

Deliverable 4.2

Technical reports on results of verification analysis performed on bottle

The produced preforms with 100% recycled PET (R-PET), 50% R-PET/V-PET and 100% V-PET with different colors, have been blown with traditional blowing machines using different bottle molds, in order to verify the mechanical characteristics of the bottles characterized by different stretch ratios.

In particular, three different bottles design have been selected for the bottle production and characterization, as shown in the following pictures in figures 1-4.



Figure 1. Clear 1 liter bottle for seeds oils (COOP design). A) 31g 100% V-PET bottle; B) 31g 50% R-PET/V-PET bottle.



Figure 2. White 1 liter bottle for seeds oils (DANTE design). A) 31g 100% V-PET bottle; B) 31g 50% R-PET/V-PET bottle; C) 31g 100% R-PET bottles.



Figure 3. Green 1 liter bottle for seeds olis (OLITA design). A) 31g 100% V-PET bottle; B) 31g 50% R-PET/V-PET bottle; C) 31g 100% R-PET bottles.



Figure 4. Red 1 liter bottle for seeds oils (OLITA design). A) 31g 100% V-PET bottle; B) 31g 50% R-PET/V-PET bottle; C) 31g 100% R-PET bottles.

The present report focuses on the production of different 1l bottles using the preforms produced with up to 100% R-PET food grade. Then, the produced bottles have been analyzed in order to verify the quality of the packages and their suitability to be used as edible seeds oil container.

In particular, the bottles characterization has consisted in:

- - dimensional and visual analysis performed after the bottle production performed by using a caliper Borletti and with a Magna Mike 8500 by Olympus to measure the wall thickness;

- thermal analysis in dynamic mode by DSC to verify if difference occurred in thermal characteristics and in the morphology of the bottle due to the presence of R-PET and by the different composition. Thermal analysis were carried out with a Differential scanning calorimeter (DSC), DSC823 by Mettler Toledo, by setting the following thermal method:

I heating from 30 to 300°C at 10°C/min;

The percentage of crystallinity X_c was determined by using the following equation:

$$X_c = (|\Delta H_m| - |\Delta H_c|) / \Delta H^0_{PET}$$

where $|\Delta H_m|$ is the absolute value of the enthalpy of melting analyzed during the heating steps;

$|\Delta H_c|$ is the absolute value of the enthalpy of crystallization analyzed during the heating/cooling steps;

ΔH^0_{PET} is the theoretical enthalpy of fusion of PET here considered as 140,1 J/g [1].

- Top load test carried out using a dynamometer.
- The measure of Haze, Transmittance and clarity by using an Hazemeter Gardner.

Results and Discussions

The 31g bottles above described have been produced by blowing the up to 100% R-PET preforms produced during the RepackEdoils project. The bottles have been blown with industrial blowing machine by KOSME at Mataluni Spa Company using different molds of bottles, in order to verify the capability of R-PET 31g preforms to be blown in different kind of bottles shapes and therefore changing the stretch ratio between preform and bottles.

The blowing conditions set have been optimized for any kind of preform produced considering both the R-PET content and the colorant used. In particular, the blowing parameters have been set in order to ensure high production rate and high acceptability of the bottles after a visual inspection.

In the following tables 1-2-3 are summarized the principal blowing process parameters and their relative settings according to the different preforms blown.

Table 1: Optimized Blowing Condition for the different Preforms with 100% V-PET

31g 100% V-PET Preforms	Clear COOP design	White Dante design	Green Olita design	Red Olita design
Pre-Blowing Pressure	9	9	8	8
Blowing Pressure	33	34	34	34
Production rate	5000	4800	5200	5200
Percentage power of IR lamp for heating preforms [%]				
Neck position	90	100	100	100
Upper part of the body	72	85	83	83
Down part of the body	53	60	42	42
Bottom position	65	65	68	68

Table 2: Optimized Blowing Condition for the different Preforms with 100% R-PET

31g 100% R-PET Preforms	Clear COOP design	White Dante design	Green Olita design	Red Olita design
Pre-Blowing Pressure	9	9	8	8
Blowing Pressure	33	34	34	34
Production rate	5000	5000	5200	5200
Percentage power of IR lamp for heating preforms				
Neck position	85	95	95	95
Upper part of the body	70	85	80	80
Down part of the body	50	60	45	45
Bottom position	60	65	68	68

Table 3: Optimized Blowing Condition for the different Preforms with 50% R-PET/ V-PET

31g 50% R-PET/V-PET Preforms	Clear COOP design	White Dante design	Green Olita design	Red Olita design
Pre-Blowing Pressure	9	9	8	8
Blowing Pressure	34	34	34	34
Production rate	5000	5000	5200	5200
Percentage power of IR lamp for heating preforms				
Neck position	85	95	95	95
Upper part of the body	70	85	80	80
Down part of the body	50	60	45	45
Bottom position	60	65	68	68

The visual inspection of a representative sample of bottles gave positive results as regards the shape of the bottles. As concern to the main aspect of the bottles:

- For clear bottles is easy to discriminate the bottles containing R-PET from the 100% V-PET ones because when even 50% of recycled material is present in the bottles these appear yellowish, as is also possible to observe looking at Figure 1;
- white bottles with pure R-PET material are also quite critical in the aspect, as is also possible to notice in Figure 2;
- Red and Green bottles produced with R-PET material are completely indistinguishable from pure virgin PET bottles.

The weight and dimensional characteristics of the produced bottles have shown that all the different bottle typologies show a good material distribution along the bottle side wall and high symmetry between the two sides of the bottle.

However, the 50% R-PET/ V-PET green and red bottles are characterized by a thinner wall thickness in the body region and an a thicker bottom compared with pure V-PET and R-PET

samples analyzed. This behavior is most probably due to the lower intrinsic viscosity of the preform produced with 50% of R-PET that makes more complex and difficult the material distribution during the blowing process.

The optical properties of the clear bottles were assessed by Haze, transmittance and Clarity measurements.

In table 4 are reported the optical characteristics of the 31g clear bottles at different percentages of R-PET.

Table 4. Optical characteristics of clear bottles produced with different R-PET concentrations.

Bottles	Transmittance [%]	Haze [%]	Clarity [%]
31g clear 100% R-PET	90,0±0,1	2,97±0,20	97,8±0,1
31g clear 50% R-PET/V-PET	90,6±0,4	2,72±0,32	97,5±0,2
31g clear 100% V-PET	91,6±0,5	1,49±0,24	97,0±0,1

The bottles with R-PET show an higher haze value than the virgin PET bottles, that can be partially attributed to a different crystallinity content. In fact, it has previously observed, analyzing the produced preforms (see Deliverable 4.1 page 7) that the presence of R-PET promote a higher tendency to crystallize during the cooling. In order to determine the percentage of the produced bottles a dynamic heating was carried out on the bottles, sampling the body of each typology.

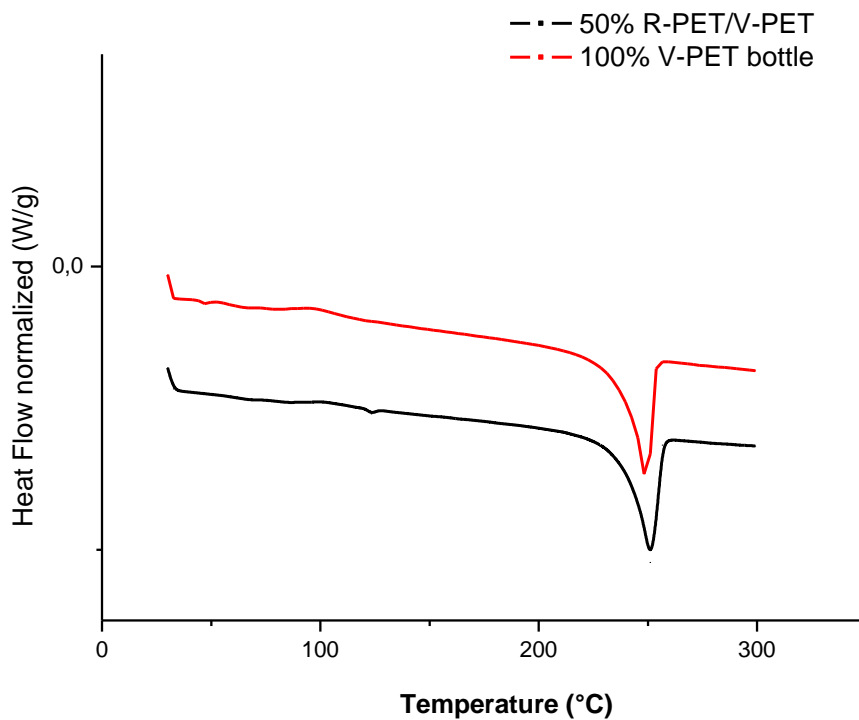


Figure 5. First Heating thermograms for 50% R-PET/V-PET and 100% V-PET bottles.

The DSC analysis have confirmed a slight increase the melting enthalpy and then the presence of a higher crystallinity percentage for the R-PET bottles (25% for 50% R-PET bottles and about 22% for 100% V-PET samples).

Finally, in order to characterize the mechanical resistance of produced bottles with R-PET compared to pure V-PET ones, Top Load analyses have been carried out on the different bottles blown during this research project.

Looking at figure 6, it is possible to underline that the presence R-PET material do not significant affect the mechanical resistance to the top compression, but the design of the bottle can significant improve the mechanical characteristics of the container, because it is possible to optimize the stretch ratios and increase the polymer chain orientation.

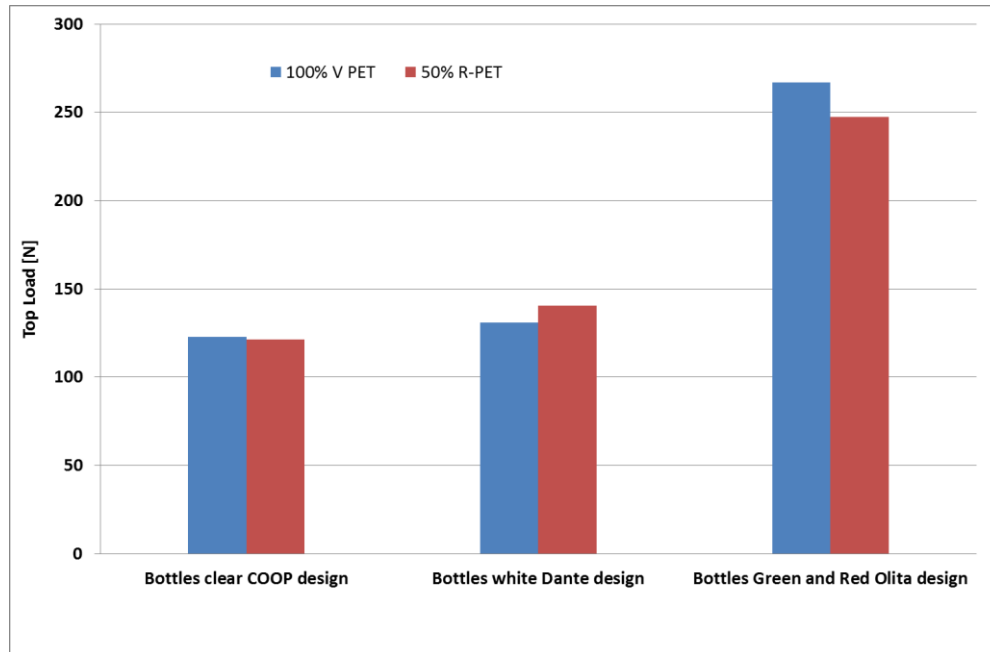


Figure 6. Top Load histograms of the different bottles produced.

Conclusions

The blowing process of the R-PET has not shown specific issues. The preforms can be blown with traditional industrial blowing machine without reducing the production rate and guaranteeing a similar bottle quality than pure virgin PET ones, only optimizing the process parameters.

In particular, the colored bottles are almost indistinguishable from pure virgin PET bottles both in appearance than in mechanical performances. Moreover, even if the presence of recycled material affects the optical characteristics of PET bottles, the Haze and transmittance values analyzed are not as critical. Therefore, clear bottles with 50% R-PET/V-PET can also be produced for Edible seeds oils market especially if polyester sleeves are used as bottle labels.

References

- (1) Iroh J.O. Polymer Data Handbook. Poly(etilene terephtalate) section. Oxford University Press: New York 1999.