Abstract

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1 Introduction

2 Experimental activity

2.1 Compounding (Internal Mixer)

The process has been carried out with the instrument Thermo Haake Rheomix 600.
The processing parameters used are the following:

- Temperature T = 200°C
- time t = 10 min
- Rotation speed $\omega = 50$ rpm

The sample used has the same composition as Plate 1 sample with a mass of 32.05 g.

2.2 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 **??**.

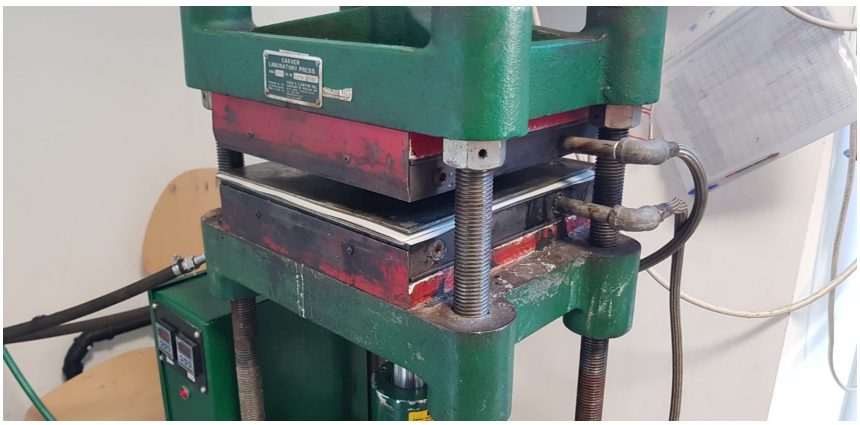


Figure 1: Carver press for compression molding.

The material to be pressed is put between two stainless steel plates, one having a frame of 12 × 12 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.

2.2.1 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.

2.3 Injection molding: sample evaluation

Different speciments 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);

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

$$Shrinkage = \frac{\Delta x}{x_0} \tag{1}$$

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]).

2.4 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 $\frac{\text{cN}}{\text{dtex}}$ 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.

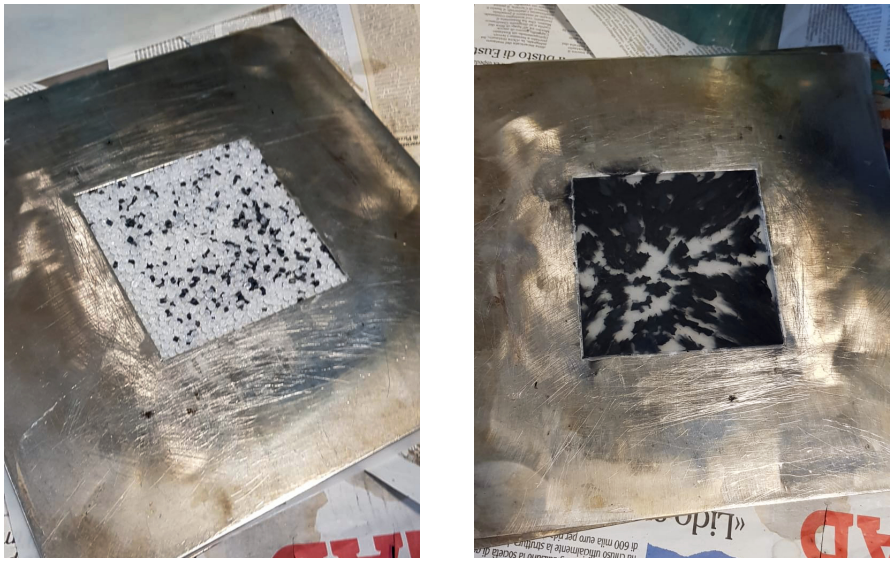
$$\sigma_b = T_{DR} \cdot \rho \cdot 100 \tag{6}$$

where ρ is expressed in g/cm^3.

3 Results and discussion

3.1 Compression molding

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



(a) (b)

Figure 2: Mold (a) before and (b) after compression.

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

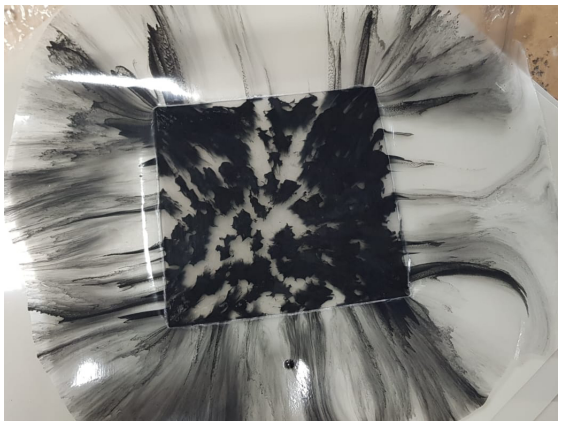


Figure 3: Plate produced in compression molding.

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 1: Mass of the sample before and after pressing.

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

3.1.1 Production of dumbbell specimens

In Figure **??** the dumbbell specimens produced are displayed.

Figure 4: Produced specimens from compression molding plate.

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.

3.2 Injection molding: sample evaluation

ISO speciments are reported in Figure 5.

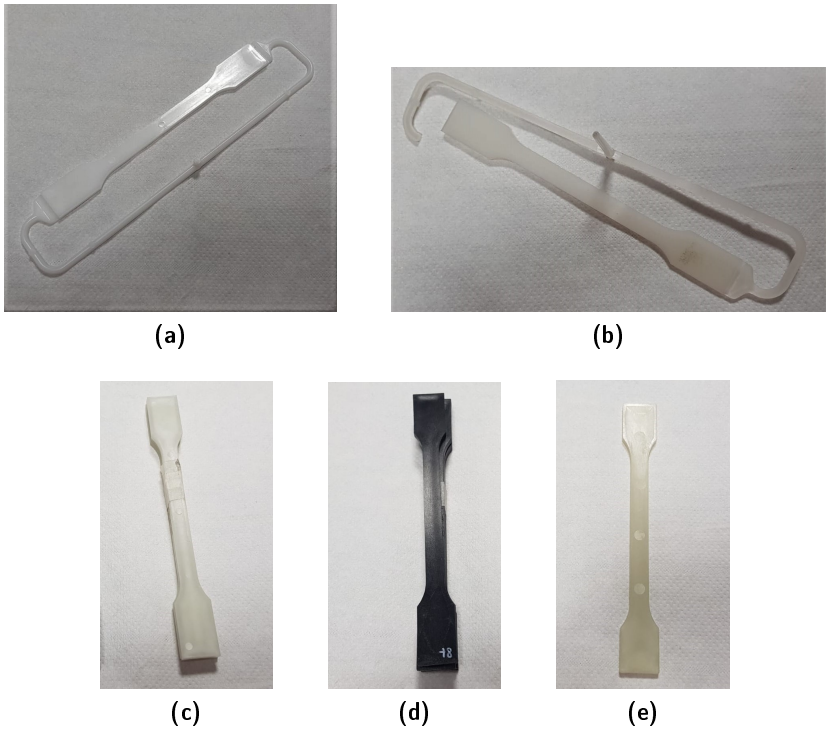


Figure 5: ISO samples: a) POM; b) PA11; c) PP-GF30; d) PP-GF35; e) PA6-GF50.

In Table 2 different types of ISO speciments are classified with their sizes.

Table 2: ISO speciments 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 3 the values of the shrinkage of ISO samples are reported.
From Table 3 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 speciments are reported in Figure 6.
In Table 4 different types of ASTM speciments are classified with their sizes.

In Table 5 the values of the shrinkage of ASTM samples are reported.
From Table 5 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 6 weights and densities of ASTM samples are reported.
For PE/PP blend it has been considered a blend constituted by PE/PP 50%. From Table 6 it can be noticed that, for the same volume, weights of samples are in accordance with values of densities taken from literature.

Table 3: 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

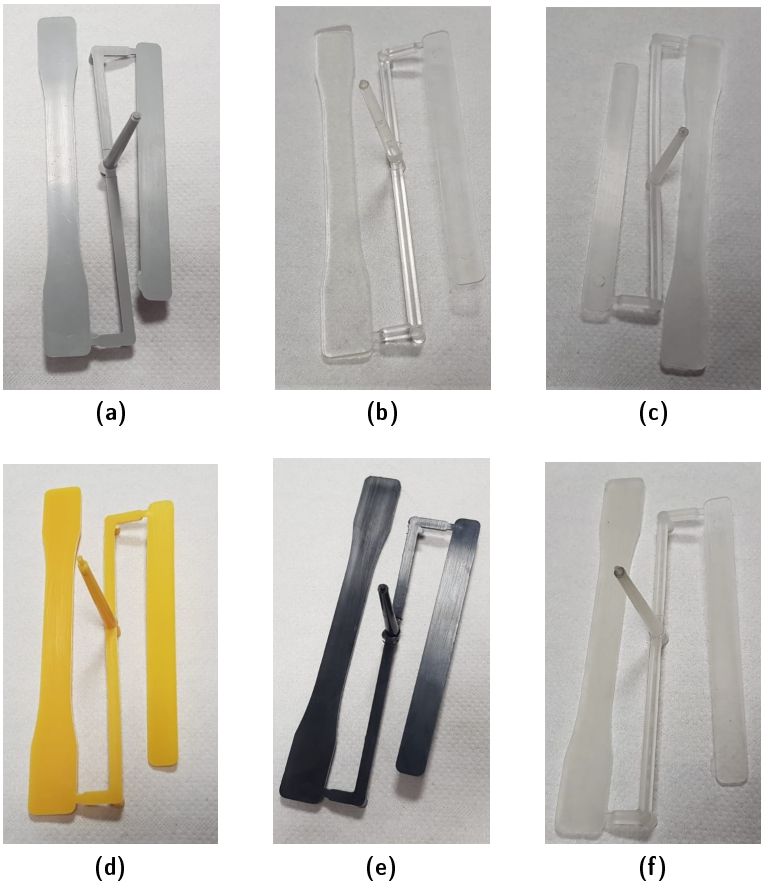


Figure 6: ASTM samples: a) ABS; b) COC; c) PP; d) HDPE; e) PE/PP blend; f) PA11.

Table 4: ASTM speciments and characteristics.

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

Table 5: 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

Table 6: 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	13.3	0.86 − 0.95
PA11	14.00	1.04

3.3 Filament analysis

Diameter and titer measurements are reported in Table 8.

Table 7: 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 **??**.
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 **??**.

References

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

Table 8: Diameters and fineness measurements of collected fibers.

Sample	$\mathbf{T_{as}}$ $(\frac{\text{cN}}{\text{dtex}})$	**DR**	$\mathbf{T_{DR}}$ $(\frac{\text{cN}}{\text{dtex}})$	$\sigma_\mathbf{b}$ (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

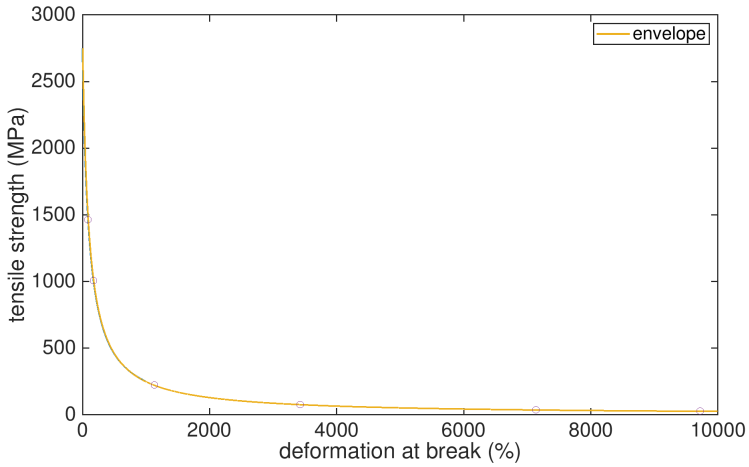


Figure 7: Stress-strain relation for PP fibers.