




Recyclability of a bio-based biocomposite under different reprocessing conditions

Vincenzo Titone¹, Francesco Paolo La Mantia^{1,2,*} , and Luigi Botta^{1,2}

¹ *Departement of Engineering, University of Palermo, Viale delle Scienze, 90128 Palermo, Italy*

² *INSTM, National Interuniversity Consortium for Material Science and Technology, Via Giusti 9, 50125 Florence, Italy*

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ABSTRACT

The present paper studies the effect of reprocessing, on both dry and wet reprocessing conditions, on the rheological and mechanical properties of a biocomposite made of a bio-based, biodegradable polymer with 40 wt% wood flour. The results show that the viscosity of the matrix decreases as the number of extrusions increases, with the greatest reduction for samples processed under wet conditions. The same behavior was observed for the biocomposite, but since the viscosity of the biocomposite is much higher than that of the matrix, this causes a greater reduction of molecular weight. Mechanical results show that properties such as tensile strength and elongation decrease as the number of extrusions increases for the matrix. In terms of maximum variations, the tensile strength experiences a decrease of approximately 18%, while the elongation at break shows a more significant reduction of about 40%. Conversely, the biocomposite shows an increase in tensile strength (about 30%) and elongation at break (about 90%) after the first extrusion and then a slight decrease, but with values of tensile strength and elongation higher than those of the virgin biocomposite.

Introduction

Over the years, plastic has become an integral part of our society, supported by its amazing properties as well as by its low cost and easy availability. However, improper disposal has resulted in large volumes of plastic waste in both hemispheres, posing serious environmental and ecological problems, with potentially irreversible consequences [1, 2]. In this regard, researchers and industry have worked hard in recent

decades to reduce plastic pollution by developing biodegradable and renewable alternatives to conventional plastics. However, the main disadvantages of these plastics usually require different filler materials to overcome or at least minimize the low rigidity, toughness and cost of these materials [3, 4].

Biocomposite materials constitute a new class of materials adopted for several applications, including agricultural and food-packaging [5–11]. However, for the purposes of sustainability and sustainable

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Address correspondence to E-mail: francescopaolo.lamantia@unipa.it

E-mail Addresses: vincenzo.titone@unipa.it; luigi.botta@unipa.it

development, the future of new materials is increasingly linked to their recycling possibilities.

Today, indeed, although mechanical recycling of conventional polymers has been widely reported, [11–18] recycling of biodegradable polymers [19–23] and, in particular, biocomposites is still an open field of research [24–27, 29].

Lopez et al. [24] studied the effect of reprocessing by injection molding of composites reinforced with chemitermic paste (CTMP). The results showed the same trends as pristine matrices, with a reduction in tensile, flexural, and impact strength with the number of reprocessing, although maintaining good recyclability. Similarly, Bourmand et al. [25] observed the recycling behavior of a biocomposite L-poly-(lactide)-poly-(butylene-succinate)-flax, emphasizing that this biocomposite has a recycling behavior suitable for three to four cycles. Chaitanya et al. [26] evaluated the recyclability of poly-(lactide)/Sisal biocomposites up to 8 extrusions. The results showed that it is not recommended to go further than the third recycle, but that biocomposites recycled up to the third recycle, the authors emphasize, can be used to make products for low- and medium-strength nonstructural applications. Lagazzo et al. [27] studied the recyclability of poly (3-hydroxybutyrate-co-3-hydroxyvalerate/Sisal and poly-(lactide)/Sisal biocomposites, showing that all the biocomposites become more brittle with recycling, but the properties of the biocomposites are still maintained up to the third cycle, demonstrating their promising recyclability.

Based on the above, given the growing interest in the use of natural fillers in biodegradable polymers, wood flour (WF) emerges as a viable candidate. In fact, in a recent study, Allaf et al. [28] reported interesting results in poly(ϵ -caprolactone)/wood sawdust biocomposites, as well as interesting results on recycling a biocomposites commercial blend (BioFlex F2110) with wood flour (WF) were reported by Morreale et al. [29]

In this paper, a sample of commercially available Bioplast 105 was extruded by using a twin-screw extruder with 40 wt% wood flour (WF). The aim of this paper was to evaluate the effect of reprocessing on the rheological and mechanical properties of the biocomposite using a single-screw extruder. In particular, extrusion cycles were performed on both dry and wet samples in order to evaluate the effect of humidity on the rheological and mechanical properties of the reprocessed materials up to three successive extrusion cycles.

Materials and methods

Materials

The material used in this work was a biopolymer supplied by Biotec® (Emmerich am Rhein, Germany) under the commercial name Bioplast 105, renamed of us BP. The main properties provided by the supplier [30] are as follows in Table 1.

The composition is unknown, but it is presumably a blend of PLA and PBAT with a PLA/PBAT ratio of about 1.5 as the bio-based carbon share is 67%. The wood flour (WF) used in this work was kindly supplied by La.So.Le. (Percoto, Italy). This filler, with a diameter range between 300 and 500 μm , has been used in a previous paper [29].

Manufacturing of composites

An OMC twin-screw extruder (OMC, Saronno, Italy) with a screw diameter of 19 mm and a length-to-diameter ratio of 35 mm was used to prepare the BP-based composite. The filler concentration used was 40% by weight. The temperature profile used was 140–145–150–160–170–180–180 °C (die), screw speed was set at 150 rpm and gravity feed at 10 rpm. The extrusions were carried out on dried conditions. The drying of both BP and WF was performed under vacuum at 60 °C and 80 °C overnight, respectively.

Processing

The processing of Bioplast 105 (BP) and its biocomposite (BP/WF) was performed by using a single-screw extruder (Thermo Scientific HAAKE PolyLab QC, Karlsruhe, Germany). The temperature profile was set at 150–160–170–180 °C, and the screw rotation speed was 60 rpm. Extrusion tests were performed on both dry and wet samples. In the first case, —dry— the material was dried overnight under vacuum at 60 °C before each extrusion. In the second case —wet— the

Table 1 Main properties of Bioplast 105

Name	MFI, g/10 min	Melting point, °C	Bio-based carbon share, %
Bioplast 105	4.1	155–175	67

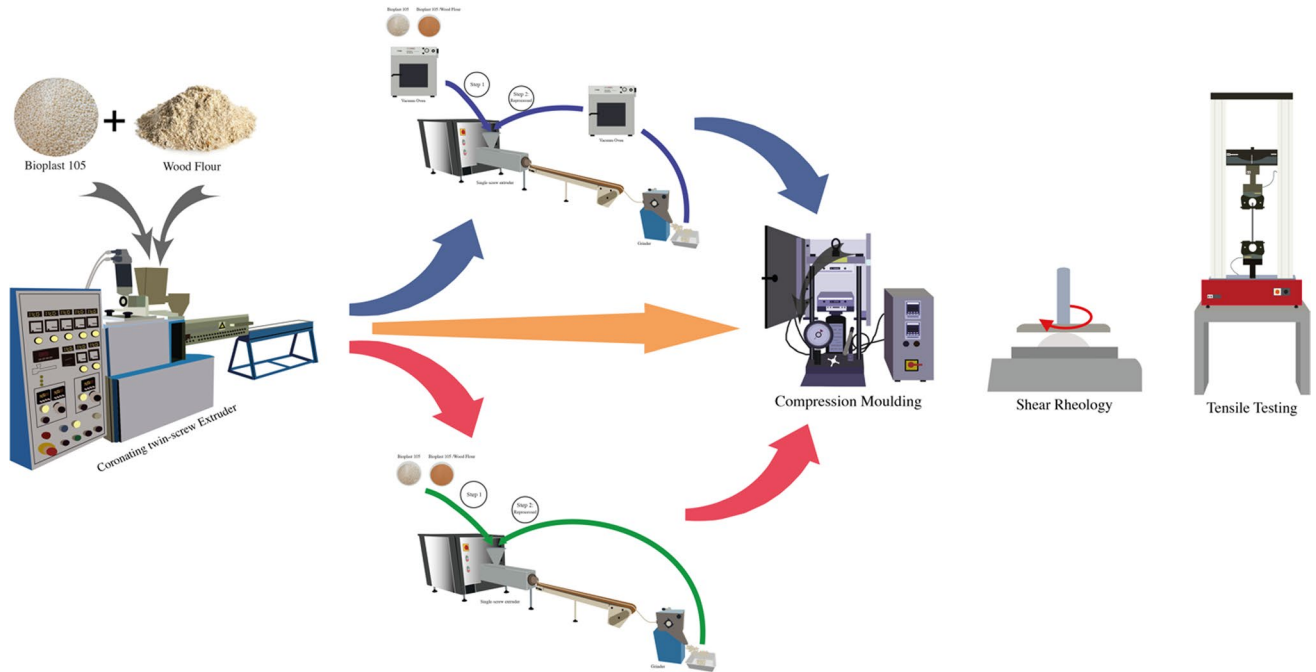


Figure 1 Illustrative images of the procedures used in this work.

Table 2 Blend codes and the processing undergone by each sample

Sample code	Processing	Conditions
BP	Unprocessed	–
d-BP _{EXT}	Extruded 1 time	Dry
d-BP _{RE1}	Extruded 2 times	Dry
d-BP _{RE2}	Extruded 3 times	Dry
w-BP _{EXT}	Extruded 1 times	Wet
w-BP _{RE1}	Extruded 2 times	Wet
w-BP _{RE2}	Extruded 3 times	Wet

Table 3 Bicomposites codes and the processing undergone by each sample

Sample code	Processing	Conditions
BP/WF	Unprocessed	–
d-BP/WF _{EXT}	Extruded 1 time	Dry
d-BP/WF _{RE1}	Extruded 2 times	Dry
d-BP/WF _{RE2}	Extruded 3 times	Dry
w-BP/WF _{EXT}	Extruded 1 times	Wet
w-BP/WF _{RE1}	Extruded 2 times	Wet
w-BP/WF _{RE2}	Extruded 3 times	Wet

both components were kept in ambient conditions (~ 25 °C and ~ 50% RH).

Figure 1 illustrates the processes used in this work.

Tables 2 and 3 show the blend codes of the bicomposites and the processing undergone by each sample, respectively.

Characterization

Rheological measurements were performed on ARES G2 rotational rheometer using 25 mm parallel plates at temperatures of 180 °C with a gap of about 1.5 mm between the plates. The storage modulus, loss modulus and complex viscosity were measured as function

of frequency (0.1–100 rad/s) within the linear viscoelastic regime of the sample.

Mechanical (tensile) tests were performed according to ASTM D638-14 [31] using an Instron universal testing machine (Instron, mod. 3365, High Wycombe, UK). The samples were produced using a Carver laboratory hydraulic press (Carver, Wabash, IN, USA) at a temperature of 180 °C and a mold pressure of 300 psi through compression molding. Afterwards, rectangular strips were cut from the specimens, measuring 90 mm in length, 10 mm in width, and approximately 0.5 mm in thickness.

The tests were performed with a crosshead speed of 1 mm/min up to 3% strain. Thereafter,

the crosshead speed was increased to 100 mm/min until the specimen ruptured. The values of the Elastic modulus, tensile strength and elongation at break are average of at least 8 measurements (data reproducibility: $\pm 7\%$).

Thermal properties were evaluated by differential scanning calorimetry (DSC). DSC measurements were performed by a DSC-131 Setaram (Setaram, Hillsborough Township, NJ, USA), under a nitrogen gas atmosphere, using 10 ± 2 mg of sample and a 10 °C/min heating rate up to 200 °C/min.

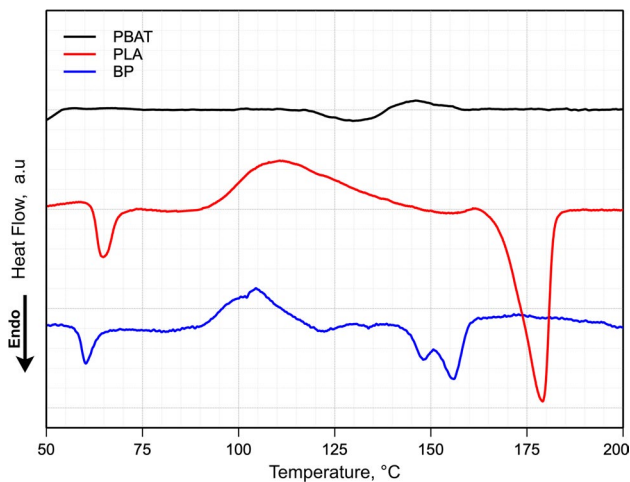
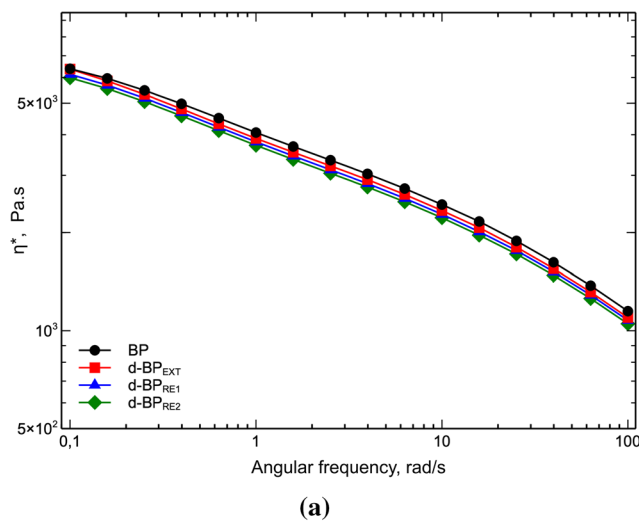


Figure 2 DSC heating curves of neat PBAT, neat PLA and BP.



Results and discussion

Figure 2 shows the thermograms of neat poly(butylene adipate-co-terephthalate) (PBAT), neat poly(lactic acid) (PLA) and BP.

According to Fig. 2, the blend seems made of PBAT and PLA. PBAT is an amorphous polymer with a T_g of about -34 °C and PLA is a semicrystalline polymer with a melting temperature between 160 and 180 °C. However, as reported by Farsetti et al.[32], the presence of PBAT provokes a decrease of the melting temperature of the PLA phase and a split of the melting peak into two peaks as evident in the calorimetric curve of the sample investigated in this work. Because the producer declares a bio-based carbon share of 67% and that only the PLA is a bio-based polymer, it is reasonable consider that the PLA/PBAT ratio is about 3 . Of course, additives could be also present in this blend.

In Fig. 3a, b the complex viscosity curves of all the samples are reported. The viscosity decreases with increasing the number of extrusions and the reduction is larger for the samples processed in wet conditions.

This behaviour is put in evidence in Table 4 where the complex viscosity at the frequency of 0.1 rad/s, the lowest frequency measured, are reported. After the second reprocessing step, and then after three extrusions, the viscosity of the sample processed after drying is reduced only by about 6% , while the reduction of the sample processed in wet condition is reduced of about 17% .

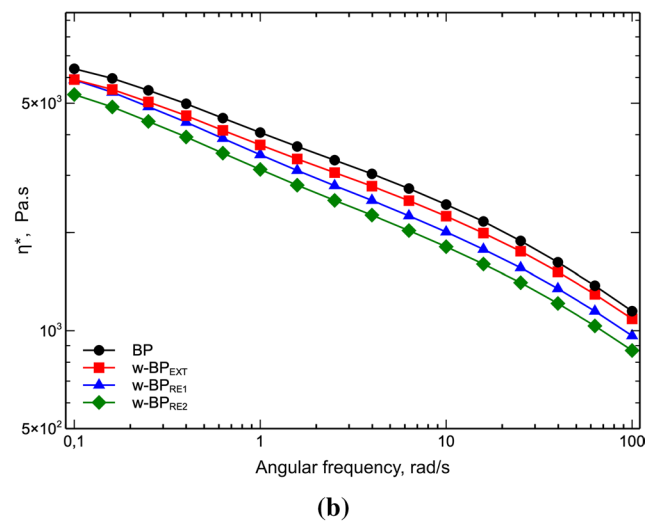


Figure 3 Complex viscosity, η^* , as a function of frequency of BP: (a) d-BP, (b) w-BP.

Table 4 Viscosity values at 0.1 rad/s (Pa s) of BP and the reprocessed samples d-BP and w-BP

η^* at 0.1 rad/s, Pa s	BP		BP _{EXT}	BP _{RE1}	BP _{RE2}
	6392	Dry	6358	6135	5986
		Wet	5912	5901	5316

From these curves it is not possible to put in evidence the Newtonian viscosity which occur at lower frequencies. It is, however, to evaluate the Newtonian viscosity form the Ferry’s equation [33]

$$\frac{1}{\eta} = \frac{1}{\eta_0} + b\tau \tag{1}$$

where τ is the shear stress, and η_0 and η the Newtonian viscosity and the viscosity at a given value of the shear stress respectively. Therefore, plotting $1/\eta$ versus τ , (see Fig. 4) the intercept at $\tau = 0$ is the inverse of the Newtonian viscosity.

Table 5 shows the Newtonian viscosity values obtained according to the Ferry’s equation and the

dimensionless values with respect to the unprocessed matrix: BP is the unprocessed matrix; BP_i is the matrix reprocessed i times.

The decrease of the viscosity means a decrease of the molecular weight of the two components of the blend is, of course, due to the breaking of the macromolecules due to the thermomechanical stress undergone by the macromolecules. The larger decrease of the molecular weight is due to the hydrolytic chain scission undergone by the two polyesters in presence of water.

In Fig. 5 the complex viscosity curves of the biocomposites are reported for all the samples.

The biocomposites do not show any Newtonian plateau and this behavior is typical of composites systems [7, 9, 10]. The upturn shown by these samples at low frequency is very impressive as the viscosity increases of more than one decade with respect to the viscosity of the polymer matrix. A very unusual feature is that for the samples reprocessed in dry conditions the sample reprocessed two and three times and then after two or three extrusions show flow curves higher than the sample processed only one time. Indeed, after a significant decrease,

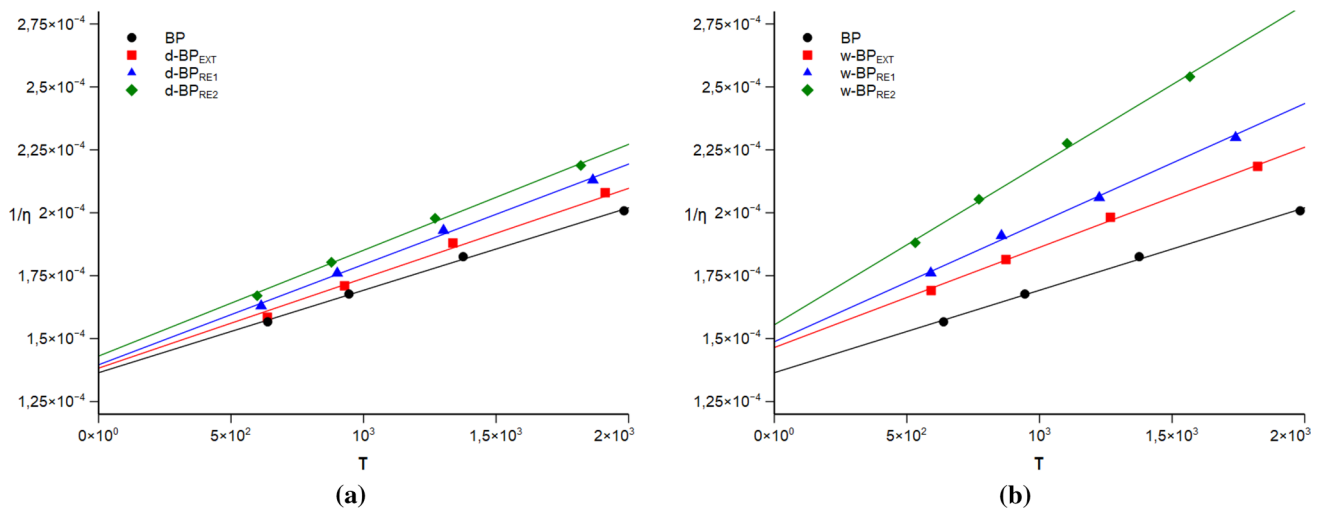


Figure 4 Ferry’s plot of BP and reprocessed sample: (a) d-BP, (b) w-BP.

Table 5 Viscosity values according to Ferry’s equation and dimensionless values versus BF of the reprocessed samples d-BP and w-BP

Conditions	Property	BP/WF _{EXT}	BP/WF _{RE1}	BP/WF _{RE2}
Dry	η_0 according to Ferry’s equation	7246	7194	6993
	η_0 (BP _i)/ η_0 (BP)	0.985	0.978	0.951
Wet	η_0 according to Ferry’s equation	6849	6756	6451
	η_0 (BP _i)/ η_0 (BP)	0.931	0.918	0.877

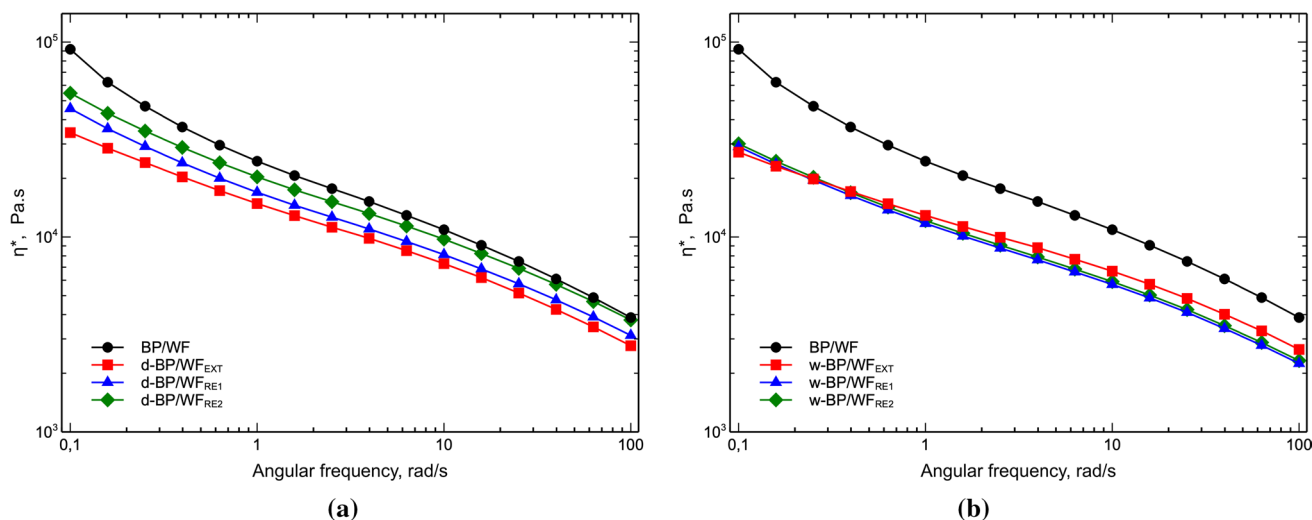


Figure 5 Complex viscosity, η^* , as a function of frequency of BP/WF: (a) d-BP/WF, (b) w-BP/WF.

Table 6 Viscosity values at 0.1 rad/s (Pa s) of BP/WF and the reprocessed samples d-BP/WF and w-BP/WF

η^* at 0.1 rad/s, Pa s	BP/WF	BP/WF _{EXT}	BP/WF _{RE1}	BP/WF _{RE2}
	91844	Dry 34314	45565	54678
		Wet 27226	29078	30067

the viscosity increases with the number of extrusions. This effect is very important for the sample processed in dry conditions and very less in the sample processed in wet conditions, as put in evidence in Table 6 where the viscosity at the lower frequency is reported for all the samples.

As reported above, the thermomechanical degradation is the only driving force of the degradation during processing for the dried sample and this is proportional to the viscosity of the polymer system:

$$\tau = \gamma \times \eta \quad (2)$$

where γ and η are the shear rate and the viscosity in the processing conditions.

The viscosity of the biocomposite is much larger than that of the matrix and this causes a larger reduction of molecular weight. As reported in our previous papers [19, 20], the copolyester PBAT can undergo both chain scission, but also branching and cross-linking during degradation.

In Fig. 6 the curves of the storage modulus, G' , are reported for all the samples.

The slope of the G' curves of the biocomposite processed after drying, dramatically changes with the number of extrusions and, in particular, the slope decreases with increasing the processing steps. This behaviour suggests that the melt becomes more elastic with the processing and this can be due in the melt only by formation of cross-linked structures. Unfortunately, due to the low amounts of both PBAT in the blend and of the cross-kinked structures, a direct measurement of these structures was not possible (Table 7).

The mechanical properties of the matrix and of the biocomposite are reported in Table 8.

The biocomposite is extremely more rigid than the matrix. Indeed, the elastic modulus, E , is strongly enhanced by the presence of the filler, while the elongation at break, EB, is dramatically reduced and the biocomposite is fragile, while the matrix is ductile.

In the Figs. 7, 8 and 9a, b the dimensionless values of E , TS and EB are reported as a function of the number of extrusions. The dimensionless values have been calculated as the ratio between the values at each extrusion divided by the same value of the same virgin polymer system.

The elastic modulus slightly increases with the number of extrusions for the matrix processed in both conditions. On the contrary, the elastic modulus of the biocomposite processed in both conditions decreases after one extrusion and then increases after the two next processing steps.

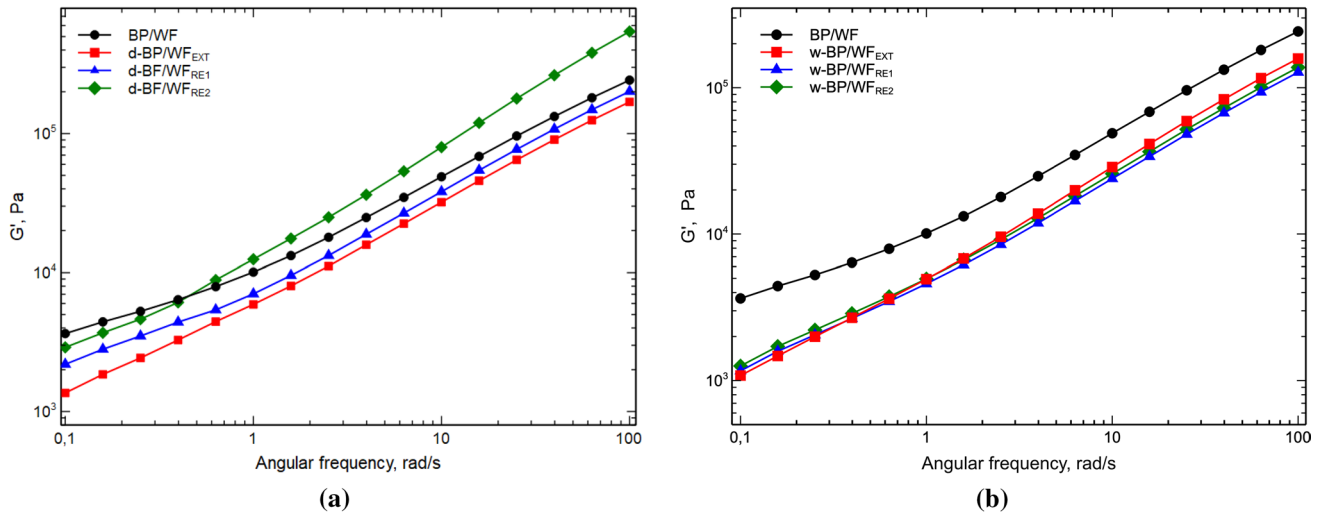


Figure 6 Storage modulus, G' , as a function of frequency of BP: (a) d-BP, (b) w-BP.

Table 7 Slope G' values of BP/WF and reprocessing samples of d-BP/WF

	BP/WF	d-BP/WF _{EXT}	d-BP/WF _{RE1}	d-BP/WF _{RE2}
Slope G'	0.403	0.631	0.534	0.503

Table 8 Elastic modulus (E) tensile strength (TS) and elongation at break (EB) of BP and BF/WF

	E , MPa	TS, MPa	EB, %
BP	183 ± 32	10.9 ± 1.2	209 ± 33
BP/WF	587 ± 83	8.97 ± 1.4	3.2 ± 0.6

Both tensile strength and elongation at break of the matrix processed in both conditions decrease and the decrease is larger for the sample processed in wet conditions. The biocomposite, on the contrary, shows an increase of both TS and EB after the first extrusion and then a decrease. However, even after three extrusions, the values of TS and EB are higher than those of the virgin biocomposite. The behavior of the matrix can be attributed to the chain scission undergone by the macromolecules during the processing for both thermomechanical stress and for the hydrolytic chain scission for the sample processed in wet conditions. The interpretation of the complex behavior shown by the curves

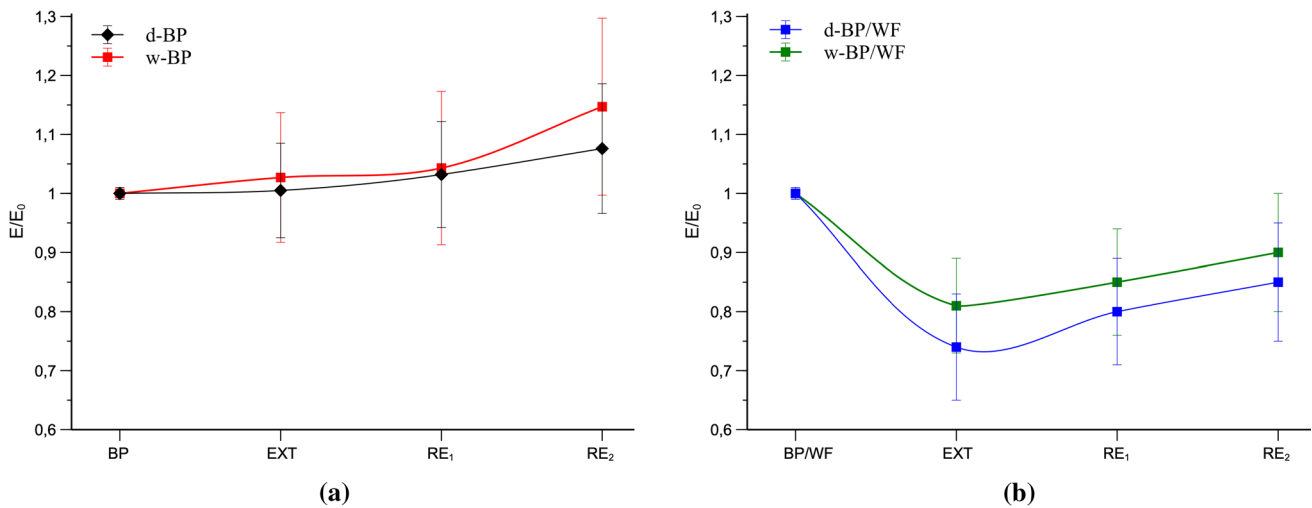


Figure 7 Dimensionless elastic modulus, E , as a function of reprocessing cycles: (a) BP, (b) BP/WF.

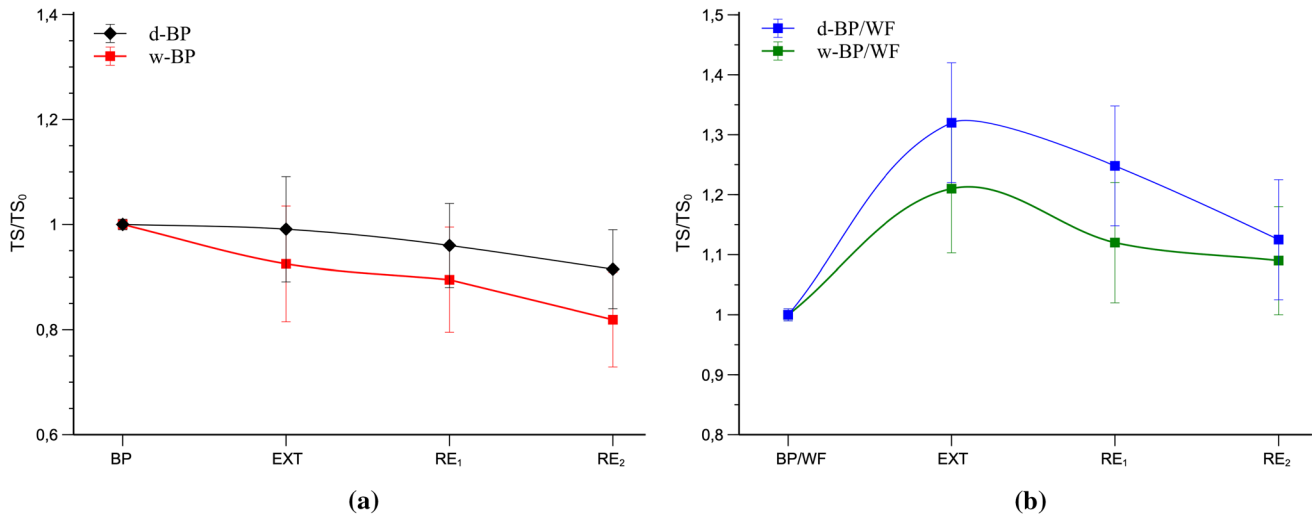


Figure 8 Dimensionless tensile strength, TS, as a function of reprocessing cycles: (a) BP, (b) BP/WF.

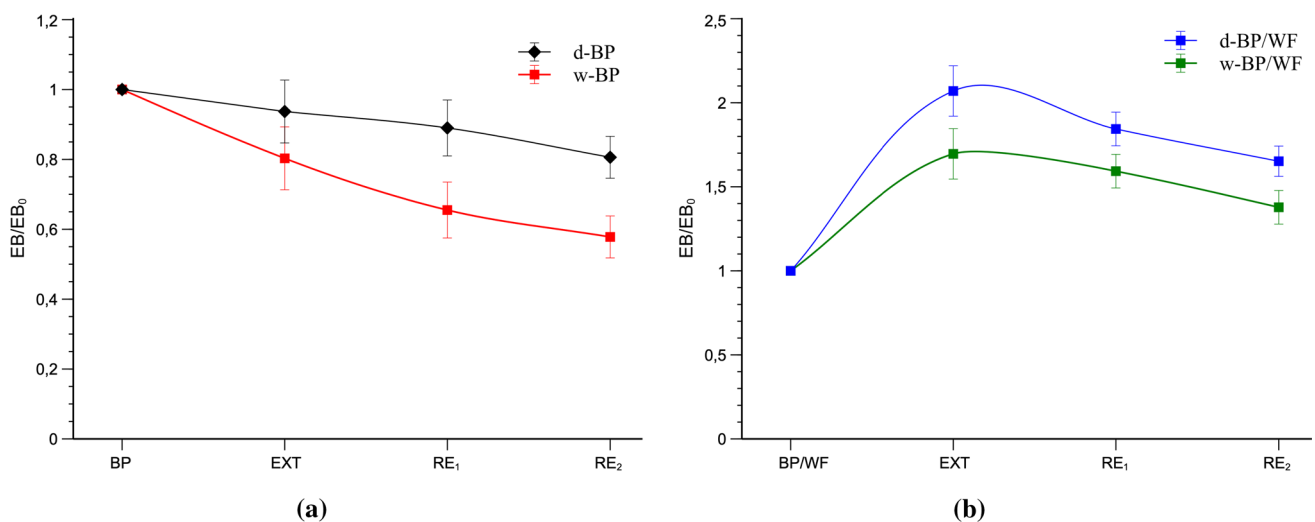


Figure 9 Dimensionless elongation at break, EB, as a function of reprocessing cycles: (a) BP, (b) BP/WF.

of dimensionless tensile strength and elongation at break can be ascribed to the competition between chain scission occurring in both the polymeric components of the biocomposite and on the formation of cross-linking in the PBAT phase, already assumed. Moreover, the lower molecular weight of the PLA phase can give rise to an increase of crystallinity as demonstrated by Fig. 10 and summarize in Table 9.

The competition between the contemporary decrease of molecular weight, increase of crystallinity and formation of cross-linking gives rise to the behavior of the curves reported before.

Conclusions

In this paper, a commercial biodegradable blend with the trade name Bioplast 105 was extruded by using a twin-screw extruder with 40 wt% wood flour. Thereafter, both the matrix and the biocomposite have been reprocessed, both dry and wet, up to three cycles through a single screw extruder. Rheological and mechanical properties have been evaluated. The results show a decrease in molecular weight due to the chain scission of the macromolecules due to the thermomechanical stresses, with a greater decrease

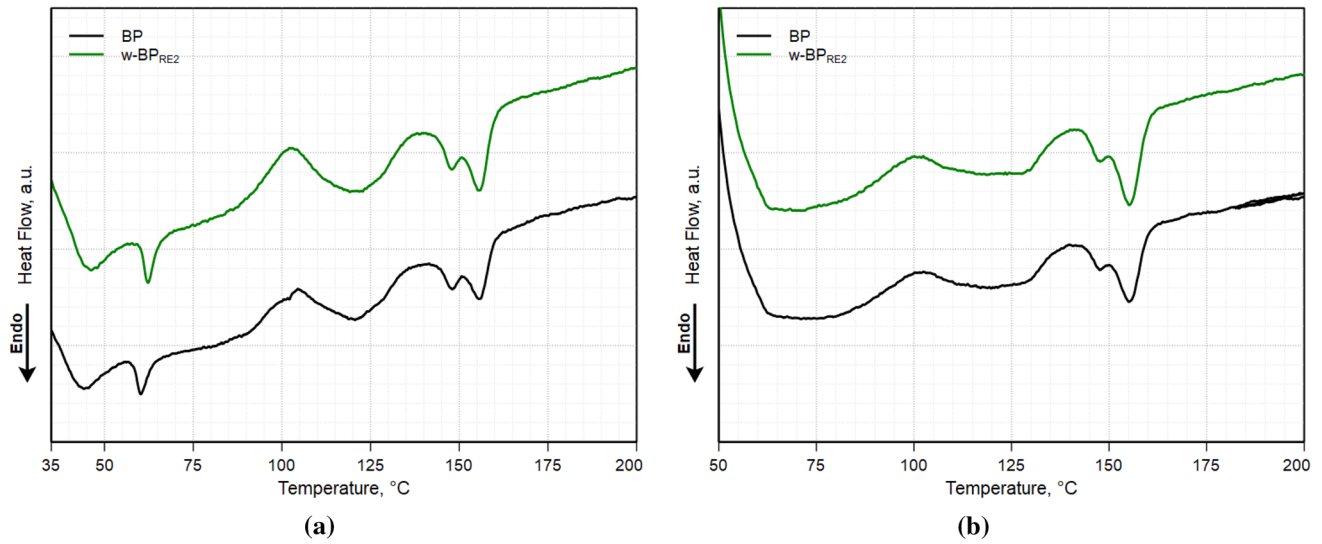


Figure 10 DSC thermograms of the BP and w-BP_{RE2} samples: (a) 1 scans, (b) 3 scans.

Table 9 Data obtained from DSC curves of BP and w-BP_{RE2} samples

Scan	T_g , °C	T_m , °C	ΔH_{cc} , j/g	ΔH_m , j/g
<i>BP</i>				
1	59.8	138.4–165.6	4.76 ± 0.07	3.82 ± 0.05
3	–	139.6–169.9	3.68 ± 0.04	4.43 ± 0.06
<i>w-BP_{RE2}</i>				
1	62.4	139.4–169.0	4.89 ± 0.10	4.35 ± 0.05
3	–	140.1–170.2	3.67 ± 0.02	4.68 ± 0.07

in the case of samples treated in wet conditions, as result of the scission hydrolytic due to the presence of water. Moreover, this decrease of molecular weight is more pronounced in the case of the biocomposite. Mechanical tests showed a decrease in properties as the number of reprocessed increases, mainly in wet samples. Notably, the maximum variations observed were a decrease of 18% in tensile strength and 40% in elongation at break. The biocomposites, on the other hand, showed an increase in mechanical properties after the first extrusion (30% for the tensile strength and 90% for the elongation at break) and a decrease with increasing number of reprocessed, while remaining above the values of unprocessed biocomposites. Ultimately, all investigated samples maintain a good recyclability.

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Author contributions

Conceptualization: FPLM; methodology: FPLM; validation: VT and LB; formal analysis: VT; investigation: VT; data curation: VT; writing—original draft preparation: VT and FPLM; writing—review and editing: FPLM, VT and LB; visualization: LB; supervision: FPLM. All authors have read and agreed to the published version of the manuscript.

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Data and code availability

The data presented in this work are available on request from the corresponding author.

Declarations

Conflict of interest The authors declare no conflict of interest.

Ethical approval Not applicable.

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