Deliverable 4.3

Technical report on the results of storage tests to define shelf-life of oils in PCR-PET packaging

The effect of oils packaging in recycled PET in terms of product quality and stability was evaluated. The oil aging was determined by the application of analytical and sensory techniques, which are able to monitor the evolution of oil quality key indicators.
Storage tests were carried out in order to evaluate over time differences in oils quality due to the packaging in recycled PET in comparison with standard PET.
On the basis of the data obtained the shelf-life of the oils stored in the new packages under normal conditions of distribution and marketing was defined.

Tests in accelerated conditions

Tests were carried out in order to verify if the use of PCR –PET instead of PET affects the quality parameters of the bottled oils also in stressed conditions of storage.
Sunflower oil (provided from Olio Dante S.p.A.) was bottled in PET bottles deriving from the blow-molding of 31 g different preforms obtained with the equipment installed during the RE-PACK project.
The filled bottles (PET standard or made with PCR PET) were maintained in a climatic chamber at 40°C for three months and during the test several parameters analytical and sensorial regarding oils quality were monitored.

The following table resumes the experimental plan used for the test:

TEST IN ACCELERATED CONDITIONS (oven at 40°C)			
Bottles	**Duration of the test**	**Analysis on the oils**	**Sampling**
Clear PET	90 days	Free Acidity, Peroxides value, spectrophotometric indices, vitamin E, Panel test, volatiles compounds	0, 10, 40 and 90 days
Clear 50% RPET			
White PET			
White 50% RPET			
Green PET			
Green 50% PET			
Green 100%PET			

Photo 1. *PCR-PET and PET bottles filled with sunflower oil stored in a climatic chamber for 90 days at 40°C for the accelerated shelf-life test.*

The materials and methods used for the test are briefly described below.

Oil Quality indices

Free acidity, spectrophotometric indices and peroxides number were carried out according to by EU regulation for olive oil (EU Reg. 2568/1991).

Vitamin E (tocopherols) in oils

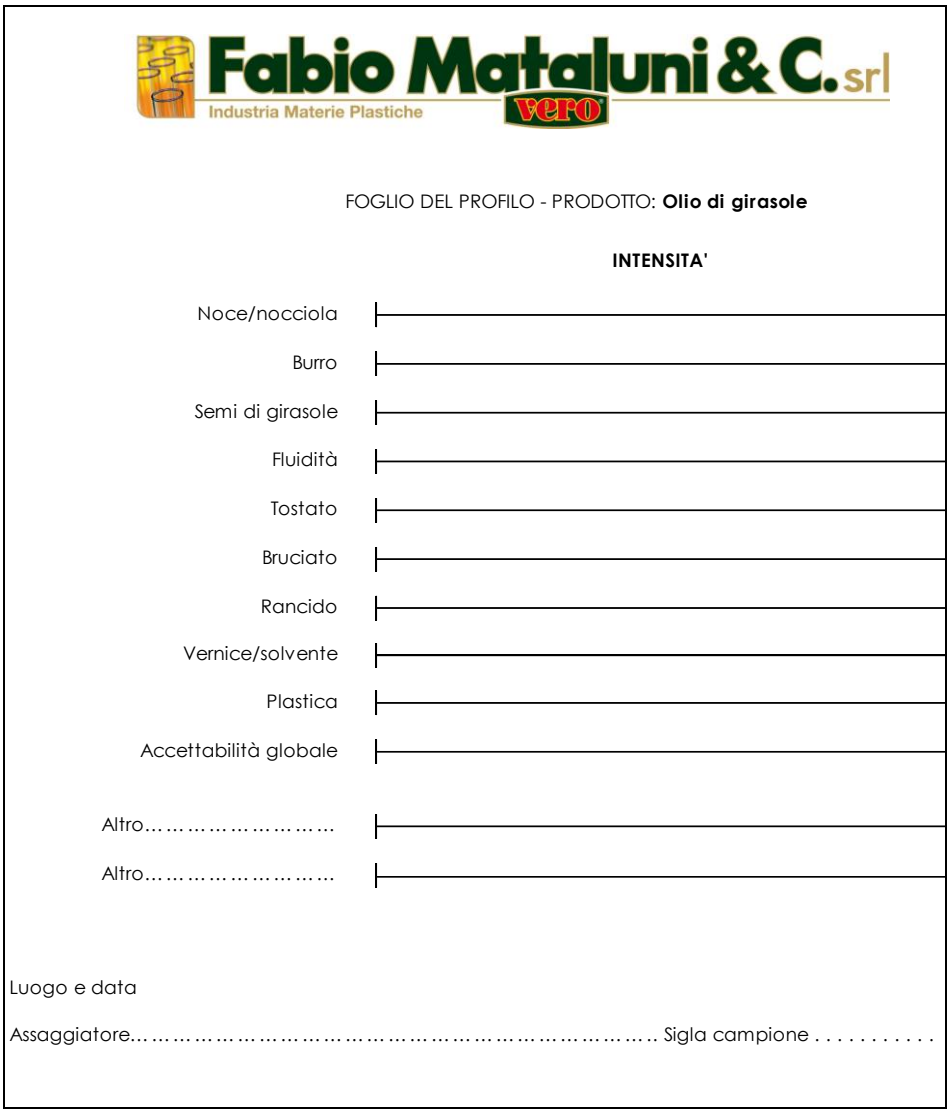
Tocopherols content was determined by reversed phase HPLC analysis on a LC-10AD Shimadzu (Milan, Italy) liquid chromatograph equipped with a SPD M10A VP diode array detector (Shimadzu, Milan, Italy). The chromatographic separation was achieved on a 5 µm ODS-3 Prodigy (250 mm, 4.6 mm i.d.) reversed-phase column (Phenomenex, Macclesfield, UK). The elution conditions were those reported by Tonolo and Marzo (1989) and Savarese et al. (2013a)

Volatile compounds in oils headspace by SPME GC/MS Analysis

Volatile compounds were extracted by SPME using a 1 cm length 50/30 µm DVB-CAR-PDMS fiber (Supelco, Bellefonte, USA) and analyzed by GC/MS. Sunflower oil samples (3 mL) were equilibrated for 10 minutes in a 20 mL vial at 40 °C. Fiber was exposed for 30 minutes. The GC analysis was performed according to the method reported by Savarese et al. (2013b)

Sensory analysis - Panel test

Sensory assessment of sunflower oil was carried out according to the method indicated by EU regulation for olive oil (EU Reg. 2568/1991) with slight modifications. Ten trained judges analyzed the odor, aroma and the overall sensation (flavor) of the samples and evaluated them on a test sheet which was reported below:



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FOGLIO DEL PROFILO - PRODOTTO: **Olio di girasole**

INTENSITA'

Noce/nocciola
Burro
Semi di girasole
Fluidità
Tostato
Bruciato
Rancido
Vernice/solvente
Plastica
Accettabilità globale

Altro……
Altro……

Luogo e data

Assaggiatore…… Sigla campione . . . . . .

Figure 1. *Sheet used for descriptive tests.*

Panellists were required to evaluate, using a non-structured 10 cm length hedonic scale, several sensory attributes, namely nut, butter, sunflower seeds, fluidity, (from light yellow to brown), "roasted food" smell, any other undesirable smell (burnt, rancid, painty, solvent, plastic, ….), overall acceptability.

Furthermore, oil samples stored for different times in virgin PET and in R-PET bottles were subjected to a discriminant test. Discrimination testing is a technique employed in sensory analysis to determine whether there is a detectable difference among two or more products. The test uses a trained panel to discriminate from one product to another.

Triangular tests were carried out according to the methodology recommended by 4120:2004. Three samples were presented, two of which are identical and the other one different, and the subject was asked

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to taste and smell them and to to state which product they believe is the odd one out. The data related to the evolution of the some sunflower oil parameters were reported in the following tables and figures.

Table 1. *Parameters in sunflower oils packed in clear bottles in PET and in 50% PCR-PET during oven test.*

PET CLEAR vs PCR PET 50% CLEAR					
		Initial Value	Storage at 40°C		
			After 10 days	After 40 days	after 90 days
PET virgin	Peroxide Value ($meqO_2/kg$)	0.43 ± 0.06	2.70 ± 0.28	2.80 ± 0.08	4.22 ± 0.11
PCR-PET 50%		0.43 ± 0.06	2.97 ± 0.50	3.26 ± 0.12	4.49 ± 0.17
PET virgin	K_{232}	2.57 ± 0.26	2.91 ± 0.15	2.31 ± 0.33	2.52 ± 0.11
PCR-PET 50%		2.57 ± 0.26	2.66 ± 0.27	2.10 ± 0.14	2.33 ± 0.06
PET virgin	K_{270}	1.92 ± 0.24	2.03 ± 0.08	1.85 ± 0.08	1.85 ± 0.10
PCR-PET 50%		1.92 ± 0.24	1.99 ± 0.17	1.74 ± 0.05	1.82 ± 0.01
PET virgin	ΔK	-0.15 ± 0.00	0.31 ± 0.05	0.23 ± 0.05	0.25 ± 0.01
PCR-PET 50%		-0.15 ± 0.00	0.34 ± 0.08	0.19 ± 0.02	0.21 ± 0.00
PET virgin	α-tocopherol (mg/kg)	793.9 ± 50.1	754.84 ± 31.9	769.4 ± 13.3	728.7 ± 13.8
PCR-PET 50%		50.1 ± 0.0	754.3 ± 24.0	755.4 ± 11.2	703.3 ± 43.3
PET virgin	Free Acidity (% oleic acid)	0.05 ± 0.00	0.04 ± 0.01	0.05 ± 0.01	0.04 ± 0.00
PCR-PET 50%		0.05 ± 0.00	0.04 ± 0.00	0.05 ± 0.01	0.04 ± 0.00

Table 2*. Parameters in sunflower oils packed in white colored bottles in PET and in 50% PCR-PET during oven test.*

PET WHITE vs PCR PET 50% WHITE					
		Initial Value	Storage at 40°C		
			After 10 days	After 40 days	after 90 days
PET vergine	Peroxide Value ($meqO_2/kg$)	0.43 ± 0.06	3.03 ± 0.19	3.59 ± 0.02	4.89 ± 0.08
PCR-PET 50%		0.43 ± 0.06	2.91 ± 0.16	3.01 ± 0.04	4.61 ± 0.04
PET vergine	K_{232}	2.57 ± 0.26	2.41 ± 0.15	2.15 ± 0.22	2.35 ± 0.03
PCR-PET 50%		2.57 ± 0.26	2.51 ± 0.10	2.20 ± 0.28	2.28 ± 0.04
PET vergine	K_{270}	1.92 ± 0.24	1.89 ± 0.25	1.78 ± 0.10	1.85 ± 0.04
PCR-PET 50%		1.92 ± 0.24	1.93 ± 0.06	1.73 ± 0.18	1.79 ± 0.01
PET vergine	ΔK	-0.15 ± 0.00	0.25 ± 0.01	0.20 ± 0.03	0.20 ± 0.00
PCR-PET 50%		-0.15 ± 0.00	0.24 ± 0.01	0.21 ± 0.03	0.20 ± 0.01
PET vergine	α-tocopherol (mg/kg)	793.9 ± 50.1	740.6 ± 12.0	774.4 ± 6.0	776.7 ± 24.7
PCR-PET 50%		793.9 ± 50.1	711.5 ± 32.6	769.0 ± 9.7	755.8 ± 12.6
PET vergine	Free Acidity (% oleic acid	0.05 ± 0.00	0.05 ± 0.01	0.05 ± 0.01	0.05 ± 0.01
PCR-PET 50%		0.05 ± 0.00	0.06 ± 0.01	0.04 ± 0.01	0.04 ± 0.01

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Table 3. *Parameters in sunflower oils packed in green colored bottles in PET, in 50% PCR-PET and in 100% PCR-PET during oven test.*

PET GREEN vs PCR PET 50% GREEN vs PCR PET 100%					
		Initial Value	Storage at 40°C		
			After 10 days	After 40 days	after 90 days
PET vergine	Peroxide Value (meqO2/kg)	0.43 ± 0.06	2.53 ± 0.06	3.09 ± 0.07	4.30 ± 0.05
PCR-PET 50%		0.43 ± 0.06	2.51 ± 0.10	3.15 ± 0.07	4.21 ± 0.14
PCR-PET 100%		0.43 ± 0.06	2.71 ± 0.07	3.23 ± 0.05	4.53 ± 0.19
PET vergine	K_{232}	2.57 ± 0.26	2.27 ± 0.17	2.30 ± 0.07	2.34 ± 0.09
PCR-PET 50%		2.57 ± 0.26	2.38 ± 0.17	2.11 ± 0.15	2.55 ± 0.07
PCR-PET 100%		2.57 ± 0.26	2.20 ± 0.23	1.96 ± 0.04	2.39 ± 0.12
PET vergine	K_{270}	1.92 ± 0.24	1.79 ± 0.08	1.84 ± 0.00	1.83 ± 0.03
PCR-PET 50%		1.92 ± 0.24	1.76 ± 0.08	1.76 ± 0.07	1.91 ± 0.05
PCR-PET 100%		1.92 ± 0.24	1.73 ± 0.17	1.67 ± 0.03	1.84 ± 0.04
PET vergine	ΔK	-0.15 ± 0.00	0.22 ± 0.03	0.22 ± 0.02	0.21 ± 0.00
PCR-PET 50%		-0.15 ± 0.00	0.24 ± 0.04	0.19 ± 0.03	0.25 ± 0.02
PCR-PET 100%		-0.15 ± 0.00	0.24 ± 0.02	0.15 ± 0.01	0.22 ± 0.02
PET vergine	a-tocopherol (mg/kg)	793.9 ± 50.1	744.3 ± 25.9	773.9 ± 15.5	730.3 ± 27.1
PCR-PET 50%		793.9 ± 50.1	704.9 ± 6.8	735.0 ± 5.9	723.1 ± 1.9
PCR-PET 100%		793.9 ± 50.1	714.0 ± 6.8	742.2 ± 8.1	721.9 ± 14.4
PET vergine	Free Acidity (% oleic acid)	0.05 ± 0.00	0.05 ± 0.01	0.05 ± 0.01	0.05 ± 0.01
PCR-PET 50%		0.05 ± 0.00	0.04 ± 0.00	0.04 ± 0.01	0.04 ± 0.01
PCR-PET 100%		0.05 ± 0.00	0.05 ± 0.01	0.05 ± 0.01	0.05 ± 0.01

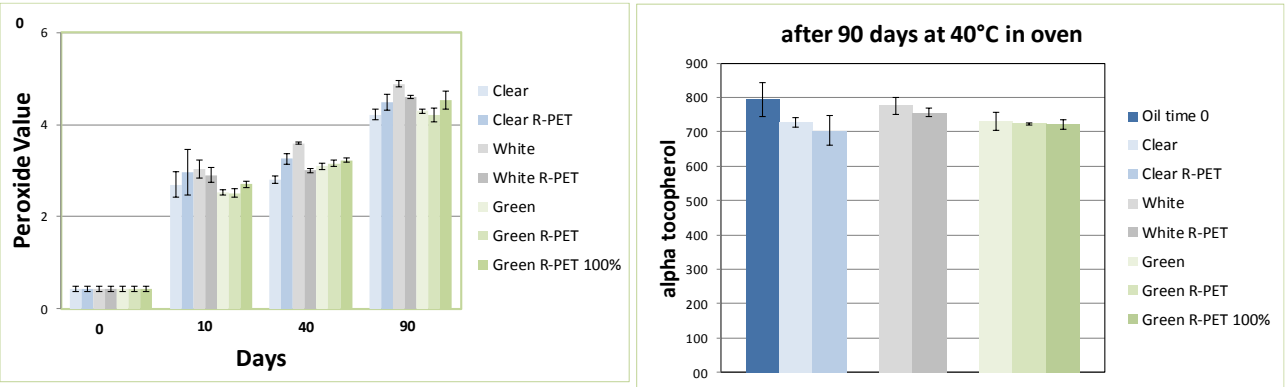

Figure 2. *Evolution of Peroxide Value in oils packed in different bottles during the oven test (left) and of alpha tocopherol content in sunflower oils after 90 days of storage in oven at 40°C.*

The data related to primary (peroxide value and k232) and secondary (k270 and delta k) oil oxidation products showed that non-significant differences were found in the oils due to packaging. Also other

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parameters linked to commercial (free acidity) and nutritional (alpha-tocopherol) indices confirm that the use of R-PET in the package doesn't affect the quality of the oils.

The evaluation of any unpleasant odors and flavors in oil deriving from the use of recycled packaging was carried out by the organoleptic analysis comparing sensory attributes of oils in PET and R-PET. Two types of sensory test were used: a descriptive test which tries to provide description of the sensory qualities of food and involves detection and description of both qualitative and quantitative sensory attributes. In particular, the quantitative aspect or intensity expresses the degree to which a characteristic is present and is expressed by trained tasters which assign a value on a scale.

Also the discrimination testing which is a technique employed in sensory analysis to determine whether there is a detectable difference among two or more products was used. The test uses a trained panel to discriminate from one product to another.

The main results regarding the sensory analysis aimed to evaluate the effect of the employing PCR-PET for the packaging of edible oils are reported below.

Photo 2. *Panelists in the panel room during a sensorial descriptive test on sunflower oils bottled in PET and PCR-PET and stored in oven at 40°C for several days.*

Table 4. *Results of the descriptive tests carried out on oils packed in different PET and PCR—PET and stored in oven at 40°C for several days. In the table the average, the standard deviation and the median of the scores ascribed by panelists to the main sensorial attributes detected in oils are reported.*

	Nut/Hazelnut			Butter			Sunflower seeds			Fluidity			Roasted			Overall Acceptability			Rancid			Painty/Solvent			Burnt			Plastic		
	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median
Sunflower oil initial	2.1	1.1	1.9	3.1	1.0	2.6	4.2	1.5	4.2	5.3	1.3	5.3	1.5	0.5	1.4	5.2	1.9	4.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
after 10 days at 40°C																														
Clear	2.4	2.1	1.6	1.6	0.5	1.4	4.4	1.9	4.6	5.5	1.8	6.0	1.7	0.7	1.5	5.5	2.2	5.3	0.4	0.7	0.0	0.1	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Clear R-PET	2.0	0.9	1.8	2.9	1.0	3.0	4.2	1.8	4.0	5.1	1.6	5.5	2.0	0.9	2.0	4.8	2.1	4.3	0.2	0.5	0.0	0.1	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
White	1.9	0.2	2.0	3.4	1.5	3.3	5.1	0.9	5.0	4.5	2.3	3.6	2.3	1.8	1.8	5.0	2.4	4.9	0.3	0.7	0.0	0.1	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
White R-PET	1.7	0.6	1.6	2.8	1.2	3.0	4.3	1.5	4.5	5.0	2.2	4.5	2.1	1.7	1.8	4.6	1.4	4.9	0.2	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Green	1.4	0.6	1.7	3.4	2.4	3.3	4.7	2.1	5.0	4.8	2.3	4.5	1.9	0.8	1.9	6.0	3.3	7.0	0.1	0.3	0.0	0.2	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Green R-PET	0.9	0.2	0.9	3.0	1.2	3.0	4.7	1.5	4.7	4.9	2.3	4.6	1.5	0.8	1.5	5.9	1.9	5.9	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Green 100% R-PET	1.4	0.4	1.4	2.8	2.5	1.4	4.3	2.2	4.8	3.7	1.8	3.9	1.9	1.3	2.0	5.6	2.8	5.8	0.1	0.2	0.0	0.2	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0
after 40 days at 40°C																														
Clear	2.5	0.7	2.5	3.7	2.7	3.3	4.5	2.6	4.8	5.3	2.1	5.9	1.4	0.5	1.2	5.2	2.5	4.7	0.3	0.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.3	0.7	0.0
Clear R-PET	2.3	1.1	2.3	3.7	2.6	3.3	3.8	1.2	4.3	5.4	2.9	4.6	1.5	0.8	1.6	5.3	2.2	4.6	0.3	0.8	0.0	0.1	0.2	0.0	0.1	0.2	0.0	0.4	0.9	0.0
White	1.4	0.3	1.5	3.2	2.0	3.1	3.8	1.2	4.3	4.3	1.7	4.6	2.0	1.3	2.0	4.5	2.7	4.2	0.6	1.3	0.0	0.3	0.6	0.0	0.3	0.6	0.0	0.4	0.9	0.0
White R-PET	1.1	0.4	1.2	3.3	2.1	2.7	4.0	1.6	4.3	4.4	1.9	5.2	1.6	0.9	1.7	4.9	2.5	5.2	0.4	0.7	0.0	0.3	0.5	0.0	0.3	0.5	0.0	0.3	0.5	0
Green	0.8	0.3	0.8	3.4	2.4	2.9	4.2	3.1	3.6	4.7	2.6	4.8	1.3	0.7	1.2	4.5	1.9	4.0	0.4	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.3	0.4	0.2
Green R-PET	1.0	0.1	1.0	3.6	2.4	2.6	4.2	3.3	3.9	4.8	2.5	4.5	1.5	1.2	1.0	4.6	1.4	4.4	0.2	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.4	0.0
Green 100% R-PET	0.6	0.6	0.6	3.2	2.3	2.6	4.3	2.8	3.8	4.4	3.1	5.0	1.2	1.0	0.7	4.5	2.4	4.6	0.2	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
after 90 days at 40°C																														
Clear	0.5	0.5	0.6	3.3	2.7	2.2	4.3	2.1	3.7	4.3	2.6	3.6	0.9	1.2	0.6	4.9	2.4	4.2	0.2	0.6	0.0	0.3	0.7	0.0	0.0	0.0	0.0	0.4	0.6	0.0
Clear R-PET	1.0	1.4	1.0	3.5	3.9	2.5	4.7	2.8	3.7	5.0	2.5	4.5	1.2	1.2	1.3	4.0	2.8	2.8	0.6	1.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.7	0.7	0.4
White	1.0	1.7	0.0	2.7	1.7	1.9	4.2	1.5	4.6	4.9	2.6	4.5	2.1	3.0	0.8	5.1	2.1	4.8	1.2	2.0	0.0	1.1	2.8	0.0	0.9	1.5	0.0	0.4	0.8	0.0
White R-PET	0.8	1.4	0.0	3.1	1.9	2.6	3.9	2.3	5.5	5.2	2.6	5.0	1.8	2.8	1.0	5.6	1.7	5.1	1.0	1.1	0.5	0.2	0.5	0.0	0.6	1.2	0.0	0.6	0.9	0.0
Green	1.1	1.4	0.6	3.3	2.2	2.8	4.3	3.3	4.0	5.0	2.0	5.1	0.7	1.0	0.5	5.3	2.3	4.8	0.1	0.3	0.0	0.2	0.5	0.0	0.5	0.8	0.0	0.4	1.0	0.0
Green R-PET	1.1	1.7	0.0	3.1	1.6	2.8	1.8	2.2	1.1	4.9	2.1	4.8	0.7	0.8	0.6	4.5	2.2	4.4	0.4	1.0	0.0	0.3	0.7	0.0	0.5	1.1	0.0	0.7	1.4	0.0
Green 100% R-PET	0.7	1.2	0.0	3.1	2.8	2.3	0.8	1.6	0.0	4.5	1.9	4.0	0.6	0.5	0.8	4.1	2.6	4.7	0.3	0.7	0.0	0.4	0.9	0.0	0.7	1.2	0.0	0.4	1.0	0.0

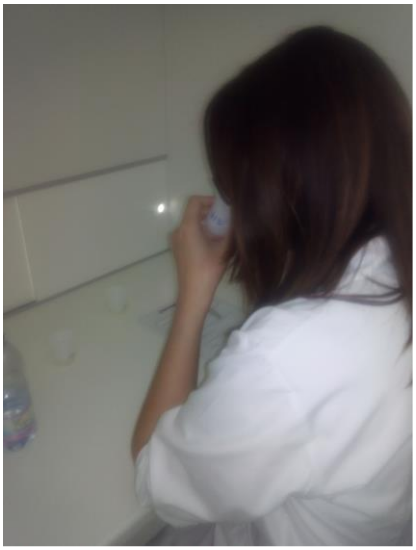

Photo 3. *Sheet used and taster in the panel room during a triangular test carried out on sunflower oils bottled in PET and PCR-PET and stored in oven at 40°C for several days.*

Table 5. *Results of the triangular tests carried out on oils packed in PET and in R-PET and stored stored in oven at 40°C for several days.*

	Correct answers	Preference Clear	Preference Clear R-PET	Indifferent preference	Result
Clear vs Clear 50% PET					
10 days	25%	38%	25%	38%	No significative difference (p> 0.95)
40 days	33%	33%	33%	33%	No significative difference (p> 0.95)
90 days	38%	25%	38%	38%	No significative difference (p> 0.95)
White vs White 50% PET					
	Correct answers	**Preference White**	**Preference White R-PET**	**Indifferent preference**	**Result**
10 days	29%	43%	14%	43%	No significative difference (p> 0.95)
40 days	33%	33%	17%	50%	No significative difference (p> 0.95)
90 days	43%	14%	29%	57%	No significative difference (p> 0.95)
Green vs Green 50% PET					
	Correct answers	**Preference Green**	**Preference Green R-PET**	**Indifferent preference**	**Result**
10 days	29%	14%	29%	57%	No significative difference (p> 0.95)
40 days	29%	29%	29%	43%	No significative difference (p> 0.95)
90 days	50%	67%	17%	17%	No significative difference (p> 0.95)
Green vs Green 100% PET					
	Correct answers	**Preference Green**	**Preference Green R-PET**	**Indifferent preference**	**Result**
10 days	14%	43%	14%	43%	No significative difference (p> 0.95)
40 days	33%	50%	33%	17%	No significative difference (p> 0.95)
90 days	50%	50%	33%	17%	No significative difference (p> 0.95)

Tests results indicated that no particular sensory attribute derived from R-PET. Both polymers are able to preserve the sensory quality of the oils also in stressed storage conditions.

In order to verify the possible migration of volatile substances from the packaging to the oil, the instrumental analysis of oils headspace by Solid Phase Micro Extraction (SPME) sampling and subsequent Gas Chromatography Mass Spectrometry (GC/MS) analysis was also carried out.

The Headspace technology is a technique developed to elucidate the odorous compounds present in the air surrounding various objects also foods.

The main volatile compounds found in oil stored in different PET and PCR-PET and their evolution during oven test are reported in the following tables.

The headspace analyses showed that no particularly volatile compounds derived from the use of R-PET for bottles production. Some volatile compounds deriving from the polymer used (ethylene glycol monovinyl ether, butyl glycol, ethylene glycol, phenol) have been found, but their amount was almost equal in the headspace of the oils and independent of the type of material used (PET and R-PET).

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Table 6. *Main volatile compounds found in headspace of sunflower oils packed in clear bottles in PET and in 50% PCR-PET during oven test.*

Retention Time (min)	Compound name	After 40 days at 40°C		After 90 days at 40°C	
		Clear	Clear R-PET	Clear	Clear R-PET
4,037	pentane	77.2	109.2	221.7	305.3
4,378	hexane	19.3	28.2	54.3	64.5
5,119	heptane	16.4	25.7	29.4	32.8
5,532	cyclohexane	6.7	5.7	3.4	5.4
5,820	n.i.	_	_	2.1	3.1
6,659	octane	46.5	57.8	60.2	65.4
7,413	acetone	8.2		1.5	3.6
7,787	1-octene	19.3	28.5	19.9	21.4
8,297	2-propenal (acrolein)	74.3	81.9	68.1	80.9
8,632	n.i.	15.1	15.9	_	_
8,787	2-octene	_	_	12.7	16.9
9,227	butanal	_	_	2.0	3.0
13,007	pentanal	12.7	17.2	32.1	45.7
14,553	alpha pinene(?)	53.7	47.4	51.5	40.7
15,982	ethylene glycol monovinyl ether	41.1	60.6	10.1	9.0
17,480	hexanal	128.4	180.8	239.6	391.9
20367	n.i.	_	_	_	5.8
22,000	2 octanone/2 heptanone	_	_	3.2	6.3
22,086	heptanal	8.3	11.2	7.8	7.5
23,735	trans 2-hexenal	16.9	19.8	13.7	12.4
23,996	2-penthyl furane	6.3	6.3	4.6	3.4
24,576	1-pentanol	21.2	24.5	28.5	29.7
25,873	n.i.	3.7	5.3	2.4	1.8
26,467	2-octanone	_	_	1.7	1.1
26,590	octanal	4.0	4.9	3.0	2.8
27,179	n.i.	9.4	6.9	2.8	3.0
28,255	trans 2-heptenal	132.1	178.8	91.5	76.9
28,730	n.i.	_	_	_	1.2
28,851	1-hexanol	_	2.2	3.2	3.3
29,818	n.i.	9.6	14.5	3.4	4.5
30,859	nonanal	27.5	37.6	7.9	2.8
31,147	butyl glycol	_	_	3.0	1.7
32,498	trans 2-octenal	9.3	13.9	6.2	8.3
32,653	3-octenol	12.4	17.4	13.7	11.7
33,558	n.i.	6.5	3.2	3.4	2.3
33,974	furfural	2.7	4.3	_	_
34,137	ethylhexanol	10.5	14.2	12.9	7.3
35,153	n.i.	_	_	3.6	_
35,418	n.i.	4.8	5.9	1.6	2.5
36,348	n.i.	4.5	5.6	4.6	3.3
36,487	trans 2-nonenal	7.6	5.3	5.0	3.4
40,253	ethylene glycol	3.4	4.5	9.7	1.7
41,182	hexanoic acid, vinyl ester	_	_	1.1	1.8
42,385	n.i.	_	_	_	_
43,233	n.i.	_	_	6.3	5.3
44,475	n.i.	_	_	1.1	1.1
46,053	trans trans 2,4-decadienal	6.2	3.7	2.8	1.7
51,910	phenol	11.9	25.4	_	_

Table 7. *Main volatile compounds found in headspace of sunflower oils packed in white bottles in PET and in 50% PCR-PET during oven test.*

Retention Time (min)	Compound name	After 40 days at 40°C		After 90 days at 40°C	
		White	White R-PET	White	White R-PET
4,037	pentane	91.5	97.2	231.3	262.0
4,378	hexane	21.9	18.1	50.3	56.4
5,119	heptane	24.5	28.2	42.0	40.1
5,532	cyclohexane	15.6	11.7	3.9	4.3
5,820	n.i.	_	_	1.7	2.5
6,659	octane	60.5	57.2	63.5	70.4
7,413	acetone	_	_	2.4	2.5
7,787	1-octene	23.3	24.0	24.4	24.3
8,297	2-propenal (acrolein)	86.2	90.2	74.5	73.0
8,632	n.i.	18.7	9.7	_	_
8,787	2-octene	_	_	14.4	17.2
9,227	butanal	_	_	1.8	2.1
13,007	pentanal	16.7	15.0	38.8	39.7
14,553	alpha pinene(?)	102.7	65.9	35.8	11.2
15,982	ethylene glycol monovinyl ether	59.0	48.4	9.8	7.7
17,480	hexanal	203.0	223.1	326.2	323.1
20367	n.i.	_	_	_	4.2
22,000	2 octanone/2 heptanone	7.5	8.0	3.7	5.8
22,086	heptanal	10.6	13.1	9.1	11.4
23,735	trans 2-hexenal	26.7	26.6	17.5	17.8
23,996	2-penthyl furane	8.4	6.7	5.4	7.4
24,576	1-pentanol	24.1	25.6	27.4	25.8
25,873	n.i.	6.1	6.9	2.1	2.6
26,467	2-octanone	_	3.4	1.6	1.3
26,590	octanal	7.7	5.9	4.7	3.6
27,179	n.i.	10.0	5.3	3.7	4.0
28,255	trans 2-heptenal	190.6	204.3	90.6	93.5
28,730	n.i.	_	_	_	1.5
28,851	1-hexanol	_	3.7	3.9	4.1
29,818	n.i.	_	2.4	2.7	3.8
30,859	nonanal	15.9	18.6	7.2	5.2
31,147	butyl glycol	_	_	6.4	3.9
32,498	trans 2-octenal	13.2	16.7	7.0	12.8
32,653	3-octenol	20.4	21.6	14.1	16.1
33,558	n.i.	_	4.6	3.0	2.4
33,974	furfural	6.1	_	_	_
34,137	ethylhexanol	_	_	21.4	13.3
35,153	n.i.	_	_	7.7	_
35,418	n.i.	_	_	2.2	2.1
36,348	n.i.	8.8	8.1	4.3	4.5
36,487	trans 2-nonenal	9.2	10.4	6.4	5.4
40,253	ethylene glycol	_	_	1.9	2.4
41,182	hexanoic acid, vinyl ester	_	_	3.9	4.2
42,385	n.i.	_	_	3.5	1.4
43,233	n.i.	_	_	2.3	3.8
44,475	n.i.	_	_	_	1.8
46,053	trans trans 2,4-decadienal	11.7	8.8	4.0	3.5
51,910	phenol	_	_	6.0	2.7

Table 8. *Main volatile compounds found in headspace of sunflower oils packed in green bottles in PET, 50% PCR-PET and in 100% R-PET during oven test.*

Retention Time (min)	Compound name	After 40 days at 40°C			After 90 days at 40°C		
		Green	Green R-PET	Green R-PET 100%	Green	Green R-PET	Green R-PET 100%
4,037	pentane	88.4	90.1	93.3	269.3	204.0	329.6
4,378	hexane	23.7	27.4	29.1	64.9	43.7	65.4
5,119	heptane	13.5	27.4	17.9	46.6	47.4	54.4
5,532	cyclohexane	6.8	13.3	9.9	4.7	3.8	5.4
5,820	n.i.	_	_	_	1.7	5.2	2.9
6,659	octane	54.0	56.4	52.4	58.8	85.3	59.7
7,413	acetone	_	_	_	2.5	_	3.1
7,787	1-octene	20.9	23.7	23.3	23.7	24.9	18.9
8,297	2-propenal (acrolein)	69.6	85.3	88.1	69.3	73.4	76.5
8,632	n.i.	29.1	_	27.2	_	_	14.0
8,787	2-octene	_	20.4	_	16.4	16.6	_
9,227	butanal	_	_	_	1.7	1.5	1.7
13,007	pentanal	15.3	15.6	15.6	38.8	46.5	37.3
14,553	alpha pinene(?)	96.6	40.3	79.6	14.7	17.9	13.4
15,982	ethylene glycol monovinyl ether	48.3	62.2	163.1	7.5	12.5	8.8
17,480	hexanal	239.3	174.1	154.3	297.2	447.4	260.2
20367	n.i.	_	_	_	_	4.4	2.7
22,000	2 octanone/2 heptanone	7.0	4.0	11.3	3.4	8.0	2.7
22,086	heptanal	11.8	10.9	13.6	9.2	12.6	7.8
23,735	trans 2-hexenal	26.8	27.0	28.8	15.0	26.6	14.8
23,996	2-penthyl furane	9.2	10.5	_	5.2	12.2	4.2
24,576	1-pentanol	28.4	27.9	28.3	26.8	33.5	26.9
25,873	n.i.	8.8	3.7	7.1	_	_	2.5
26,467	2-octanone	_	4.7	9.0	_	2.2	0.9
26,590	octanal	6.1	3.0	6.8	_	9.6	2.6
27,179	n.i.	6.8	_	9.5	_	5.9	3.1
28,255	trans 2-heptenal	176.5	147.0	256.2	78.2	123.7	81.2
28,730	n.i.	_	1.5	_	_	4.4	1.1
28,851	1-hexanol	_	3.1	_	3.0	6.9	3.3
29,818	n.i.	_	5.4	30.4	3.0	6.9	4.0
30,859	nonanal	24.9	30.9	55.8	5.9	16.8	5.0
31,147	butyl glycol	_	_	12.1	2.6	21.2	1.7
32,498	trans 2-octenal	13.6	9.8	19.5	5.1	22.3	8.5
32,653	3-octenol	18.7	15.8	30.7	12.2	22.0	13.0
33,558	n.i.	4.5	5.0	4.7	2.7	4.1	2.8
33,974	furfural	_	3.0	9.1	3.7	5.5	_
34,137	ethylhexanol	8.4	10.7	46.1	7.8	64.5	4.2
35,153	n.i.	_	6.2	20.8	_	11.5	3.8
35,418	n.i.	_	3.8	15.6	1.1	5.6	1.5
36,348	n.i.	8.5	5.4	9.5	4.4	12.7	4.1
36,487	trans 2-nonenal	7.9	7.2	12.1	5.2	17.3	3.9
40,253	ethylene glycol	_	_	12.8	7.3	8.0	2.0
41,182	hexanoic acid, vinyl ester	_	_	_	2.8	9.0	2.3
42,385	n.i.	_	_	10.2	2.0	48.4	_
43,233	n.i.	_	_	91.9	3.2	_	2.6
44,475	n.i.	8.1	_	46.3	_	6.9	_
46,053	trans trans 2,4-decadienal	10.7	5.0	12.4	2.1	10.6	2.5
51,910	phenol	_	_	18.1	_	34.3	_

Volatile compounds deriving from the oxidation of fatty acids, mainly linoleic acid, were also found. Aldehydes (pentanal, hexanal, 2-eptenal, 2-ottenal, 2-nonenal, 2,4 decadienal, nonanal) and hydrocarbons (pentane, heptane and octane) are the most abundant compounds of the oils headspace. Only little and non-significant differences for some of these compounds were detected in oils stored in different bottles. Their quantity varies in the oils mainly, as expected, due to increase of the time that they are kept at 40°C.

In conclusion, the data which are collected during the storage tests showed that the packaging in R-PET does not affect the quality of the bottled oil also in stressed conditions.
The same behavior concerning oxidation indices, volatiles compounds, nutritional parameters and organoleptic quality was noticed during accelerated shelf-life test in oils stored in R-PET and in virgin PET.
This trend was confirmed also in shelf-life test carried out at room temperature and under diffused light as described below.

Tests at room temperature (Photo 2)

Commercial sunflower oil was packed in seven different bottles and stored at 25°C ± 4°C until 6 months. Samples were placed on a shelf to standardize the light intensity received by each bottle. Light intensity was measured by using a digital luxometer ISO-TECH (Taiwan). During the night, light intensity was absent. During the day, the average light intensity was 400 LUX, in consideration to the natural (sunlight) and artificial light (neon lamps). The experiment took place for 6 months, and samples were taken each 3 month. For each sampling time, 3 bottles were opened and analyzed.

The following table resumes the experimental plan:

TEST IN ACCELERATED CONDITIONS (room temperature (23 ± 5°C and diffused light)			
Bottles	**Duration of the test**	**Analysis on the oils**	**Sampling**
Clear PET	180 days	Free Acidity, Peroxides value, spectrophotometric indices, vitamin E, Panel test, color, volatiles compounds	0, 90, 180 days
Clear 50% RPET			
White PET			
White 50% RPET			
Green PET			
Green 50% PET			
Green 100%PET			

Photo 4. *PCR-PET and PET bottles filled with sunflower oil stored on a shelf for 180 days at room temperature (23±5°C) in diffuse lighting condition for the shelf-life test.*

Materials and methods used for the analysis of bottles oils were the same of those reported for the accelerated test. The following figures showed the main results of the test:

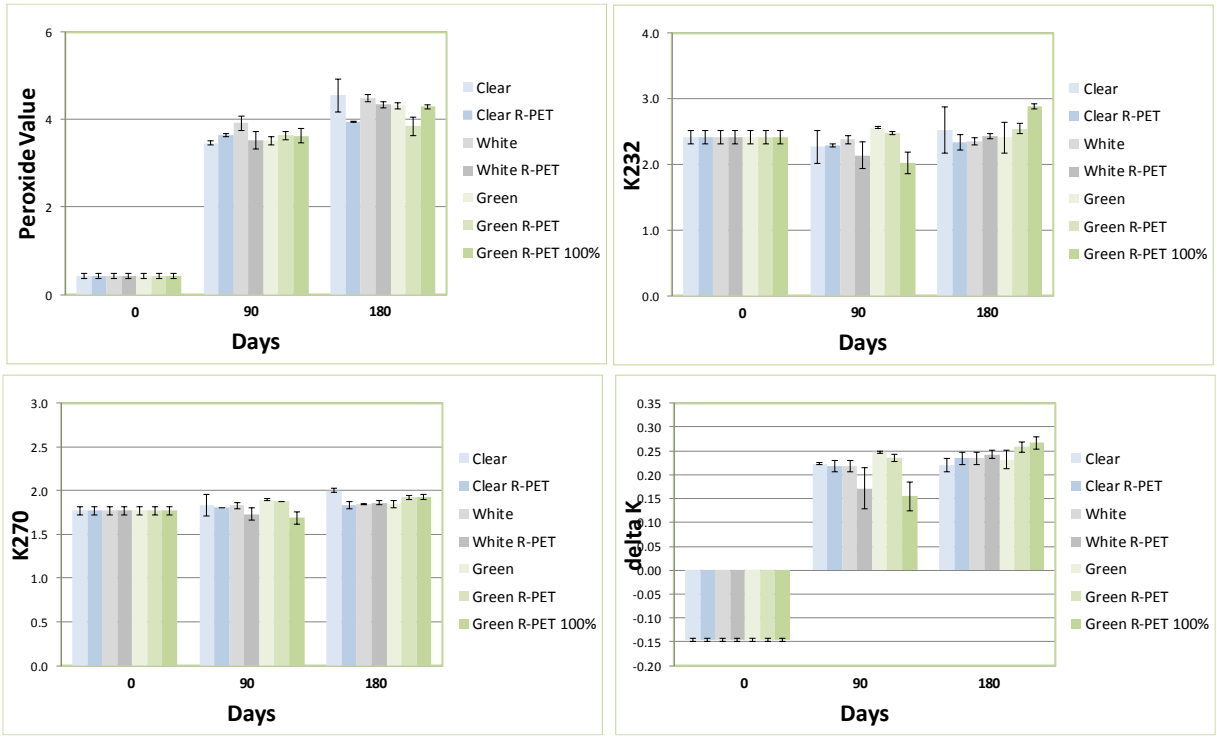

Figure 3. *Evolution of Peroxide Value, K232 (conjugated dienes index of primary oxidation products), K270 and delta k (indices of secondary oxidation products) in oils packed in different bottles during the shelf–life test (room temperature and light).*

Quality indices related to the oxidative stability of the oils showed an increase over the months without being influenced by the type of container and polymer (Figure 3).

To highlight any abnormal smells and tastes eventually transferred by R-PET packaging in R-PET, the oils were subjected to organoleptic analysis. As for the test in an oven, the oils at the end of the six months of storage, were tested by descriptive and discriminated testing (triangular test).

As shown by the tables below, differences in sensory parameters relative to the container. were not significant. The data thus confirm that the R-PET used instead of PET for the production of bottles for food oils has no negative effect on the quality of bottled oils in the course of storage.

Table 9. *Results of the descriptive tests carried out on oils packed in different PET and PCR—PET and stored in at room temperature for six months days. In the table the average, the standard deviation and the median of the scores ascribed by panelists to the main sensorial attributes detected in oils are reported.*

	Nut/Hazelnut			Butter			Sunflower seeds			Fluidity			Roasted			Overall Acceptability			Rancid			Painty/Solvent			Burnt			Plastic		
	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median	Average	St.dev	Median
Sunflower oil initial	2.1	1.1	1.9	3.1	1.0	2.6	4.2	1.5	4.2	5.3	1.3	5.3	1.5	0.5	1.4	5.2	1.9	4.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
after 180 days at 25°C																													
Clear	1.6	0.6	0.6	3.1	2.9	2.2	3.9	1.3	3.4	4.6	2.5	3.6	2.0	2.5	0.8	4.2	2.7	3.8	0.5	1.0	0.0	0.5	0.7	0.0	0.7	0.9	0.4	0.3	0.6	0.0
Clear R-PET	1.5	0.2	0.2	3.0	2.8	1.8	3.3	2.1	3.8	4.7	2.5	3.9	1.8	2.0	1.2	4.1	2.2	3.4	0.3	0.7	0.0	0.2	0.4	0.0	0.5	0.8	0.0	0.6	1.0	0.0
White	1.8	2.5	2.5	4.4	2.3	3.6	2.9	2.5	3.5	4.1	2.8	3.5	2.0	0.8	1.9	6.1	1.7	5.2	0.5	0.8	0.0	0.2	0.4	0.0	0.3	0.6	0.0	0.2	0.5	0.0
White R-PET	2.2	2.1	2.1	3.9	2.6	2.8	2.7	2.7	2.6	4.2	2.9	4.1	1.7	0.5	1.9	6.0	1.9	5.3	0.4	0.7	0.0	0.1	0.2	0.0	0.6	1.0	0.0	0.4	0.7	0.0
Green	1.8	2.5	2.5	4.1	2.5	3.5	3.3	2.5	3.1	4.6	2.6	4.2	2.2	1.1	1.6	5.8	2.4	5.0	0.1	0.3	0.0	0.1	0.3	0.0	0.1	0.3	0.0	0.2	0.6	0.6
Green R-PET	1.7	2.5	2.5	4.1	2.5	3.4	3.4	2.5	3.2	4.7	2.8	4.2	2.4	1.6	2.2	5.9	2.4	5.1	0.2	0.4	0.0	0.3	0.5	0.0	0.2	0.4	0.0	0.3	0.7	0.7
Green 100% R-PET	1.7	2.3	2.3	4.2	2.0	3.5	3.5	2.6	3.6	5.0	2.1	4.5	2.3	1.3	2.0	5.8	1.6	5.5	0.1	0.2	0.0	0.1	0.2	0.0	0.1	0.1	0.0	0.2	0.5	0.5

Table 10. *Results of the triangular tests carried out on oils packed in PET and in R-PET and stored stored at room temperature (25°C ±4) and light for six months.*

	Correct answers	Preference Clear	Preference Clear R-PET	Indifferent preference	Result
Clear vs Clear 50% PET					
180 days	33%	17%	50%	33%	No significative difference (p> 0.95)
White vs White 50% PET					
180 days	17%	17%	17%	67%	No significative difference (p> 0.95)
Green vs Green 50% PET					
180 days	50%	17%	50%	33%	No significative difference (p> 0.95)
Green vs Green 100% PET					
180 days	0%	33%	33%	50%	No significative difference (p> 0.95)

Definition of shelf-life

From the discussion of the data presented in this report it was shown that the R-PET is equivalent to virgin PET regarding on the effect of packaging on the evolution of commercial and sensory quality of bottled oils. Therefore in determining the shelf-life, the same considerations made for the oil in PET are to be done also for packaging made in R-PET. The normal shelf life of a sunflower oil is therefore 18-20 months after bottling both PET and R PET.

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