular Economy Package [1] and its subsequent action plans, which foresee a common EU recycling target of 65% of all packaging waste by 2025 (and 75% by 2030), including a 55% recycling target specifically for plastics packaging.

Mechanical recycling, in which the solid plastic waste is sorted, washed, shredded and re-processed as flake or granulate, is the most ubiquitous method for the recycling of plastics. The alternative of chemical recycling is under strong development, but not in widespread industrial use at this time [2].

Intake material to the recycling process can largely be divided into post-industrial (or ‘pre-consumer’) and post-consumer materials. Typically, post-industrial recyclates have the following advantages over their post-consumer counterparts [2]:

- I. The materials are clean, meaning uncontaminated by organic waste and pollutants like wood, paper or other plastics;
- II. Quite often, these are mono-materials. But even if this is not the case, then:
- III. The composition is usually known, both in terms of the composing polymer(s) and their amount (in the case of multi-material products).

The recyclate under investigation in the current research is a post-industrial material. It is not a mono-material, but it is clean and the composition is known.

Multi-material polymer recyclates, when reprocessed via extrusion (or injection molding), are in fact polymer blends, which pose their own challenges in terms of processability and properties when unmodified. Thermodynamically speaking, the composing polymers will not mix [3, 4]. This is particularly the case for our material combination of polar polyethylene terephthalate (PET) in apolar polypropylene (PP). The polar PET will form large spheres within the PP matrix, thus reducing mechanical properties, impact strength foremost amongst them [5, 6]. Common methods to mitigate these effects include the addition of impact modifiers [7], which have a toughening effect when added in sufficient amount, or the more expensive compatibilizers [8]. Compatibilizers are typically grafted (co-)polymers, in which functional groups like maleic acid anhydride (MA) or glycidyl methacrylate (GMA) are grafted onto a polymer, compatible with the matrix (like PP) [5, 9, 10]. Some compatibilizers backbones are rubbers themselves [11, 12], thus adding the functional effect of an impact modifier. Typically, the addition of impact modifiers or rubber-based compatibilizers will result in increased toughness and impact resistance, at the expense of properties like stiffness and strength [5].

The goal of the current study is to effectively recycle a post-industrial PET-contaminated PP polymer (furthermore referred to as the recyclate) into a high-impact consumer product, following the *Design from Recycling* method.

## DESIGN FROM RECYCLING

The design of plastic products has a large impact on both their recyclability (at end-of-life, EoL) and the degree to which they can incorporate recycled materials (at start-of-life, SoL).

*Design for Recycling* is, via the Ecodesign Directive [13], heavily promoted by the European Commission within the framework of the Circular Economy. It is a well-known product development strategy in which new products are developed so that they can be recycled at their EoL. It entails easy separation of different materials and an all-round efficient material use [14]. The strategy is part of a virgin material’s SoL.

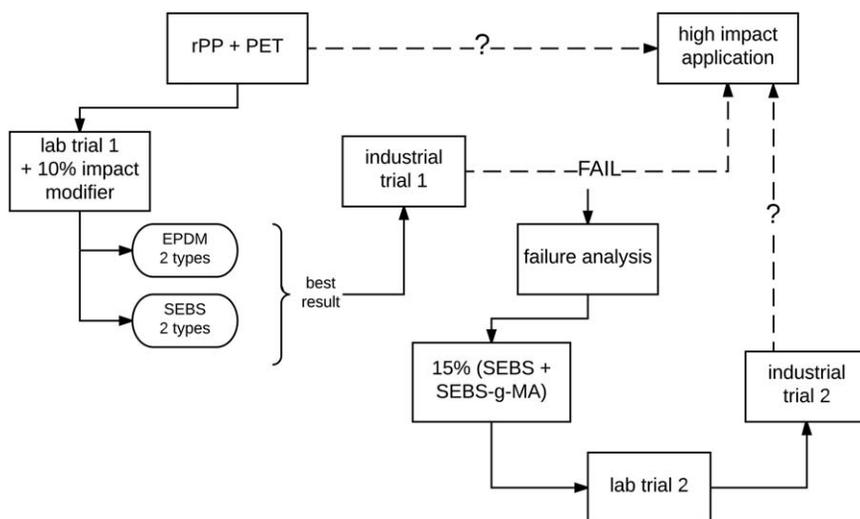


FIG. 1. Schematic of the case study.

In the European Commission's latest Circular Economy Package (CEP), it was proposed to make mandatory a 'product design...to make it easier and safer to dismantle, reuse and recycle electronic displays' [15]. It is expected that other product categories will follow. Additionally, Design for Recycling is encouraged via the implementation of Extended Producer Responsibility (EPR) schemes, wherein the EoL costs will factor as an economic incentive to producers [15].

Design for Recycling, however, only covers the EoL in terms of potential recyclability.

The authors would like to advocate also considering design at the product's EoL and this is where Design from Recycling comes in [16, 17]. In Design from Recycling, the secondary raw material originating from the recycled polymer waste of a previous product's EoL is the starting point of a new product development. Design from Recycling involves the following key aspects [16]:

- Identifying the recycled polymer's strengths and weaknesses through extensive characterization;
- Matchmaking between the recycled material's characteristics and potential (new or existing) products;
- Adapted product (and mold) design for manufacturing of the products in recycled polymers;
- Identifying acceptable (cost-effective) strategies for the upgrading of the material quality (to product requirements) where necessary. This usually involves small amounts of additives like stabilizers or compatibilizers;
- Through life cycle analysis and life cycle costing, quantifying the overall resource efficiency of the whole process, thus ensuring the best possible use of the recycled polymers as well as demonstrating to the broader public the gain that is to be had by using these secondary material sources.

Design from Recycling does not need to be closed-loop. It is perfectly valid – and often necessary – to valorise a recycled material outside its original product application. For example, this could be because a packaging material cannot (regulatorily) be re-used in a food-contact application or because the recycled material has an entirely different composition and properties set

compared to the virgin (e.g. recycling of multilayers, which become blends).

Design for and from Recycling are in fact complementary strategies that, when applied together, can truly bring a material full-circle.

## CASE STUDY – MATERIALS AND METHODS

### Setup of the Case Study

An overview of the case study is made in Fig. 1.

The recycled material is a post-industrial PET-contaminated extrusion-grade PP (discussed further below). The material is meant to be side-cycled into another product of the same company, which is referred to as a 'high impact application'. Boundary conditions for the recycled material to go to the company's selected new product were defined as follows:

- The polymer must be sufficiently viscous for sheet extrusion. As an industrial quick-referral property for this, MFI values are used (even if full viscous analysis would give a more complete view). An indicative upper limit is set at 10 g/10 min (250°C, 2.16 kg);
- A minimum flexural modulus of 900 MPa is required, as well as a flexural strength of minimum 25 MPa;
- A minimum Charpy notched impact strength of 8 kJ/m<sup>2</sup> (22°C) is required.

An analysis of the base recycled material showed that MFI and flexural mechanical properties were sufficient, but that impact resistance was clearly lacking (discussed further below). Therefore, an impact modification additive was to be compounded. This does not add an extra processing step to the recycling process: as the recycled material is a low-density flake; regranulation is required anyway. During the first stage of the case study, four different impact modifiers were pre-selected. 10 wt% of each of these additives was compounded into the recycled blend on a lab scale and all series were tested for flow, flexural and impact properties. Two types of ethylene propylene diene monomer (EPDM) and two types of styrene-ethylene-butene-styrene (SEBS) were tested. The EPDM rubbers were

TABLE 1. Nomenclature and composition of the different blends.

Name	BASE (wt%)	Additive (wt%)	Additive(s)
BASE	100	-	-
EPDM1	90	10	Nordel IP 3722P (Dow)
EPDM2	90	10	Novalene 7300P (NOVA polymers)
SEBS1	90	10	KRATON G 1645 M polymer (Kraton)
SEBS2	90	10	KRATON G 1657 M polymer (Kraton)
SEBS2+CA	85	12.5 + 2.5	12.5%: KRATON G 1657 M polymer (Kraton) 2.5%: KRATON FG 1901 G polymer (Kraton)

selected because they are generally cheaper and the compatibility with the PP matrix might be higher, based on the PP content in the backbone (additionally to the ethylene content, which is present in both types of impact modifier) [18]. The SEBS rubbers were selected because the styrene groups introduce a certain polarity, which has the potential to interact better with the PET contamination [5].

One impact modifier, which provided the best combination of flexural and impact properties, was selected as the additive for a large-scale industrial trial. This was one of the SEBS rubbers.

Roughly, 500 kg of the recyclate was industrially compounded with 10wt% of this impact modifier and then processed by the company (through extrusion and thermoforming) into samples of the new product. These samples, however, unexpectedly failed the drop test to which they were submitted. Both extruded sheet and failed parts were subjected to a failure analysis, the conclusions of which led to a second lab trial in which the rubber phase was increased to 15 wt%. Failure analysis also indicated that the inadequate toughness may have been caused by the lack of compatibility between the PP matrix and the dispersed PET contamination. Even if previous lab-scale testing had shown no negative effect of the PET on the properties of the ‘as is’ mix, it showed to be relevant when creating the ternary blend with SEBS. Therefore, half of the additional 5wt% rubber component was not the pure SEBS, but a compatibilizing agent (CA), consisting of a similar SEBS backbone, grafted with a functional maleic anhydride (MA) group, further referred to as SEBS-g-MA. This new blend formulation was subjected to similar tests in a second lab trial, the results of which were sufficiently impressive that the blend was approved for a second industrial trial.

### Materials

The base material is a post-industrial polypropylene (recyclate) with 2 to 4 wt% PET contamination provided by a local plastic converter (Samsonite, Oudenaarde, BE), in the form of flakes. It is the matrix for five formulations with impact modifying additives, one of which includes also a CA), described in Table 1.

The impact modification additives used are:

- I. EPDM1: *Nordel 3722P* from Dow with a 71% ethylene content, a density of 0.88 g/cm<sup>3</sup> and a Mooney viscosity of 18 MU;
- II. EPDM2: *Novalene 7300P* from NOVA polymers with a 60% ethylene content, a density of 0.915 g/cm<sup>3</sup> and a Mooney viscosity of 60 MU;

III. SEBS1: *G 1645 M* from Kraton, with a density of 0.89 g/cm<sup>3</sup> and 13% polystyrene (PS) content. Reported MFI is 3.5 g/10 min at 230°C and 2.16 kg;

IV. SEBS2: *G 1657 M* from, with a density of 0.89 g/cm<sup>3</sup> and a 13% PS content. Reported MFI is 8 g/10 min at 230°C and 2.16 kg;

V. SEBS2 + CA: 12.5 wt% of SEBS2 and 2.5 wt% SEBS grafted with maleic anhydride from Kraton (FG 1901 G) with a density of 0.92 g/cm<sup>3</sup> and a 30% PS content and 1.4 to 2% grafted MA. Reported MFI is 5 g/10 min at 200°C and 5 kg.

### Experimental

**Lab-Scale Compounding and Processing.** The base recyclate material was not dried prior to processing. The additives dried at least 4 h at 60°C before manual mixing with the base material and compounding to moldable granules on a Coperion ZSK18 co-rotating twin-screw extruder at 260°C and 300 rpm. Injection molding into rectangular test bars (3 × 13 × 126 mm<sup>3</sup>) was done on an Engel 80 ton injection machine at 260°C, with an injection speed of 60 mm/s, a (specific) holding pressure of 680 bar for 15 s and 25 s cooling time.

**Physical and Mechanical Testing.** Test bars were conditioned at room temperature for minimum two days prior to testing in a climate-controlled room (50% R.H., 22°C). All mechanical tests were performed on injection-molded parts.

The melt flow rate (MFI, g/10 min) was monitored according to ISO 1133 (2005) at a temperature of 250°C, and load of 2.16 kg on a Zwick plastometer 4100 instrument, based on the mass-measurement method. The higher temperature of 250°C was selected to allow all PET contaminations to melt completely.

Ten test bars were notched 2 mm and tested for Charpy — impact strength according to ISO 179-1/1eA (2000) at 22°C on a Tinius Olsen apparatus (model Impact 503) with a 2 Joule pendulum and testing speed of 2.91 m/s.

A three-point bending test, according to ISO 178 (2010) standard, was performed on an Instron 4464 flexural testing machine, with Bluehill software (vs 2.6). An Instron static load cell of 2kN was used. The crosshead was set to a speed of 2 mm/min until 15 mm flexural displacement.

The flexural modulus  $E_f$  was determined as a segment modulus in the linear region, between 0.2 and 0.9% strain. Flexural stress  $\sigma_f$  was calculated as the highest occurring stress.

Where relevant, differential scanning calorimetry (DSC) was conducted on samples of about 20 mg. Apparatus used is a Netzsch 204F1, under inert nitrogen atmosphere. Two

TABLE 2. MFI and mechanical properties for the blended materials.

Blend	MFI (g/10 min)	$E_f$ (MPa)	$\sigma_f$ (MPa)	Impact (kJ/m <sup>2</sup> )
BASE	4.7 ± 0.2	1474 ± 20	39.0 ± 0.4	3.6 ± 0.3
EPDM1	4.3 ± 0.3	1233 ± 11	33.8 ± 0.2	8.2 ± 0.6
EPDM2	5.5 ± 0.2	921 ± 35	28.8 ± 0.8	4.4 ± 0.6
SEBS1	5.6 ± 0.1	789 ± 57	27.4 ± 0.5	6.6 ± 0.6
SEBS2	5.9 ± 0.3	1072 ± 21	30.7 ± 2.3	10.0 ± 0.9

consecutive cycles of heating and cooling are run at 10K/min, between the temperatures of 25–300°C.

All data were run through a statistical software program, SPSS Statistics 24 and screened for extremes. The extremes were deleted in order to work within the 95% confidence interval. All results are reported as mean ± standard deviation (S.E.) of minimum five measurements.

#### Pilot-Scale Industrial Compounding and Processing.

Selected materials were compounded industrially by QCPolymers (Netherlands) at 260°C on a 60 mm co-rotating twin screw with a screw speed of 260 rpm, at throughput of 50 kg/h.

Compounds were transported back to the first company, extruded (230°C) into sheets at a thickness of 2.75 mm and subsequently thermoformed into the intended high-impact product. Finally, they were subjected to a proprietary drop test (specific to the product) at room temperature.

**Morphological Evaluation.** The samples from the industrial trial and the second lab test were immersed in liquid nitrogen and consequently fractured. Then, they were submerged for 30 min in tetrahydrofuran (THF, Biosolve Ref. 0020220602BS unstabilized) at 60°C with the purpose of etching out the SEBS phase.

The broken and etched samples were sputtered with gold by a Baltec SCD005 sputter coater. Micrographs were obtained on a SEM instrument FEG SEM JEOL JSM-7600F 202, with an accelerating voltage of 20kV.

All images presented in this manuscript are taken at a magnification of 5000x, the white scale bar is 1  $\mu$ m.

## CASE STUDY – RESULTS AND DISCUSSION

### Impact Modifier Selection

The results of the first lab trial, meant for the selection of the impact modifier for the industrial trial, are summarized in Table 2.

Small changes in MFI are observed which are consistent with the properties of the additives used. All tested materials stay well below the imposed functional limit of 10 g/10 min. Likewise, all materials meet the imposed requirement for flexural strength. For the EPDM rubbers, it is noteworthy that the addition of EPDM1 results in a significantly higher strength and stiffness than for EPDM2, as well as providing a better toughness. This is attributed, via the higher ethylene content of EPDM1, to a better crystallinity of the PE phase [19, 20]. This hypothesis is corroborated by a DSC analysis of the blends (curves not shown), in which a (small) separate PE crystalline melting peak is more clearly observed for the blend with EPDM1 than for the EPDM2.

Regarding the SEBS additives, SEBS2 is clearly a better fit with the BASE material; all considered mechanical properties are higher. SEBS1 was indeed reported to contain a softer rubber segment [21], leading to lower strength and stiffness. It is somewhat unexpected, to then observe also lower impact strengths for the use of this rubber. However, SEBS2 does have a lower viscosity, which will allow it to break up more easily into well-dispersed smaller domains [3], which in turn is expected to lead to better toughness properties. It has been well documented that ligament distance (or ligament thickness) is a determining factor for the toughness of rubber-enhanced blends [22, 23]. The critical ligament thickness is determined by the matrix; for PP this has been found to between 0.1 and 0.8  $\mu$ m [24–26].

As modulus and impact strength are the primary properties upon which further selection is based, these are plotted in a materials selection graph for all blends in Fig. 2. From this, it is easily observed that SEBS2 offers the best combination of resulting properties. It is the only additive to clear the impact strength prerequisite comfortably. On average, EPDM1 - which does have a higher stiffness—passes the lower limit of 8 kJ/m<sup>2</sup>, but only just and not with its entire 95% confidence interval. The criterion of a minimum modulus of 900 MPa is also met by SEBS2, if only just (1072 ± 21 MPa).

Therefore, despite the higher price, SEBS2 is selected as the additive for the consequent industrial trial.

### Industrial Trial and Failure Analysis

The compounded batch was used for the manufacturing of the intended high-impact component, through extrusion of sheets and subsequent thermoforming of these sheets into the final product. The prototypes thus created were submitted to a company-proprietary drop test, in which materials with the specifications mentioned under 3.1 are not expected to fail, according to internal standards. However, the parts did fail.

Materials were returned for failure analysis. This includes the compounded granulate, extruded sheets and thermoformed product. The granulate was injection molded into test bars, which is

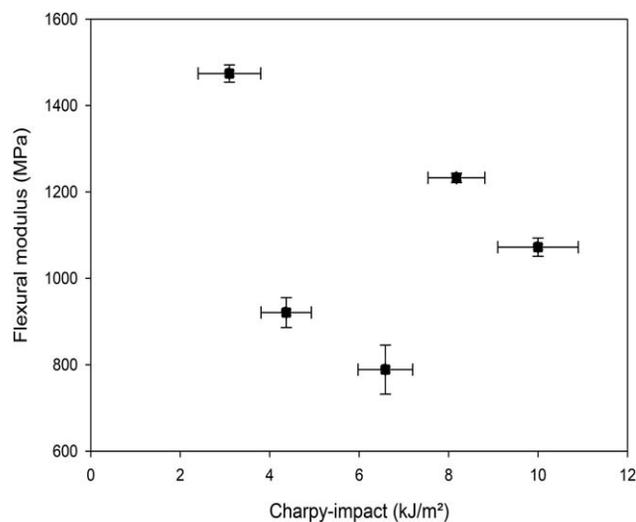


FIG. 2. Flexural modulus vs. Charpy Impact strength for the base material and the different blends with 10 wt% impact modifier.

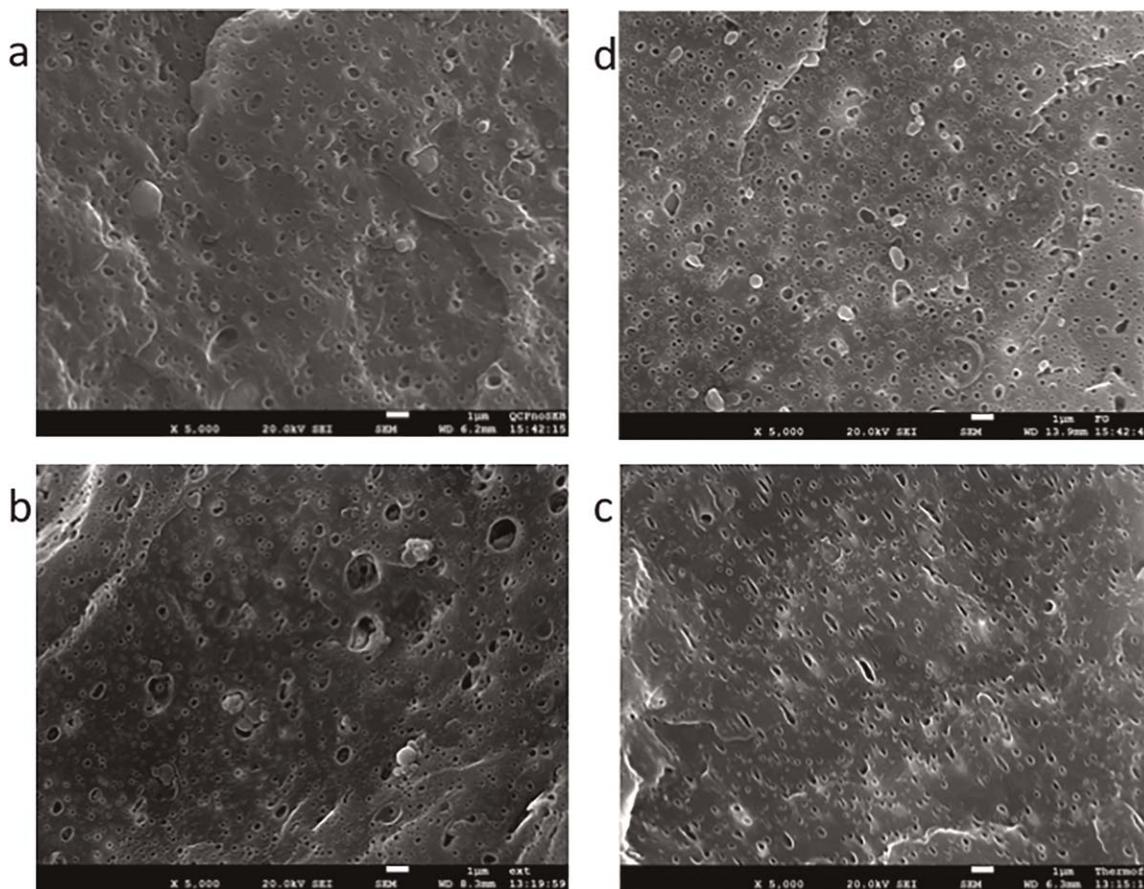


FIG. 3. SEM images (5000x, white scale bar is 1  $\mu\text{m}$ ) of (a) injection moulded SEBS2, (b) extruded SEBS2, (c) extruded + thermoformed SEBS2 and (d) injection moulded SEBS2 + CA.

the form on which properties were tested in the first lab trial. In the failure analysis, primarily the effect of the different processing steps was investigated. To this end, samples from each step (compounding, compounding + extrusion, compounding + extrusion + thermoforming) were examined via SEM imaging to evaluate the dispersion of the rubber phase. Images of all three samples are shown in Fig. 3a–c. The SEBS phase was etched out during sample preparation. Figure 3d is to be disregarded for this part of the discussion.

In Fig. 3a, it is observed that, coherent with the good impact properties obtained in the first trial, the SEBS is well dispersed into small domains. Spherical SEBS zones vary in diameter, but mostly remain below 1  $\mu\text{m}$  in diameter. Distribution as well, is observed to be very good, resulting in a homogeneous patchwork with small ligand distances, typically also within the critical range of 0.1–0.8  $\mu\text{m}$ . Both the small sizes of the domains and the small ligand distances are known to contribute significantly to the toughness of the blend [22, 23].

The contaminating PET phase can also be observed as remaining (not etched out) spherical domains within the PP matrix, with sizes that range from well below to well over 1  $\mu\text{m}$ .

Figure 3b shows the morphology after sheet extrusion. While compounding and injection molding are high-shear processes, sheet extrusion is a low-shear process. This allows the previously well-dispersed SEBS to coalesce once more into larger

domains [27–31], as can be observed from Fig. 3b. Many of the small domains remain present, but new large (etched out) spheres are clearly seen as well. Distribution and dispersion of the PET phase appear to be unaffected by the extrusion step. This decreased degree of dispersion for the impact modifier will negatively affect the impact strength of the material [22]. The adverse structural effects of the processing are further developed during the thermoforming step, as can be seen in Fig. 3c. During the thermoforming process, the spherical SEBS domains are elongated into fibril-like ellipsoids. Such a deviation from the spherical shape will further reduce toughness of the blend [22].

Hence, it is concluded that the effective impact properties of the material have been diminished from those of the original compound, due to the structural changes induced by the different processing steps.

No obvious remedies on the level of the processing itself present themselves. Therefore it is decided to further improve the toughness of the original blend, so that it is more robust to the change in properties throughout suffered by the processing into the final product.

#### Final Formulation Selection

A potential final formulation is selected, based on the following two arguments: (i) an increase of the total amount of rubber will further reduce ligand thickness and toughen the blend.

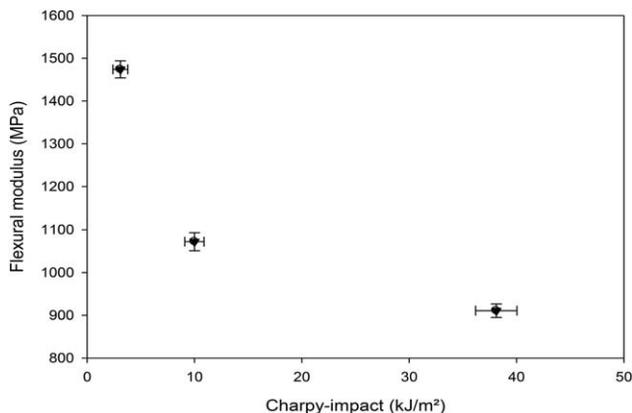


FIG. 4. Flexural modulus vs. Charpy Impact strength for the base material (BASE), the blend with 10% SEBS2 (SEBS2) and the blend with 12.5 wt% SEBS2 + 2.5 wt% SEBS-g-MA (SEBS2 + CA).

However, the SEBS2 blend only just met the criterion for stiffness, so the additional amount of rubber phase cannot be too high. A new percentage of 15 wt% is selected. (ii) The PET phase does not appear to be well dispersed into the matrix. The SEBS backbone by itself is not able to act as an adequate CA for the PET, based only on the potential interactions with the styrene blocks. Therefore, we opt to include in the 15wt% SEBS a small amount of 2.5wt% CA, being a SEBS-g-MA, which is expected to effectively compatibilize the PET. MA has been found to be a good CA for PET in a PP matrix [5]. Additionally, a CA will increase the interfacial adhesion between the PP matrix and dispersed PET phase, thus improving impact properties as energies can now be transferred more effectively between the two domains [4].

The blend is named SEBS2 + CA and test parts are prepared as before. In this second lab trial, the flexural properties of the new SEBS2 + CA blend are found to be:  $E_f = 911 \pm 16$  MPa and  $\sigma_f = 31.7 \pm 0.5$  MPa. Impact strength was a spectacular  $38.1 \pm 1.9$  kJ/m<sup>2</sup>. These results are compared to the BASE recycle and the SEBS2 blend in Fig. 4.

The morphology of SEBS2 + CA is shown in Fig. 3d, where it can be compared to that of the SEBS2 blend in the adjacent Fig. 3a. The dispersion of the (etched out) SEBS phase is of similar quality as previously observed for SEBS2. There is of course a larger amount of rubber present, resulting directly in more SEBS domains and a further reduction of the ligand distance. Given the large increase in toughness, it can be assumed that the ligand thickness has been sufficiently reduced to obtain the brittle-to-ductile transition of the blend [22, 32]. The effect of the CA, however, is also obvious from the much better dispersion of the PET phase. All observed PET particles are now under 1  $\mu$ m in size, mitigating also the embrittling effect of the non-compatible polar PET in the apolar PP matrix [5]. Both of these morphological effects lead to the dramatically increased impact strength, which is nearly quadrupled.  $E_f$  is now an average 911 MPa, which is only just sufficient for the pre-set lower limit of 900 MPa. As a safety factor and following the Design from Recycling principles, it is decided to adapt also the geometry of the final product, by increasing slightly the sheet thickness to compensate for the lower stiffness. This change does not compromise the function of the product.

The new formulation is thus approved for testing in a second industrial trial, with a small product design adaption (increase wall thickness) to compensate for the lower stiffness. It is expected that the CA will not only affect the PET, but will also lower the interfacial tension between the PP matrix and the dispersed SEBS phase during sheet extrusion, thus mitigating the previously observed coalescence of the SEBS into larger domains [33].

#### Final Industrial Trial

As shown in Fig. 2, the second industrial trial is still underway at the time of publication. While the final formulation was selected based on the convincing experimental evidence presented in this manuscript, we have also learned that the industrial-scale processes might affect these properties. It is therefore not yet guaranteed that this new formulation will effectively lead to the new commercial product. It remains to be seen if the properties of the compound will suffer due to either the compounding step or the sheet extrusion/thermoforming process.

To date, the industrial scale compounding has been effected and the properties of the compound have been verified to satisfaction. Resulting modulus is  $903 \pm 51$  MPa and impact strength is 44.1 kJ/m<sup>2</sup>.

The extrusion of sheets was also finished to satisfaction. The final steps of thermoforming and drop testing of the assembled products remain pending.

## CONCLUSIONS

In this manuscript, the basic principles of Design from Recycling are presented. By making recycled polymers fit-for-use in designated products, (re-)designing products specifically for the recycled polymers or a combination of both, new opportunities are created to valorise precious polymer waste.

A illustrative industrial case study has been presented, applying the Design from Recycling strategy to a contaminated recycle, meant for a new high-impact product application. On the material side, the recycle is upcycled with an additive formulation for impact modification and on the product side; the proposed product geometry is adapted to the effective properties of the upcycled blend.

During the industrial case, it was demonstrated that processing of blend materials has a significant impact on the product properties, via the induced morphological changes. It is important to be aware of these influences when predicting product properties based on standardized lab tests, so as not to mismatch expectations.

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