PolyCE

Post-Consumer High-tech Recycled Polymers for a Circular Economy

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DEM = Demonstrator, pilot, prototype, plan designs
DEC = Websites, patent filing, press & media actions, videos, etc.
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Dissemination Level: PU

PU = Public
CO = Confidential, only for members of the consortium, including the Commission Services

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Disclaimer

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1. Introduction

Whirlpool Corporation is the leading major appliance manufacturer in the world with approximately $20 billion in annual sales, 77,000 employees, and 59 manufacturing and technology research centres in 2019. The company markets Whirlpool, KitchenAid, Maytag, Consul, Brastemp, Amana, Bauknecht, JennAir, Indesit, and other major brand names in nearly every country throughout the world.

Whirlpool Corporation’s commitment to sustainability began half a century ago, in 1970, when we established our Office of Environment. Today we are accelerating our progress by delivering on our commitments to improve the environmental efficiency of our products and plants while making a positive impact on people and the planet.

Environmental insights from our materiality assessment and measurement processes helped guide our products, plants, and practices approach to focus on five key programs: Carbon, Plant Efficiency, Circular Economy, Design for Environment, and Sustainable Home Innovations.

Within the Circular Economy program, the update of recycled plastics is one key initiative and we made our Pledges to the EU Commission on the uptake of recycled plastics by 2025. We joined the PolyCE consortium to help delivering in our commitments.

1.1. Task description

PolyCE intends to introduce feasible innovations, both technologic and systemic, showcasing, through several demonstrators at industrial scale, circular economy solutions engaging all actors across the value and supply chain.

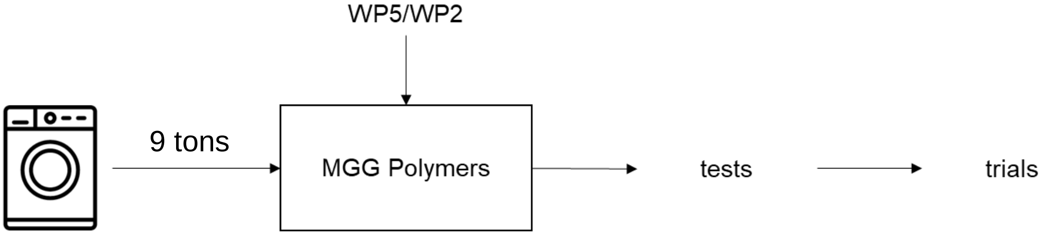
The main goal of Task 7.7 is to bring together the knowledge gained from other work packages into large-scale demonstrators for the effective re-use of PCR plastics from WEEE. This will result in individual parts and or products produced with these materials at the expected level of quality and performances.

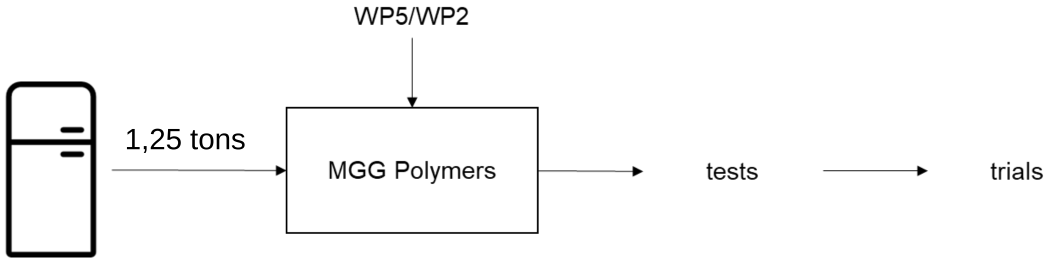
The scope includes performing multiple series of material tests on the selected PCR Plastics at the laboratories of the academic partners to characterize the key physical-mechanical performances and to subsequently perform large scale tests on the components / products at the facilities of the industrial partners or at their suppliers site.

Whirlpool will focus on components for washing machines made of PCR-Polypropylene ($CaCO_3$ filled), and potentially refrigerators components made of PCR-Polystyrene.

The demonstrator starts with the separate treatment of end of use appliances in dedicated ‘clusters’ such as washing machines cluster and/or Refrigerator cluster, or other sub-cluster that might raise needed according to the development of the previous tasks, to obtain 20 tonnes of plastics that will be sent to MGG Polymers for the recycling.

After multiple discussion it was decided that these quantities would not be needed to perform the required trials. So, in total 9 tons of plastics from the washing machine cluster and 1,25 tons of the drawer clusters were sent for treatment at MGG polymers. The plastic is recycled according to the specifications on PCR plastics in WP5 and the Design-from-Recycling strategy (WP2) will be tested in Whirlpool's laboratory (and/or at Academia's laboratories) for validation. The validation includes lab-scale material testing and in case of positive results, production-scale moulding trials.





Main challenges to be validated refer to the quality, performances (visual and physical) and legislative compliance of PCR plastics that should not compromise the whole product and component consumer requirements. Existing tooling and processes should not be changed, meaning the PCR plastic should be suitable for the use in current equipment. Additional benefits such as environmental and/or economic impact will be assessed during the project.

The demonstrator is designed to extensively cover stages such as product design, recycling and use of PCR plastics.

1.2. Task approach

In this section the approach applied to select i) polymer type and ii) component application is described.

Large Domestic Appliances (LDA) such as washers, dryers, dishwashers, refrigerators & Freezers, cooking appliances are made using a combination of steels/metals and plastics (both thermoplastics and thermosets) with an average % of plastics that go from 2%-30% depending on the specific appliance.

Within the thermoplastic fraction, the most used polymers are **Polypropylene** (mainly mineral filled) and **Polystyrene** (high impact) which are mainly used in washers and refrigerators respectively.

For those reasons, Whirlpool decided to focus the scope of the demonstrator's activity on:

- **Polypropylene $CaCO_3$ filled** used in washers to produce tubs;
- **High Impact Polystyrene** used in Refrigerators/Freezers to produce liners and door liners.

These two applications will cover approx. 35% of the total thermoplastic used by Whirlpool.

On the other end, these two components / applications pose a very challenging technical threat because of the following requirements:

1. Tubs have to support **high mechanical and chemical stresses** because of the high rotating spin of the drum and the presence of detergents. **Failure on the tub might impact the durability of the whole product**.
2. Liners / Door liners require **Food-Grade certification**.
3. Both components must be free of 'legacy substances' and be **RoHS** and **REACH compliant**.

In order to ensure the quality and reliability of the components/products selected Whirlpool decided to apply the very same technical requirements applied in the current production (using virgin materials), so neither the technical specification at material level, nor the performance requirements at product level have changed compared to the current baseline requirements

Moreover, in order to limit the potential capital expenses that might penalize the entire business-case on the use of PCR plastics, Whirlpool would like to adopt a 'drop-in' solution, meaning the PCR-plastic material should be processable with the existing injection-moulding tooling and within the current design. Recognizing this might pose additional challenges, we'll verify the feasibility of this specific requirement through the study.

2. Large Domestic Appliances - Washer tub application

2.1. Approach

Figure 1: Washer tub

Currently washing machines are collected and treated within the LDA cluster (Large Domestic Appliance) that includes washers, dryers, dishwashers, cooking appliances.

For this study, it was decided to narrow down the cluster, in order to limit the diversity of plastics and try to minimize (plastic) contamination coming from other appliance types. This maximizes the chances of obtaining a recycled filled-PP grade suited for production of washer tubs.

After discussion within the consortium it was decided to collect, treat and test the following clusters:

- Washers only (collection of only washing machines from the LDA waste stream);
- Washer tubs (collection of only washers drums from the LDA waste stream).

The collection and treatment of washing machines and tubs was performed by ERION in collaboration with its suppliers on the Italian territory. During the treatment phase, plastic flakes samples were collected and sent to KUL, MGG and UL for analysis. Each project partner performed a different set of analysis on the plastic flakes. KUL performed flake composition and colour analysis, MGG performed flake composition analysis and standard material properties and UL performed compliancy tests and physical material properties.

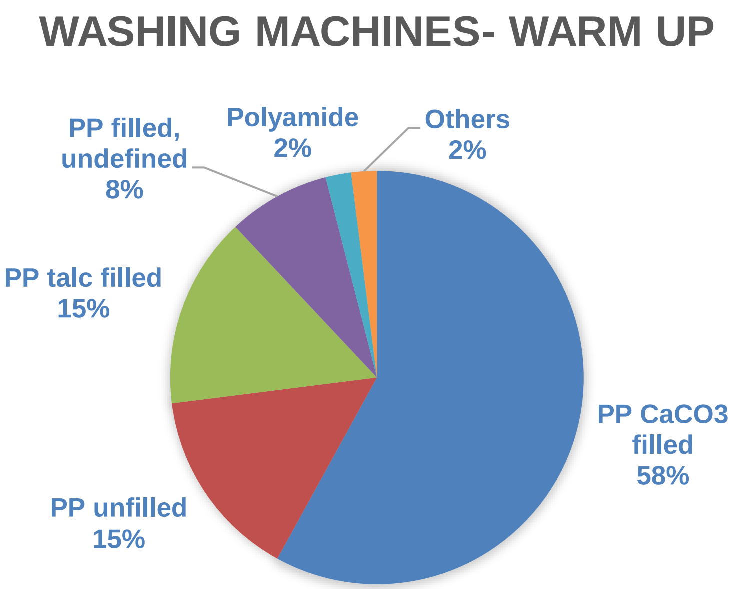
2.2. Material analysis (performed by KUL)

The analysis of the flake sample[1] composition resulted in:

Table 1: Flake sample composition washers and washers tub cluster

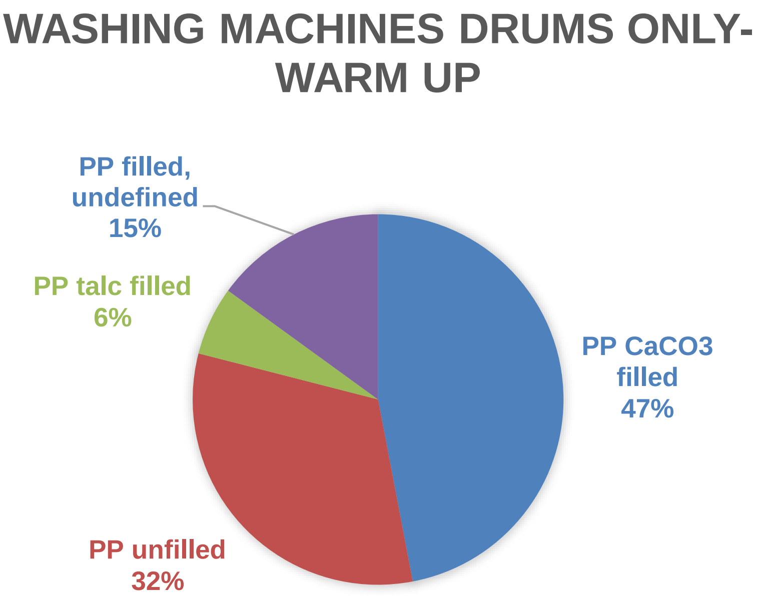
Material	Washers (wt%)	Washer tubs (wt%)
Plastics	98.4	99.9
Metal	0.3	<< 0.1
Rubber	0.8	0
Cables, PVC	0.1	<< 0.1
Glass, Concrete, Ceramics	0.1	0
Wood	0	0
Foam	0	0
Others	0.3	0.03

The plastic fraction analysed by the FTIR spectroscopy of 50 samples resulted in the following composition:



[1] Flake sample: mixed plastic flakes material sample obtained after pre-treatment of the cluster, most metals, ceramics and organics sorted out. More detailed information can be found in deliverable 7.1.

The FTIR analysis could distinguish between talc and calcium carbonate filled PP. However, the threshold levels are not determined by a calibration curve and therefore an uncertainty needs to be taken into account when interpreting the results. The PP filled, undefined samples could not be identified in detail, however it is very likely that it contains a mixture of talc and calcium carbonate as peaks of both minerals are present.



A higher amount of PP unfilled is observed in the ‘drums-only’ cluster compared to the entire washing machine samples. In addition, no plastic beside PP was found.

In conclusion: Because the results did not show a significant difference in the quality/contamination of the plastic fraction of the flakes after pre-treatment **it was decided to proceed with the cluster named ‘washers only’**. In this case it was shown that the extra time, labour and space needed to sort parts instead of machines is not justified with an increase of material quality. However, compared to current pre-treatment protocols an improvement was noted.

The input plastic flakes have been processed (washed, sorted and compounded) by MGG polymers into pellets. Plastic pellets (r-PP $CaCO_3$ filled) were shipped to UL for further analysis in accordance with Whirlpool’s request (RoHS, REACH and others).

More information on collection and pre-treatment steps is available in Deliverable 7.1.

2.3. Technical requirements

Materials level requirements

Whirlpool technical requirements (TDS) were shared within the partners involved in the demonstrator activities. More information can be found in the Appendix.

Compliance with Chemical Legislation - GES0084

This document (GES0084) is the Whirlpool Materials Compliance Specification that identifies materials and substances whose use is prohibited, restricted, or require disclosure by government regulations or by health or environmental concerns. Examples of substances listed in the document are Flame Retardants, plasticizers, blowing agent.

The use of the identified materials and substances require special consideration and continuing awareness in the manufacture, use, and disposal of Whirlpool products, and the materials, parts and components used to produce and service Whirlpool products.

This Specification contains a Restricted Materials List (RML) which identifies materials that have been restricted or regulated in the past, and those that are likely to be restricted or regulated in the future. It also reflects the fact that the choice of materials and substances in Whirlpool products can have far reaching significance on product safety, reliability, consumer product acceptance, end-of-life disposal options, and the environment. Whirlpool and every supplier of any article (finished product, part, component or Materials) must comply with the requirements of this Specification.

Washer tubs Product Level requirements

Tab 2 -Reliability tests

Table 2: Standard reliability test performed on washer tubs

Dry-spin test	To provide basic test guidelines with focus to evaluate the functionality of mechanical parts on the final application. They describe stressing conditions to accelerate the effects observed over the lifetime of the appliance on the field
Rinse & Spin test	
Washing Continuous cycle test	
Life-test	
Continuous spin accelerated life-test	

2.4. Material property testing

Tests performed at UL Lab - Physical Performances

Table 3: Physical properties recycled PP CaCO3

Property	**Test Condition**	**(n)ok**
Ash content (ISO 3451-1)	3h / 600°C	ok
Density (ISO 1183)	23°C	ok
Tensile Strength (ISO 527-2)	-30°C, 23°C and 50°C	ok
Flexural Strength (ISO 178)	-30°C, 23°C and 50°C	ok
IZOD Impact (ASTM D256A)	-30°C, 23°C and 50°C	ok
HDT (ISO 75)	120°C/h, 1.8 MPa and 120°C/h 0.45 MPa	ok
Melt volume rate (ISO 1133)	190°C, 210°C and 230°C	ok

Tests performed at UL Lab - Chemical Compliance

Table 4: chemical compliance of recycled PP CaCO3

Regulation (EC) n. 1907/2006 (REACH), Annex XVII, Point 50.5 (Aromatic Polycyclic Hydrocarbons)	Complies
Regulation (EC) n. 1907/2006 (REACH), Annex XVII, Point 62 (Mercury compounds)	Complies
Regulation (EC) n. 1907/2006 (REACH), Annex XVII, Point 63 (Lead)	Complies
Regulation (EC) n. 1907/2006 (REACH), Annex XVII, Point 23.1 (Cadmium and its compounds)	Complies
Directive 2011/65/EU on the restriction of the use of certain hazardous substances in electrical and electronic equipment	Complies
Regulation (EC) n. 1907/2006 (REACH), Annex XVII, Point 20 (Organostannic compounds)	Complies
Regulation EC n. 1907/2006, article 33: determination of SVHC within the components of the sample received	Complies

Lab testing of the rPP-CaCO3 filled showed great results compared to the virgin grades currently used, with no major difference in properties. This proves that clustering and targeted closed loop recycling of plastics can result in suitable materials without the need to upgrade the properties. On top of this compliance with current regulation is also achieved. The lab-testing results are considered to be sufficient to proceed to an industrial injection moulding trial.

2.5. Injection moulding trial

Industrial scale injection moulding trials with the rPP-CaCO3 were performed at a supplier facility in Slovakia. For the trials the same moulds and processing settings were used. In total 15 complete tub samples (15 tub-fronts + 15 tub-rears) were produced. During injection moulding of the parts no issues or divergence from the target material could be noted. A slight colour difference, as seen on Figure 2, is observed but this is not seen as a critical requirement.

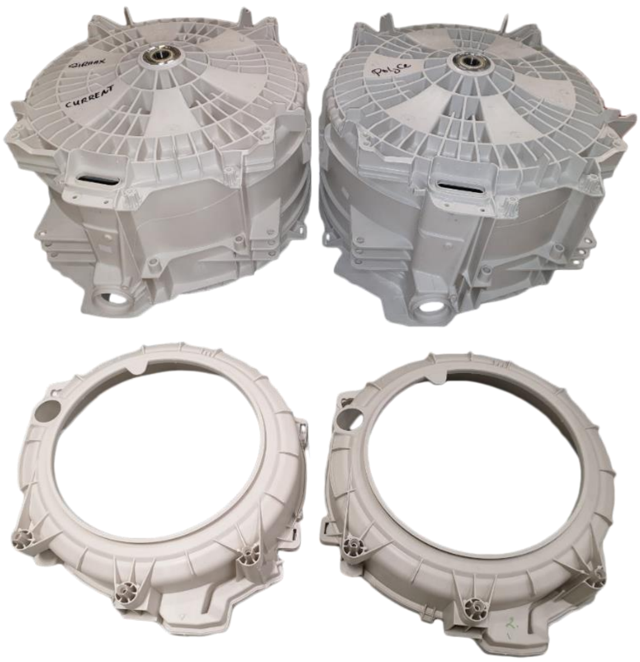


Figure 2: On the left, washer tub parts from virgin grade. On the right washer tub parts from the PCR grade.

The samples were sent to Whirlpool testing facilities to undergo the following tests.

5 samples were used for:

Table 5: Property testing of washer tub results

Type of Test	**Result**
Dimensional measurements	Pass
Welding strength	Pass
Stripping torque	Pass
Cross-section through the hub	Pass

Cross-section of counterweight pins	Pass

10 samples have been assembled to produce washing machines to be used in Reliability tests:

Table 6: Reliability tests washer tub results

Type of Test	**Status**	**Completion**
Life test (Cotton 60°)	On-going	till failure
Mechanical test	On-going	till failure
Rinse & Spin test	On-going	till failure
Transportation test	Pass	Completed

2.6. Conclusion

Several conclusions can be made from the work done with the washing machine tub trials.

Firstly, regarding the difference between the washing machines and the washing machine tubs cluster no significant difference between the two can be found. The quality of the material after pre-treatment and recycling of both clusters complies with the requirements of the target material. The extra costs involved with pre-treatment steps (extra time, labour and space needed) of the washing machine tubs could not be justified and so it was decided to continue with the washing machine cluster. Looking towards continuation after the project, discussions have been set up on the feasibility to adopt this way of clustering. As other LHA (dishwashers, dryers, …) also contain significant amounts of filled polypropylene, the first step is to set up trials on the possibility to obtain high quality rPP-$CaCO_3$ from the LHA cluster, this strategy would result in a higher probability for new business cases to be set up.

Secondly, the material testing results of the **rPP-$CaCO_3$ filled showed great results compared to the virgin grades currently used**, with no major difference in properties. This proves that clustering and targeted closed loop recycling of plastics can result in suitable materials without the need to upgrade the properties. On top of this **compliance with current regulation is also achieved**.

Industrial production trials show that the currently used virgin material can be replaced with the recycled material obtained in this work. Injection moulding trials demonstrated that there is no difference in moulding the recycled grade. Property testing of parts (weldability, drop test, assembly, functionality, weathering) show that of all tests completed at this point, the performance is according to the internal specification; at the time of writing reliability tests are still on-going.

3. Cooling and refrigeration - Fridge liner application

3.1. Approach

Currently refrigerators and freezers are already collected/treated separately from the large domestic appliances, this is because of the presence of cooling liquids/gases e.g. hydrocarbons and/or halogenated chemicals. This gives advantages for recycling as this approach already results in limited plastic grades coming from this product waste stream.

After discussion within the consortium it was decided to collect, treat and test the following clusters:

- The current cluster (collection of **only Refrigerators** from the cooling waste stream);
- Only drawers[2] 'white' (collection of **only white drawers** from the cooling waste stream);
- Only drawers 'smoky' (collection of **only smoky coloured drawers** from the cooling waste stream).
- Only drawers 'transparent' (collection of **only transparent drawers** from the cooling waste stream).

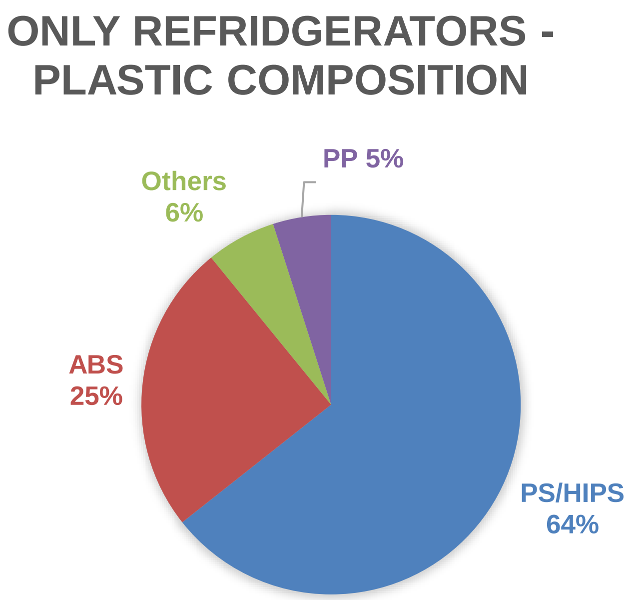
The decision was made with two applications in mind. First the entire machine would result in a HIPS grade suited for thermoforming new fridge liners. However, this imposes a problem related to food contact as contamination with PU (insolation) cannot be excluded. Looking at the fridge drawers the opportunity for contaminations is lower, so the changes to obtain food-contact approved material is higher. This clustering results in an injection moulding grade with a possible application in new fridge drawers.

[2] Internal part (vegetables drawers) of the fridge which can be easily removed manually.

3.2. Material analysis

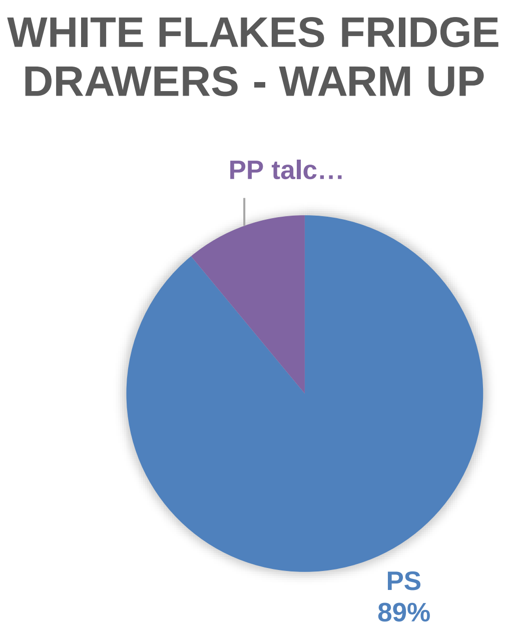
3.2.1. Current cluster only Refrigerators

The plastic composition was 65% polystyrene and 25% polypropylene. The remaining a combination of ABS and Others (see chart here below).



3.2.2. White-drawers only cluster

The plastic composition was 89% polystyrene and 11% PP based on the analysis of 35 valid measurements (as shown in the chart here below).



3.2.3. Transparent- drawers only cluster

We found 100% polystyrene based on the analysis of 42 valid measurements. A very small percentage (<1%) of plastic flakes were black and are assumed to be impurities from shredding or transport.

In conclusion: The decision was made to continue with the current way of obtaining material, **not finding a significant opportunity to reuse drawers-only PS that would justify the extra-cost for the sorting**. Moreover, the 'Transparent-drawers' cluster was partially set up to obtain a transparent material grade. However the trials showed that **it's not possible to obtain transparent grades** out of the mechanical recycling process because of impurities coming from the shredding process. A dedicated shredder for transparent materials could help resolve this issue, this however increases the costs of the material again. Feasibility trials were set-up, testing HIPS sourced from fridges as is available on the market today.

3.3. Technical requirements

<u>Materials level requirements</u>

Whirlpool technical requirements (TDS) were shared within the partners involved in the demonstrator activities. More information can be found in the appendix.

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<u>Refrigerator liners Product Level requirements</u>

Table 7: reliability test fridge & fridge door liners

Chemical Stress Resistance	To determine resistance to stress agents (chemical agents) of plastic materials under load (Environmental stress cracking) Plastic material is stressed under specific environmental conditions (chemical aging) Mechanical tests are performed before and after the aging process
Food-Grade	To determine compliance with REGULATION (EU) No 10/2011 AND AMENDMENTS

3.4. Material property testing (current cluster)

We tested two different recycled HIPS

- from MGG Polymers;
- from "recycler A" (not part of this Consortium).

Table 8: Material properties of virgin and two recycled material grades

Property	**Current Production**	**MGG Polymers**	**Recycler A**
Tensile Modulus MPa	1172	1635	1070
Yield Strength MPa	16	21	16
Tensile Strength MPa	21	21	17
Flexural Modulus MPa	1322	2064	1178
Flexural Strength MPa	31	47	33

<u>Mechanical Performances after aging - Thermal cycling</u>

Samples have been stressed for 10 cycles made of 12h @ +80°C and 12h @ -30°C (1 cycle = 24h)

Table 9: Material properties of virgin and two recycled material grades after aging

Property	Current Production	MGG Polymers	Recycler A
Tensile Modulus MPa	1262	1320	1388
Yield Strength MPa	16	21	17
Tensile Strength MPa	18	21	16
Flexural Modulus MPa	1240	2654	1260
Flexural Strength MPa	30	49	29

Mechanical Performances after aging - Environmental Stress Cracking (ESC)

Samples have been stressed for 24 hrs (room temperature RH 55%) with bending equipment and with chemical agents.

Table 10: Results Environmental stress cracking tests

	MGG Polymers		Recycler A	
	As Received	After ESC	As Received	After ESC
Flexural Modulus MPa	1483	1490	1178	1260
Flexural Strength MPa	42	42	33	33

All technical performances evaluated are considered enough to proceed with an extrusion / vacuum-forming trial. Because of material availability it was decided to proceed with the material from the “recycler A”. Materials are expected to be of similar quality.

3.5. Extrusion/Thermoforming trial (liners)

The following batches were tested with the scope of checking the ESCR which is considered one of the most critical test:

- 20% rHIPS+80% virgin HIPS[3];
- 50% rHIPS+50% virgin HIPS;
- 100% rHIPS.

[3] Virgin HIPS refers to the material used in current production of fridge and fridge door liners.

Test Plan:

- Extrusion of sheet at supplier site with the same parameters used for current production;
- Thermoform in Whirlpool plant with same parameters used in production;
- Foam the liner with current insulator foam;
- Assemble the fridge as normal production;
- Thermal cycles on thermoformed liners with selected chemical agents (ESCR).

All the composition show a similar behaviour to virgin material in term of:

- Processability (Extrusion and Thermoforming);
- Chemical resistance with polyurethane foam and oil-mix.

Two points are open:

- The colour must be optimized with master-batch (change the % and tune the colour due to more yellow background of rHIPS);
- Food Grade Compliance.

3.6. Conclusion

The overall conclusions of the HIPS trial are very positive. All properties were found suited to go to industrial trial and functionality tests (ESCR) had positive results for the intended application. **Extrusion and thermoform trials showed the material is behaving like virgin material and suitable for immediate integration in the manufacturing of fridge and fridge door liners**. From an esthetical point of view some colour optimization needs to be performed but is within the possibilities of today's technology. **From a technical point of view the material is suited for implementation**.

However, the trials were discontinued as food grade approval is a go/no-go for these trials. For now the **biggest obstacle remains Food- Grade approval**; as of today, none of the sources of recycled PS (coming from the WEEE stream Cooling Appliances) went through the process to obtain food-contact certification (EFSA Guideline + EU COMM formal authorization according to 282/2008 COMMISSION REGULATION (EC) No 282/2008 of 27 March 2008 on recycled plastic materials and articles intended to come into contact with foods and amending Regulation (EC) No 2023/2006.)

As for the information collected during this study, HIPS from WEEE is also not in the priority list from the EU COMM to be evaluated for food-grade. Until this approval is possible to obtain, **the production of 100% rHIPS fridge and fridge door liners remains impossible**.

4. Appendix

Washer tubs- Materials level requirements:

Table 1 - Physical Properties - PP Homopolymer 40% CaCO3, medium flow

Property	**Test Standard**	**Test Condition**	**Unit**	**Value**
Rheological properties				
Melt-flow rate	ISO 1133 or ASTM D1238	2.16 kg @230°C	g/10min	8.0-16.0
Mechanical Properties 23°C at 50% relative humidity				
Tensile stress at yield	ISO 527-7 ASTM D638	50 mm/min	MPa	22.0 min
Flexural modulus	ISO 178 ASTM D790	1 mm/min	MPa	2100 min
IZOD notched impact strength	ASTM D256A	23°C	J/m	30 min
Charpy notched impact strength	ISO 179-1eA	23°C	KJ/m2	2.1 min
Thermal properties				
Heat Deflection (un-annealed)	ISO 75-2B ASTM D648	0.46MPa, 120°C/h	°C	88.0 min
RTI Temperature	UL 746B	1.5 mm, impact value	°C	65 min
Other Properties				
Density	ISO 1183 ASTM D792	23°C	g/cm3	1.20-1.35
Filler Content	ISO 3541-4 ASTM D5630	23°C	%	36-44

Washer tubs- Materials level requirements test results UL

Table 2 - Physical Properties - rPP-CaCo3 - washers only cluster

Property	**Test Condition**	**Unit**	**Result**
Ash content (ISO 3451-1)	3h / 600°C	%	36.93
Density (ISO 1183)	23°C	g/cm3	1.214
Tensile Strength (ISO 527-2)	-30°C	E-Modulus MPa	7135
		Tensile Strength MPa	45.98
		Tensile strain %	1.6
	23°C	E-Modulus MPa	2975
		Tensile Strength MPa	24.14
		Tensile strain %	3.5
	50°C	E-Modulus MPa	1534
		Tensile Strength MPa	16.66
		Tensile strain %	4.2
Flexural Strength (ISO 178)	-30°C	Flexural Modulus MPa	7131
		Flexural strength MPa	97.65
		Elongation at break %	3.4
	23°C	Flexural Modulus MPa	2881
		Flexural strength MPa	46.84
		Elongation at break %	4.8

	50°C	Flexural Modulus MPa	1390
		Flexural strength MPa	28.93
		Elongation at break %	5.6
IZOD Impact (ASTM D256A)	-30°C	J/m	26.86
	23°C	J/m	29.66
	50°C	J/m	47.86
HDT (ISO 75)	120°C/h 1.8 MPa	°C	60.2
	120°C/h 0.45 MPa	°C	115.5
Melt volume rate (ISO 1133)	190°C	cm3/10min	3.45
	210°C	cm3/10min	5.29
	230°C	cm3/10min	7.84

Refrigerator liners -Materials Level requirements

Table 3 - Physical properties - HIPS, LOW FLOW, HIGH ESCR

Property	**Test Standard**	**Test Condition**	**Unit**	**Value**
Rheological Properties				
Melt mass-flow rate	ISO1133 ASTM D1238	200°C 5 Kg load	g/10min	1.8-5.0
Mechanical properties (23°C at 50% relative humidity)				
Tensile stress at yield	ISO 527-2	5 mm/min	MPa	14 min
Flexural modulus	ISO 527-2	1 mm/min	MPa	1280 min

IZOD notched impact strength	ASTM D256	23°C	J/m	80 min
Charpy notched impact strength	ISO 179-1eA	23°C	KJ/m2	8.0 min
Thermal Properties				
Heat Deflection (un-annealed)	ISO 75-2B	0.49MPa 120°C/h	°C	70 min
Vicat softening temperature	ISO 306-A	10N 50°C/h	°C	82 min
RTI temperature	UL 746B	1.5mm impact value	°C	50 min
Other Properties				
Density	ISO 1183 ASTM D792	23°C	g/cm3	1.02-1.05
Chemical Resistance	Internal Procedure			Pass
Food Grade	Internal Procedure			Pass