Abstract

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1   Introduction

2   Materials and methods

Two types of Polypropylene (PP) have been poured together in order to obtain a mixture with higher properties with respect to the matrix. For our purpose 15% in weight of flame retardant PP pellet has been added to isotactic PP (PPH-B-10-FB). In Figure 1 is reported the technical datasheet of PPH-B-10-FB. For compression molding a stainless steel plates with a frame of $12 \times 12\,\mathrm{mm}^2$ has been filled with 34 g of the mixture composed by 27.22 g of isotactic PP and 6.68 g of flame retardant PP. For injection compounding 40 g of the mixture have been weighed. In this case the misture was composed by 34.01 g of isotactic PP and 5.99 g of flame retardant PP.

Table III-1. Technical datasheet of the isotactic PP (PPH-B-10-FB).

Sample	Test method	Value
MFI (g/10min)	ASTM D1238 (190 °C and 2.16 kg)	12 ± 2
Molecular weight distribution		broad
Density (g/cm³)	ASTM 1505	0.904
Atacticity	Xylene solubles	5 % max
Vicat softening point (°C)	ASTM D1525-A (9,81 N)	154
Heat deflection temperature	ASTM D648 (455 kPa)	82
Tensile strength at yield (MPa)	D638 (50 mm/min)	35
Elongation at yield (MPa)	D638 (50 mm/min)	14
Flexural modulus (MPa)	ASTM D790 (1.3 mm/min, 1% secant)	1650 max
Rockwell hardness (R scale)	ASTM D785	111

Figure 1: Technical datasheet of PPH-B-10-FB

3   Experimental activity

3.1   Compression molding

Prepared amount of polypropylene mixture has been used for compression molding process. A press by Carver has been used, as illustrated in Figure **??**.
The material to be pressed is put between two stainless steel plates, one having a frame of 12 ×

Figure 2: Carver press for compression molding.

$12\,\text{mm}^2$ in order to produce a square plate of some mm of thickness. To avoid any adhesion with the plates, that are used to evenly distribute the pressure and to confine the melt (since the press is able to provide heat), two Mylar® foils have been put in between the steel and the material. The press has been set to produce a pressure of 8 tons, equivalent to 5.45 MPa, in the frame. The material has undergone an heat treatment under this pressure of 200°C for 10 minutes. After this, the press has been cooled with water circulating in a refrigerating circuit inside of it and the molded piece extracted. Produced plate is then visually analyzed and weighted in order to estimate weight losses.

3.1.1 Compounding (internal mixer)

The process has been carried out with the instrument Thermo Haake Rheomix 600, where the polypropylene mixture undergoes a thermal treatment of 200°C for 10 minutes. Rotation speed has been set at 40 rpm.
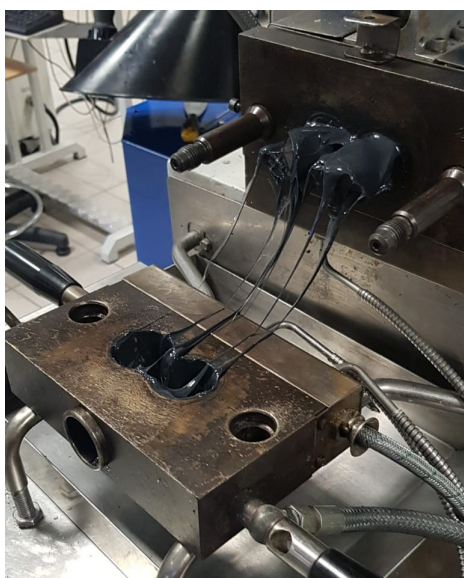

Figure 3: Rheomix 600 after usage. Remains of polypropylene mixture are visible.

The product obtained by this process has then been subjected to compression molding, using the same parameters as Plate 1. Tangential speed of the compounder is estimated to

3.1.2   Production of dumbbell specimens

After the analysis of the plate, this has been cut to obtain ISO 527-1BA shaped specimens, that are used in another laboratory activity.

3.2   Injection molding: sample evaluation

Different specimens of unknown polymeric materials have been analysed and identified. They have been produced by injection molding according to specific standards (ISO and ASTM):

- ISO 10.0 × 4.0 × 172 (mm);

- ASTM 12.7 × 3.3 × 165 (mm);

In Table 1 the types of samples are divided by standard (ASTM and ISO).

Table 1: Materials used in sample evaluation

ASTM dumbbell	ISO dumbbell
ABS	POM bi-injected
COC	PA11 mono-injected
PP	PP-GF 30 (white) and PP-GF35 (black)
HDPE Eltex yellow	PA6-GF50
PE/PP blend	
PA11	

Size of all specimens and the mold cavity have been measured through a caliper in order to evaluate the shrinkage after the process according to Equation 1.

$$\text{shrinkage} = \frac{\Delta x}{x_0} \tag{1}$$

Where Δx is the difference between the initial and final dimension (lenght, width and thickness) and x_0 is the initial dimension (lenght, width and thickness).
ASTM samples (with sprue and bar) have been weighted through the balance Mettler PM 4600 in order to compare the total weight and polymer density (taken from literature [1]).

3.3   Filament analysis

Different fibers provided by University of Trento (polypropylene, see Materials and Methods) have been analyzed: in particular the diameter of six different filament samples have been measured in order to predict their mechanical properties in function of the draw ratio and the process parameters. Diameters have been taken using micrometer [**modello**]. The filament linear density (titer, fineness) is calculated accordingly to the Equation 2.

$$t = \rho \cdot A \tag{2}$$

where t is the titer expressed in dtex, ρ is the density and A is the cross-section of the fiber, considered as circular. Titer in denier is found multiplying t by a factor 0.9. The apparent draw ratio DR is calculated using Equation 3.

$$DR = \left(\frac{D_{max}}{D_{min}}\right)^2 \tag{3}$$

where D_{max} is the maximum diameter of the collected fibers while D_{min} is the minimum diameter. The fiber strength T_{as} (tenacity as spun) is measured in $\frac{cN}{dtex}$ and it's calculated accordingly to the Equation 4.

$$T_{as} = \frac{\sigma_Y}{100 \cdot \rho} \tag{4}$$

The tenacity after drawing T_{DR} is given by Equation 5.

$$T_{DR} = T_{as} \cdot DR \tag{5}$$

The tensile strength σ_b of the fibers is calculated accordingly to Equation 6.

$$\sigma_b = T_{DR} \cdot \rho \cdot 100 \tag{6}$$

where ρ is expressed in g/cm^3.

4   Results and discussion

4.1   Compression molding

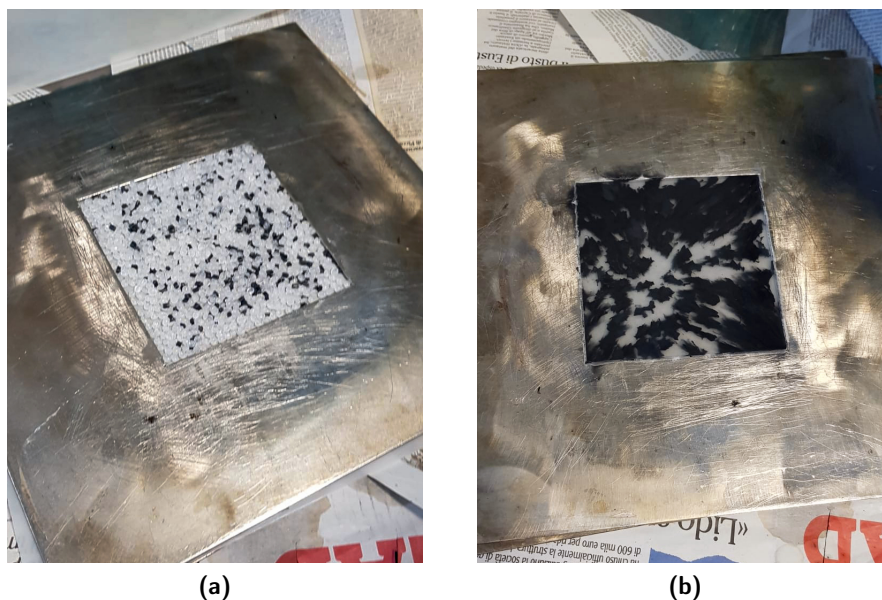The mold before and after pressing is reported in Figure **??**.

Figure 4: Mold (a) before and (b) after compression.

The produced plate after manual mixing is illustrated in Figure **??**.

Figure 5: Plate produced in compression molding.

As it can be easily noted, the distribution of black PP in the white one is absolutely non-homogeneous, typical consequence of a manual mixing of the pellets (without any homogenization mixing in between). The other evident property of the pressed plate is the flash of melt outside of the mold: this is due to the too high amount of polymer inserted in the mold. The weights of the product before and after compression are reported in Table **??**.

Table 2: Mass of the sample before and after pressing.

Mixture mass (g)	Plate mass (g)	Variation (%)
34.00	32.50	−4.41

4.1.1   Compounding (Internal Mixer)

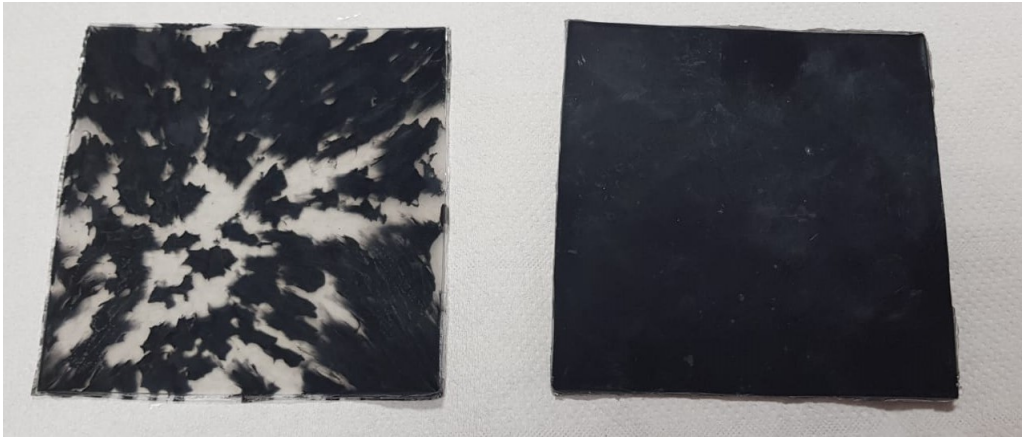A comparison between plates after compression molding si reported in Figure **??**.

Figure 6: Compressed plates: plate 1 (from pellets) at left and plate 2 (from compounding) at right.

It can be seen that subjecting the sample to compounding before compression molding, a better homogeneity can be reached. The weights of the product before and after compression are reported in Table 3.

Table 3: Mass of the samples before and after pressing.

Sample	Mixture mass (g)	Plate mass (g)	Variation (%)
1	34.00	32.50	-4.41
2	32.05	30.50	-4.84

The table shows that the weight variation is slighty highier, that can be explained by the more difficult preparation of the sample before compression, due to its volume distribution.

4.1.2   Production of dumbbell specimens

In Figure **??**-a the dumbbell specimens produced in compression molding without compounding are displayed. As it can be seen, these specimens are characterized by low isotropy and homogeneity of the mixture. This will be a probable issue when mechanically testing and in resistance to flame propagation. Since the flame retardant is a weakener of strength (it introduces organics with poor adhesion and thus stress intensifiers), these specimens may have higher deformation at break than the more homogeneous ones produced in melt compounding, showed in Figure **??**-b.

(a)

(b)

Figure 7: Produced specimens from compression molding plate after compounding.

4.2   Injection molding: sample evaluation

ISO specimens are reported in Figure 8.

(a)   **(b)**

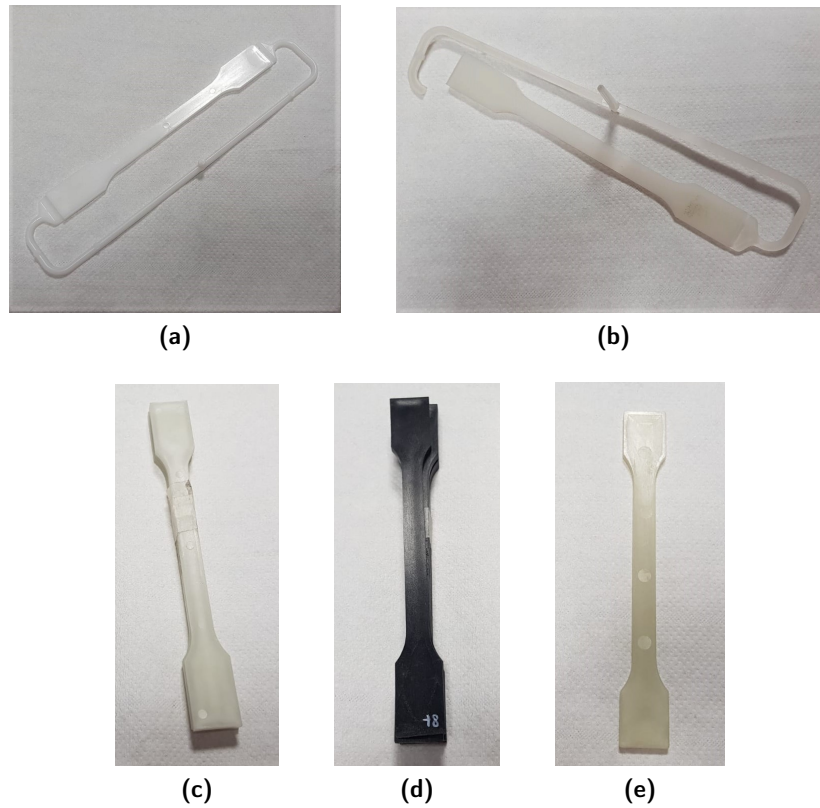**(c)**   **(d)**   **(e)**

Figure 8: ISO samples: a) POM; b) PA11; c) PP-GF30; d) PP-GF35; e) PA6-GF50.

In Table 4 different types of ISO specimens are classified with their sizes.

Table 4: ISO specimens and characteristics.

Figure	Material	Size (mm)	Description
a	POM	$9.74 \times 3.94 \times 167.06$	white and presence of cold junction
b	PA11	$9.86 \times 4.05 \times 168.98$	opaque and white
c	PP-GF30	$9.88 \times 4.00 \times 172.11$	white and stiff
d	PP-GF35	$9.80 \times 4.00 \times 171.86$	black and stiff
e	PA6-GF50	$9.99 \times 3.98 \times 154.71$	very stiff

In Table 5 the values of the shrinkage of ISO samples are reported.

Table 5: Shrinkage of ISO samples.

Samples	Longitudinal shrinkage	Transversal shrinkage	Thickness
POM	0.028	0.026	0.015
PA11	0.017	0.014	-0.012
PP-GF30	0	0.012	0
PP-GF35	0.001	0.020	0

From Table 5 it can be noticed that in reinforced polymers the shrinkage is very small, almost negligible. The presence of glass fibers gives to polymers better dimensional stability during cooling.
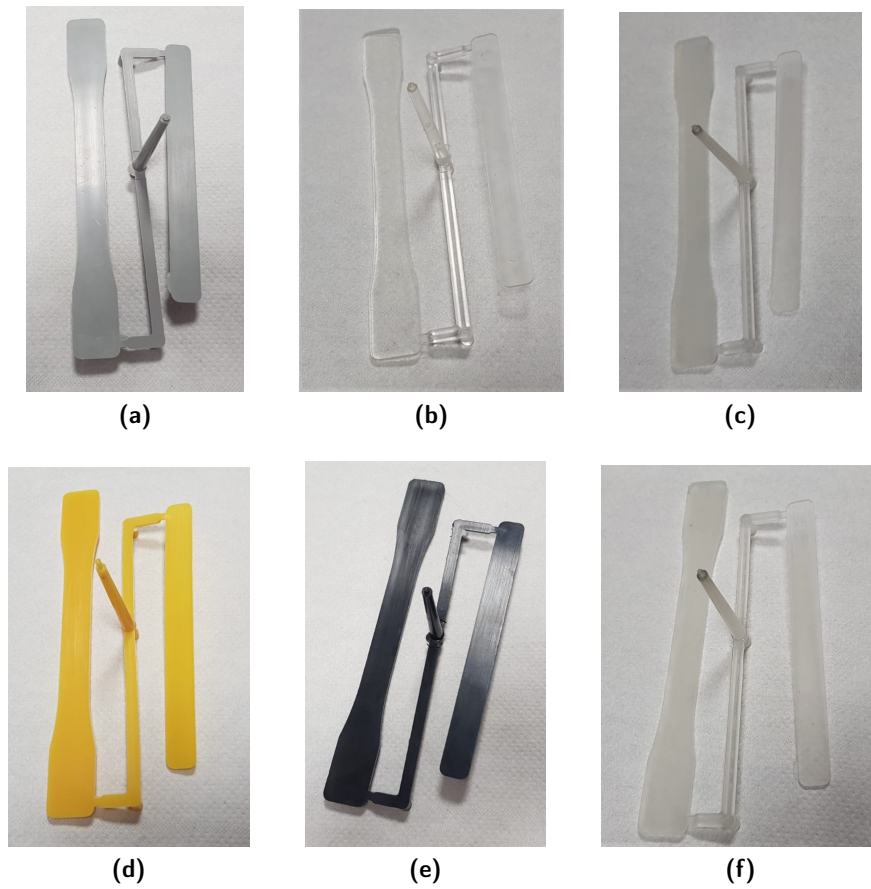
ASTM specimens are reported in Figure 9.

Figure 9: ASTM samples: a) ABS; b) COC; c) PP; d) HDPE; e) PE/PP blend; f) PA11.

In Table 6 different types of ASTM specimens are classified with their sizes.

Table 6: ASTM specimens and characteristics.

Figure	Material	Weight (g)	Size (mm)	Description
a	ABS	14.78	$12.77 \times 3.27 \times 163.55$	grey and flexible
b	COC	14.07	$12.64 \times 3.30 \times 164.04$	transparent and glassy
c	PP	12.28	$12.6 \times 3.35 \times 162.29$	opaque and flexible
d	HDPE	12.73	$12.54 \times 3.33 \times 160.0$	yellow and very flexible
e	PE/PP blend	13.30	$12.66 \times 3.31 \times 161.58$	matt black and flexible
f	PA11	14.00	$12.6 \times 3.35 \times 162.29$	very similar to PP

In Table 7 the values of the shrinkage of ASTM samples are reported.

Table 7: Shrinkage of ASTM samples.

Samples	Longitudinal shrinkage	Transversal shrinkage	Thickness
ABS	0.009	−0.006	0.009
COC	0.006	0.005	0
PP	0.016	0.003	−0.015
HDPE	0.030	0.013	−0.009
PE/PP blend	0.021	0.002	−0.003
PA11	0.016	0.003	−0.015

From Table 7 it can be observed that semycristalline polymers (such as PP, HDPE, PA11) have higher values of shrinkage respect to amourphous polymers (such ABS and COC). Amorphous polymers have random arrangement of molecules that produces little volume changes thus lower shrinkage. The higher values of longitudinal shrinkage in semycristalline polymers are partially compensated by the increase of the thickness (negative values of thickness shrinkage).
In Table 8 weights and densities of ASTM samples are reported.

Table 8: Weight and density of ASTM samples.

Samples	Weight (g)	Density (g/cm^3)
ABS	14.78	$1.04 - 1.12$
COC	14.07	1.02
PP	12.28	$0.85 - 0.94$
HDPE	12.73	$0.93 - 0.97$
PE/PP blend	13.30	$0.86 - 0.95$
PA11	14.00	1.04

For PE/PP blend it has been considered a blend constituted by PE/PP 50%. From Table 8 it can be noticed that, for the same volume, weights of samples are in accordance with values of densities taken from literature [1].

4.3 Filament analysis

Diameter and titer measurements are reported in Table 10.

Table 9: Diameters and fineness measurements of collected fibers.

Sample	Diameter (μm)	Fineness (dtex)	Fineness (denier)
F1	34	8.2	7.4
F2	87	53.7	48.3
F3	212	319.5	287.6
F4	248	437.0	393.3
F5	147	153.5	138.2
F6	41	11.9	10.7

Tenacities before and after drawing are reported in Table **??**.

Table 10: Diameters and fineness measurements of collected fibers.

Sample	$T_{as}\left(\frac{cN}{dtex}\right)$	DR	$T_{DR}\left(\frac{cN}{dtex}\right)$	σ_b (MPa)
F1	0.304	53.2	16.2	1464
F2	0.304	8.1	2.5	223
F3	0.304	1.4	0.4	38
F4	0.304	1.0	0.3	28
F5	0.304	2.8	0.9	78
F6	0.304	36.6	11.1	1007

Accordingly to literature [**?**] high strength PP fibers can reach values of 2.6 GPa and 3.5% deformation at break. Since tensile strength and deformation at break follow a hyperbolic envelope, by fitting these data with the generic equation of the hyperbole (only parameter is a constant), the following can be found, expressed in Figure **??**.
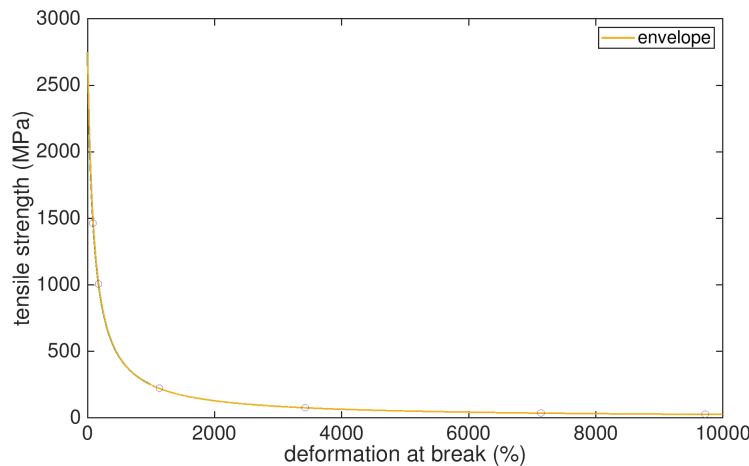

Figure 10: Stress-strain relation for PP fibers.

As it can be easily noted, the distribution of black PP in the white one is absolutely non-homogeneous, typical consequence of a manual mixing of the pellets (without any homogenization mixing in between). The other evident property of the pressed plate is the flash of melt outside of the mold: this is due to the too high amount of polymer inserted in the mold.

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References

[1] Polymer handbook, J. Brandrup, E.H. Immergut, fourth edition, Wiley 2003.