CycleWorks – Center of Excellence for Recycling

REPORT ON CYCLEWORKS ACTIVITY FOR **PARTNER XXXX** – PH0755

BACKGROUND:

YYYY produces biodegradable plastics technology. XXXX is an organic additive that enhances the biodegradation of traditional plastic products when placed in a biologically active landfill environment.

Avient produces masterbatches containing this additive that allows PET biodegradation.

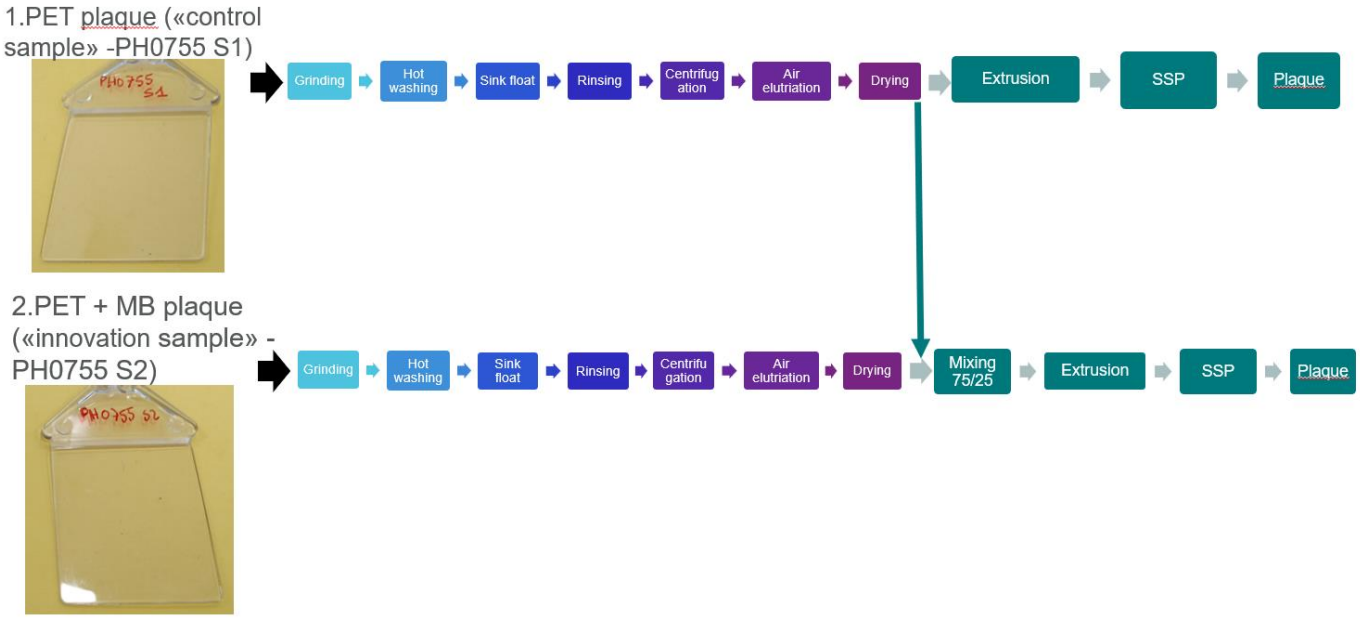
GOALS

The scope of this study is to understand the impact of this additive in a "standard" mechanical recycling process of PET bottles. Bio-Tec is asking us to execute the Recyclass recyclability evaluation protocol.

To be noticed that quoting the Recyclability Evaluation Protocol for PET Bottles from Recyclass, Chapter 2, "Following RecyClass recyclability methodology, packaging containing aluminium, metal, degradable plastics, black carbon surface, as well as PVC, PVDC and PC layers are considered disqualified for PET bottles recyclability. By consequence, packaging containing one of these features do not fall under the scope of this Protocol."

ACTIONS

The protocol steps starting from PET Plaques:



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We performed the Recyclass recyclability evaluation protocol of the plaques containing virgin PET (control sample) vs plaques with 1% of NEA0425419 (innovation sample). A virgin PET and control samples we used Indorama RamaPET N180, which is present in the list of resin allowed by the protocol.

PROTOCOL STEPS AND RESULTS

- Preparation of PET articles for evaluation

The plaques were prepared in injection molding machine Arburg 270 C GOLDEN EDITION.

The samples are:

- PH0755 S1:20 kg of control samples: 100% virgin PET
- PH0755 S2:10 kg of innovation test: 1% NEA0425419 + 99% virgin PET

Inspection Characteristic (sample thickness 1mm)	**Testing method**	**Units**	**Threshold values**	**Control sample**	**Innovation sample**
L*	CIELAB	-	-	95.18	95.24
a*	CIELAB	-	-	-0.12	-0.15
b*	CIELAB	-	-	1.62	1.60
ΔE (innovation to control)	CIELAB	-	-		0.08
Haze	ASTM D1003	%	-	1.50	1.57

- Grinding PET articles to flakes

In CyW we ground the plaques by Grinder MDS 410/200 (Hellweg). The set program is “PET”.

We obtained flakes of 6 mm average size (for both control and innovation).

- Washing and hot rinsing of flakes

The flakes had been washed in a Sorema washing lab line.

The machine set up was:

- Washing unit T(°C): 85
  Cleaner 1136: 1%NaOH + 0,3% Lialet 111-7 in water (98,7%)
  Washing process time (min): 15
  Stirrer speed (rpm): 500
- Rinsing process time (min): 10-15 min tap water up to pH=7
  Vibrating sieve bias: 60°
  Sieve size (mm): 3
  Centrifuge unit T(°C): 150
  Centrifuge time (s): 10

Residual moisture after centrifuge: 0,8% for both the control and innovation samples.
PET flakes have to be dried and crystallized in an air-forced oven and then in a vacuum oven before the extrusion process.

- Density separation:

The flotation test is made by magnetic stirrer and a becher.

The input material is 50g of PET flakes, in 500ml demi water at room temperature and stirring for 5 min at 250rpm. Then, the floating and bottom flakes were separately filtered and dried in a ventilated oven.

The floating material is 2% for both the control and innovation samples.

- Flakes drying and crystallization.

This process happened in two steps. The first is drying in air-forced oven at 100°C for 24 hrs. The second step is crystallizing in vacuum oven in nitrogen atmosphere, at 160°C for 2 hrs. The residual moisture is <50ppm for both the control and innovation samples (measured by Karl-Fischer titrator).

- Air elutriation

Air elutriation was not performed as it is not relevant for this case.

- Flakes properties characterization

Inspection Characteristic	Testing method	Units	Threshold values	Control sample	Innovation sample
L*	CIELAB	-	-	81.57	80.19
a*	CIELAB	-	-	-0.86	-1.48
b*	CIELAB	-	-	-2.16	-1.20
ΔE (innovation to control)	CIELAB	-	-		1.80
Bulk density	ASTM D1895	Kg/m^3	>500 kg/m^3	690	650
Intrinsic viscosity before washing	ISO 1628-1:2021	dL/g	-	0.720	
Intrinsic viscosity after washing	ISO 1628-1:2021	dL/g	<0.02dL/g delta to the control	0.604	0.649 Δ IV= 0.045*

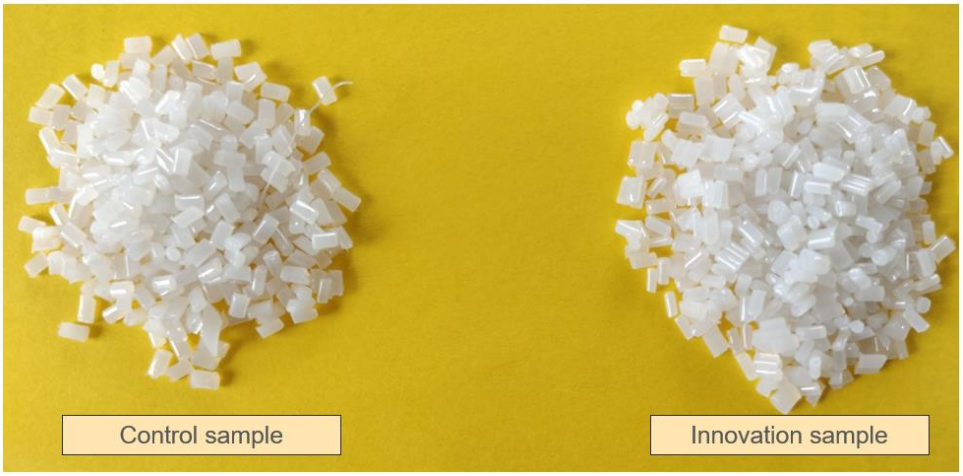
*The protocol at this point is unclear; our interpretation is the innovation sample IV should be not lower than control sample IV. But in our case IV innovation is higher than control.

- Pellet production

The pellets were produced in Labtec-30 single screw extruder.

The production temperature was 285°C, screw speed 110 rpm, pressure 35 bar, 80 micron filter and throughput 5kg/hr. No build up on screen. No fumes odor or residues on die head observed for both the control and innovation samples. Pressure was stable over the entire production time.

- Pellets properties evaluation



Inspection Characteristic	Testing method	Units	Threshold values	Control sample	Innovation sample
L*	CIELAB	-	-	77.45	77.07
a*	CIELAB	-	-	-1.33	-1.55
b*	CIELAB	-	-	-2.54	-2.11
ΔE (innovation to control)	CIELAB	-	-		0.61
Intrinsic viscosity	ISO 1628-1:2021	dL/g	IV drop of A.25 (innovation) is inferior to the IV drop of the A.0 (control) ±0.02 dL/g (IV drops are measured comparing IV of pellets to unwashed flakes)	0.598 Δ IV= 0.122	0.578 Δ IV= 0.142

- Pellets drying and crystallization.

The pellets (both standard and innovation samples) had 0.1% of humidity, after 1 night air-forced oven at 100°C .

- Solid state polymerization (SSP)

The SSP was performed at 210°C for 8hrs, by SB Plastics Machinery Moby lab scale plant.

- Solid state polymerization pellets characterization

Inspection Characteristic	Testing method	Units	Threshold values	Control sample	Innovation sample
L*	CIELAB	-	-	79.41	79.10
a*	CIELAB	-	-	-1.67	-1.93
b*	CIELAB	-	-	-0.26	0.17
ΔE (innovation to control)	CIELAB	-	-		0.59
Intrinsic viscosity	ISO 1628-1:2021	dL/g	IV curve over time to determine SSP rate from 2 to 8 hrs. SSP rate: no more than 10% delta variation to control *	0.673	0.674
Melt temperature	ISO 11357-3:2018 (Heat-cool-heat cycle at 10°C/min (heating and cooling) from 40°C to 300°C with 2 minutes of isotherm between each ramp)	°C	Melt temperature second heat: No more than 10% delta variation to control	243.06	242.76 ΔT = 0.3

*Our equipment does not permit to perform the IV measurement during the SSP process. We report the IV value after 8 hours.

The fluorescence evaluation is not relevant for this case.

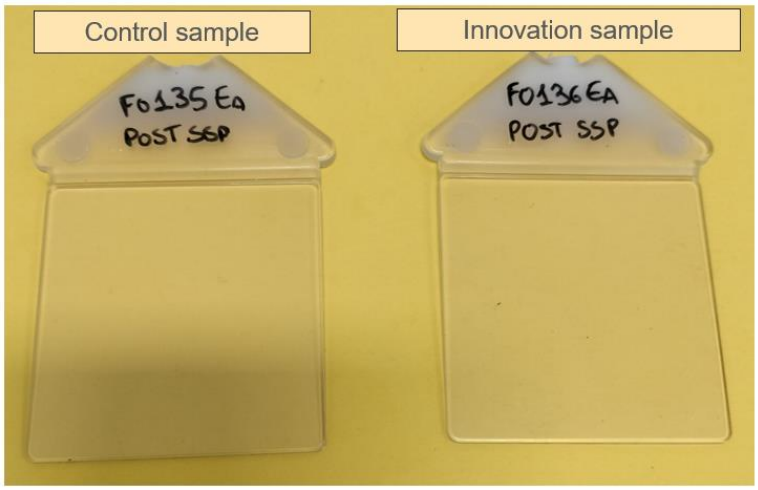
- Pellet blends composition and plaque injection molding

Pellets have been blended according to the table below; the protocol requires to prepare B.0 and B.25, while B.50 is optional:

BLEND	COMPOSITION	% VIRGIN RESIN	EFFECTIVE % CONTROL PET	EFFECTIVE % INNOVATION SAMPLE
B.0	50 % Virgin Pellet 50 % A.0	50	50	0
B.25	50 % Virgin Pellet 50 % A.25	50	37.5	12.5
OPTIONAL B.50	50 % Virgin Pellet 50 % A.50	50	25	25

The plaques were prepared in the same way described for the first run.

- Characterization test of plaques



Inspection Characteristic (sample thickness 3 mm)*	**Testing method**	**Units**	**Threshold values**	**Control sample**	**Innovation sample**
L*	CIELAB	-	>87	87.52	87.80
a*	CIELAB	-	>-3	-0.47	-0.63
Δb*	CIELAB	-	<1.5 (compared to B.0)		-0.77
ΔE (innovation to control)	CIELAB	-	-		0.84
Haze	ASTM D1003	%	-	37.24	42.66

* The plaques were measured by overlapping 3 plaques of 1 mm thickness; the measurements were repeated 3 times.

CONCLUSIONS

During the protocol execution, we had no problems with the processability of the material. From the results obtained, we can state that the innovation samples (with 1% Avient MB- NEA0425419) and the control sample (100% PET) reacted well during the recycling loop simulations.
In particular, the IV measurements of the sample after the SSP process are good: the control sample IV value and innovation sample have almost the same value.

If we look at the plaques color measurement, we can observe that the measurements of the innovation sample plaques and the control one are not significantly different. The inspection values are all on the acceptance criteria.

Reasonably, a PET bottle made with 1% XXXX- NEA0425419 should not be detrimental for PET bottle recycling according to RecyClass PET Bottle Recyclability evaluation protocol V1.0