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► **To cite this version:**

Fabio A Cruz Sanchez, Silvia Lanza, Hakim Boudaoud, Sandrine Hoppe, Mauricio Camargo. Polymer recycling and additive manufacturing in an open-source context: Optimization of processes and methods. Communication orale. 27th annual international Solid Freeform Fabrication Symposium: an additive manufacturing conference, Aug 2015, Austin, United States. hal-02518430

HAL Id: hal-02518430

<https://hal.science/hal-02518430>

Submitted on 18 Jun 2024

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Polymer Recycling and Additive Manufacturing in an Open Source context: Optimization of processes and methods

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Abstract

Polymer recycling is a way to reduce environmental impacts of accumulation of polymeric waste materials. However, low recycling rates are often observed in conventional centralized recycling plants mainly to the challenge of collection and transportation for high-volume low-weight-polymers in conventional centralized recycling plants. As the democratization of open-source 3D printers is going forward thanks to initiatives such as FabLab environments, there is a growing interest on how to use this technology to improve the efficiency of use of raw materials. Studies have been proposed in order to recycle waste polymer into open-source 3D printer feedstock. The recycling of high-density polyethylene (HDPE) issued from bottles of used milk jugs through use of an open-source filament fabricator system called RecycleBot has been evaluated. In this study, we propose an evaluation of the mechanical recyclability of Polylactic Acid (PLA), material widely used in the open-source 3D printing context, in order to establish the viability of this recycled material to be used in the open-source 3D printers. The degradation of the material's mechanical and rheological properties after a number of cycles of multiple extrusion and printing processes is evaluated. The characterization of recycled raw materials for open-source 3D printing has implications not only to reduce the environmental impact of polymers waste, but also it will allow us to understand the technical requirements and challenges for development of open-source filament recycle machine/process.

The coupling of open-source 3D printers and filament extruders can offer the bases of a new distributed polymer recycling paradigm, which reverses the traditional paradigm of centralizing recycling of polymers where is often uneconomic and energy intensive due to transportation embodied energy. Moreover, this characterization also will allow the exploration of new source of materials and new composite materials for open-source 3D printing, in order to improve the quality of products made by this technology.

1 Introduction

The development of polymer materials has allowed the manufacture of a wide range of low-cost, low weight, high performance products and it has become in a core part of technological and societal development [1]. The plastic industry is almost completely dependent of fossil oil and gas, using about 4% of worldwide oil production which it is translated in approx. 299 million metric tonnes per annum in the year 2012 [2]. One of the main concerns is the environmental impact of plastic residues because the longevity in the environment is not known with certainty. Most of the polymers manufactured today will persist for at least decades (if not millennia) [3].

Recycling processes (mechanical and feedstock recycling) of plastics are methods for reducing environmental impact and resource depletion. From the energy and environmental perspective in waste-management issues, it is well demonstrated the better performance of recycling scenarios with respect to landfilling or incineration options [4, 5]. In particular, mechanical recycling which entails the production through physical means of new plastic products from plastic waste [4, 6]. However, the difficulties of this process are mainly related to the degradation of recyclable materials, heterogeneity of plastic wastes and the logistic related to the process [7]. In fact, in the case of U.S. only 6.5% of the used plastics are recycled in conventional centralized recycling [8]. In the case of Europe, only 26% equivalent to 6.6 million tonnes in 2012 of post-consumer plastic wastes were recycled [2]. The main reasons for the low rates of recycling plastics are related to the challenges of collection and transportation because of the high volume-to-weight ratio of the polymers. There is no net economical benefit from recycling plastic materials [9].

Recently, a new prospective approach for polymer recycling has been explored coupling the facilities of Additive Manufacturing (open-source 3D printing) along with the development of open-source hardware such as household scale plastic extruders. This approach could reverse the historical trend of centralized towards distributed recycling [10–14].

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Additive Manufacturing (AM) is defined as "process of joining materials to make objects from three-dimensional (3D) model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies" [15]. One of the AM processes is Fused Deposition Modeling (FDM) where a thread of molten material, usually from a plastic filament, onto a substrate with the use of a movable head. The material is heated to a temperature slightly above its melting point within the head, then extruded through a nozzle to a substrate and cooled down until it solidifies and forms a layer.[16, 17]. Since expiration of FDM patent in the mid-2000s and using a commons-based peer production (CBPP) approach, which it is networked-based, modular and collaborative, a new form of AM have been taking place in the recent years with the development of Open-source 3D printing. Projects such as **RepRap** (or **Replicating Rapid-prototyper**) or Fab@Home [18] can be available to basically everyone. The impacts of these projects can be shown in terms of make the scaling of mass-distributed additive manufacturing of high-value objects technically feasible for everyone [19, 20]. Moreover, the cost of the printers themselves has been shown to be economically advantageous for communities like Fablab, scientific laboratories in the developing world, schools and middle-class households. Open-source 3D printing technologies have been proved to be useful tools in different fields such as Education [21, 22], medical [23–26], scientific equipment [27–29] and sustainable development [30].

In the same way that there is an economic feasibility for distributed model for 3D printers, it could be argued the potential for plastic material (3D printing filament) [14]. The most used polymers for 3D printer filament are Polylactic Acid (PLA) and Acrylonitrile-Butadiene-Styrene (ABS). Currently, different initiatives of development open-source small-scale plastic extruders for transforming post-consumer household plastic into feedstock for 3D printers [Braanker2010] have been Lyman Filament Extruder [31], the Filabot [32], Recyclebot [10]. From the economical point of view, commercial filament costs go from \$18.86-\$175.20 /Kg which it is 20 to 200 times above the cost of raw plastic. In terms of energy, the studies of [10, 13] have shown a proof of concept for high-value recycling of waste polymers where there are 69% to 82% embodied energy saving for distributed recycling over centralizing recycling using a prototype of open-source plastic extruder called "Recyclebot". Therefore, there is an interest of recycling polymeric materials for open-source 3D printing.

However, to make viable the process of distributed recycling, it is necessary to understand the physical characterization at the micro and macro level of the recycled material in order to make appropriate the quality of the material. Mechanical recycling imply a degradation process of the material. Polymeric materials are exposed to thermo-mechanical degradation during its processing where high shear forces and high temperatures can caused chain scission and chemical reactions. On the other hand, thermo-oxidative degradation can produce physical and chemical changes in the polymeric structure due to exposure to specific environmental conditions during service life. Thermo-mechanical and thermo-oxidative degradations are the responsible for the change of structural and morphological properties of the polymers such as mechanical-rheological-thermal properties, degree of crystallinity, viscosity, and molecular composition [6]. This information can be used quality assessment of the recycled material, and also, it can also provide important inputs about the controlling of the processing conditions/parameter during recycling process.

This study is focused in the recycling process of Polylactid Acid (PLA). PLA is one of the most important bio-based, biodegradable and biocompatible polymer. PLA is a thermoplastic aliphatic polyester obtained from the ring-opening polymerization of lactide, which may be derived from the fermentation of sugar feedstock [33]. PLA, as most thermoplastics, can be is employed to produce common-use articles and packaging materials such as bottles, trays, containers and so on by injection moulding, blow moulding, etc., or be extruded into fibres, films and sheets [34–36]

In the context of the recyclability, there have been several studies of mechanical recycling of PLA. The study made by [37] shows the effect of multiple (up to ten times) extrusion of PLA (type 2002D NatureWorks[®], USA) on its (1) mechanical and impact strength, (2) melt flow index (MFI), (3) temperatures of phase transitions and degradation, and (4) water vapor and oxygen transmission rates. This study used a double-screw extruder for granulation of PLA followed by laboratory injection molding press, for preparation of the test samples. Concerning mechanical properties, it is concluded that they get worse with increase of extrusion number. After ten extrusions, there were reductions in tensile strength (5.2%), the tensile strength at break (8.3%) and the impact strength (20.2%). Regarding the MFI, there is an increase of this value in 236% relative to the original PLA after ten successive extrusion processes. Concerning the thermal characteristics, results showed a slight decrease of thermal stability of PLA, lowered cold crystallization temperature and a slight reduction of the melting point with increasing number of cycles. Recycling process has no effect on the glass transition temperature. Finally, the relative increase in the permeability of water vapor and oxygen through a PLA film over the entire range of the extrusion cycles were 39 and 18% respectively.

Pillin et al. [38] worked in the evolution of thermal, rheological and mechanical properties of PLA (PLLA L9000 –92% of *L-lactide*, 8% of *D-lactide* –) with the number of recycling cycles (up to seven). The effect of two stabilizers (tropolone and quinone) in the structure of PLA is investigated. Results showed that glass transition temperature T_g decreases from 66.2°C for pure PLA to 56.5°C after 7 injection moldings. Pure PLA exhibits a very weak crystallinity ratio. However, as processing cycles increase, the crystallization increases from 0.3% to 38.5% after the second injection cycle and rises to 53.6% after the last cycle. Finally, the melting temperature T_m decreases from 171.6°C to 167°C.

Concerning the rheological measurements, the neat PLA has a viscosity η_0 of 3960 Pa.s. The viscosity was measured at 200°C at a shear rate gradient from $0.01s^{-1}$ to $100s^{-1}$ using Carreau’s model. After one injection processing, the viscosity decreases strongly to 713 Pa s. After 7 cycles the material becomes very fluid with a viscosity of 25Pa.s. These results confirm assumption of a strong decrease of M_w of recycled PLA. In fact, a decrease of the 50% of M_w is achieved after three cycles. a 70% decrease of M_w can be considered as severe degradation of PLA. In relation to mechanical properties, results showed that the processing number has no influence on tensile modulus. However, stress at break decreases from 66 MPa to 25 MPa after 7 cycles. In the same way, it is observed a decrease of strain at break from 6% to 0.8%. These phenomenons are ascribed to decrease of the molecular weight, the chain length of the polymer and the increased of the crystallinity.

From literature review, it can be found that injection process has been traditionally used as means of degradation. This study explores the mechanical recycling process for PLA using open-source 3D printers as means of degradation. A method for mechanical recycling is proposed in order to characterize the impact of mechanical, rheological and molecular properties of the recycled material. These results can contribute to understand the physico-chemical characteristics process during recycling, and ultimately, to draw conclusions of the viability of the concept of distributed recycling polymeric materials.

2 Experimental

2.1 Proposed methodology

This methodology consists of fabricate filament feedstock in order to be used for the fabrication of 3D printed mechanical test samples. The recycling process consists of the re-extrusion of the material up to of five cycles in total. For every extrusion process, a set of characterization tests are performed, namely (1) size-exclusion chromatography (SEC), (2) rheology and (3) Melt Flow Index (MFI). These tests are made after the extrusion process. It is intended correlated the mechanical performance of the 3D printing samples with the morphological structure of the recycled polymer. Figure 1 shows the methodology proposed.

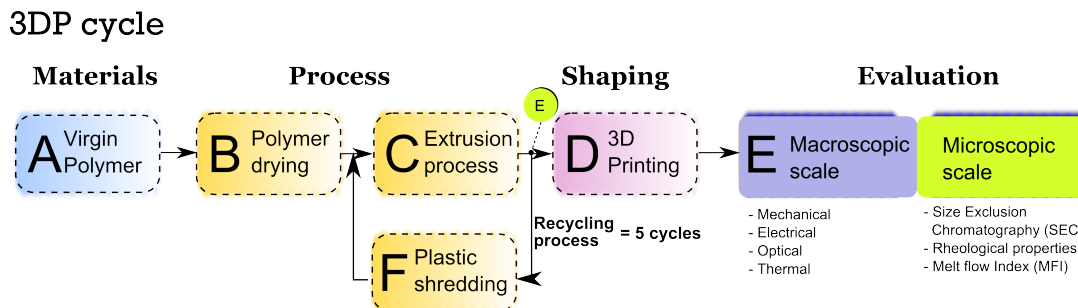


Figure 1: Overview of the proposed methodology

2.2 Materials

Poly lactide acid (PLA) type 4043D , a product of NatureWorks supplied by NaturePlast (Caen, France), was the material studied. According to the manufacture, this PLA has a density $1.24 g/cm^3$, tensile yield strength 60MPa, tensile modulus of 3600 MPa, tensile strength at break 53MPa, tensile elongation 6%, Melt Flow Index $MFI = 6 g/10min$ at 210°C, a glass transition temperature (T_g) 55-60. Prior to processing, virgin pellets were dried during 4 h at 80°C in a dehumidifiers in order to remove as much as humidity as possible from PLA pellets.

Extrusion process was performed using a laboratory scale HAAKE™ Rheomex CTW 100 OS counter-rotating conical twin screw extruder. The range speed operation is 0-250 rpm. The temperature profile selected was 160,170,180°C. The screw speed was set at 40 rpm. The feedrate in the extrusion process is established at $0.53 \pm 0.04 Kg/hr$. This process is made in order made the filament used in the process of 3D printing.

2.3 Mechanical properties

Tensile properties were study according to ISO 527. Tensile specimen is a dog-bone geometry of 150 mm length and central dimensions of $10 \times 4 mm^2$. Tensile test were made in order to evaluate the tensile strength ($\sigma_M [MPa]$), tensile stress at break ($\sigma_B [MPa]$), strain at break ($\epsilon_B [%]$), Elastic modulus ($E [MPa]$). This parameter can describe the changes in macroscopic mechanical properties of the recycled material. Static tensile tests were performed by means

of an Instron 5569 (Instron, USA) universal electromechanical testing instrument with a loading speed of $1\text{ mm}/\text{min}$, and a 50 kN load cell. An extensometer was used with an nominal length of 50 mm to determine elastic modulus. Analysis were repeated at least 5 times per material, and average of parameters were used as representative values.

The fixed factors considered in this investigation are shown in table 1.

2.3.1 Equipment

A derivative version of the RepRap machine, called FoldaRap [39] (see fig. 2) was selected for this investigation. The FoldaRap machine is a representative 3D printer among the set of open-source machines developed by the RepRap community. Indeed, as can be seen in the open-source 3D printer family tree [20], the FoldaRap derives from the main branch (XZ Head, Y Bed): Darwin-Sells Mendel-Prusa Mendel [40]

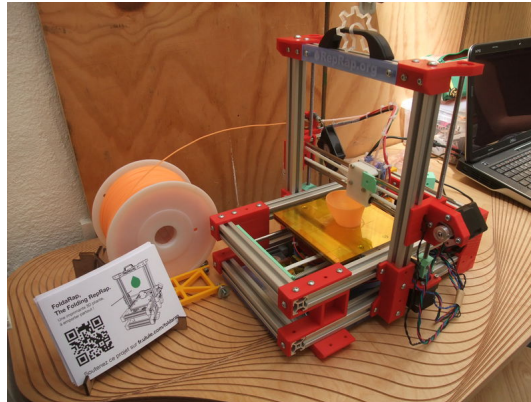


Figure 2: Open Source 3D printer -FoldaRap-

This system can be described through three fundamental axes (1) Machine architecture, (2) Electronic hardware and (3) Software [41, 42]. Regarding the FoldaRap’s architecture, it is a Cartesian 3D printer where the extrusion system can be displaced in the vertical plane XZ and the heated print bed can be displaced in the horizontal direction -Y. The work capability is $140 \times 140 \times 155\text{ mm}^3$. Using a mechanical coupling stepper motor-drive gear, the extrusion system forces a PLA polymer filament with a diameter of 1.75mm into an aluminium melt chamber, then the filament is extruded through a 0.5mm nozzle. The linear motion for positioning axis XY is achieved through machined plastic bushings and smooth rods 6mm in diameter using a transmission mechanism of timing belts and pulleys. For axis Z, threaded rods M5 and hexagonal nuts are coupled with a stepper motor with a minimum resolution of 0.00025 mm. The heated print bed is made of aluminium joined with a Peltier cell and it uses a top layer of kapton in order to improve the adherence of the piece with the print bed.

Concerning the electronic hardware and software, the FoldaRap machine uses a Melzi v2.0 controller board which makes it possible to control the machine via a USB connection [43]. Marlin is used as firmware software, Slic3r software is used to convert the .STL files into G-codes, and Pronterface software is used as the system’s host software. All these elements are open-source. FoldaRap has an indicated minimum value of layer height tested at 0.1 mm and it does not include the capacity to fabricate support structures.

Table 1 shows the parameter used in the fabrication of the test samples

Table 1: Fixed factors

| Parameters | Value | Units |
|---------------------|--------|-------|
| Bed temperature | 60 | °C |
| Nozzle temperature | 195 | °C |
| # of perimeters | 2 | |
| Top solid layers | 2 | |
| Bottom solid layers | 2 | |
| Fill density | 100 | % |
| Fill | 0/90 | |
| Travel speed | 200 | mm/s |
| Nozzle diameter | 0.5 | mm |
| Slic3r software | G-code | |

2.4 Size Exclusion Chromatography (SEC)

For SEC experiments, a Shimadzu LC10AD system was used in combination with a Shimadzu RID10A differential refractometer and a Shimadzu SPD 10Avp UV dual wavelength detector ($\lambda_1 = 254nm$ and $\lambda_2 280nm$). The column set was constituted of five 30 cm PL Gel columns with a pore size of 10 μm (from Polymer Laboratories). The solvent was analytical grade THF at a flow rate of 1 mL/min. The SEC experiments were carried out at room temperature.

2.5 Rheology

Rheological experiments of recycled PLA were performed using a parallel plate rheometer of type RDAII Rheometrics scientific. The static mode was used and the samples were prepared at 180°C. The samples were disks of 25 mm in diameter and about 1.5 mm in thickness. The viscosity was obtained using a shear rate gradient from 0.01s⁻¹ to 500s⁻¹.

2.6 Melt Flow Index (MFI)

Melt Flow Index (MFI) is an indirect technique to measure the viscosity of polymers. It is used in polymer technology as a product specification since this value gives an indication of the processing properties of the polymer. The value of MFI is expressed as the mass of polymer melt pushed from the heated cylinder of the extrusion plastometer through its precision bore orifice by its piston in a period of time, the standard units of the value being grams per ten minutes (g/10 min). The weight used was 2.16 Kg and the temperature was 180°C.

3 Results and discussion

3.1 Mechanical properties

Table 2 presents a comparison between the mechanical properties of the different recycled PLA samples. Strain at break values of the extrusion 1 are in agree with the study made by [44] using the same parameters of material (PLA) and fill (0/90). On the other hand, the average value of elastic modulus and tensile strength are lower with respect to the study. These differences can be explain by the fact that in the open-source 3D printer context, it exists a great variability of the results considering that (1) Processing parameters in 3D printing, such as extrusion temperature, can have a significant effect on the structure and properties of printed parts. (2) Machine architecture and their components required different settings that can affect part strength. (3) Filament quality in terms of diameter can influence the mechanical behavior.

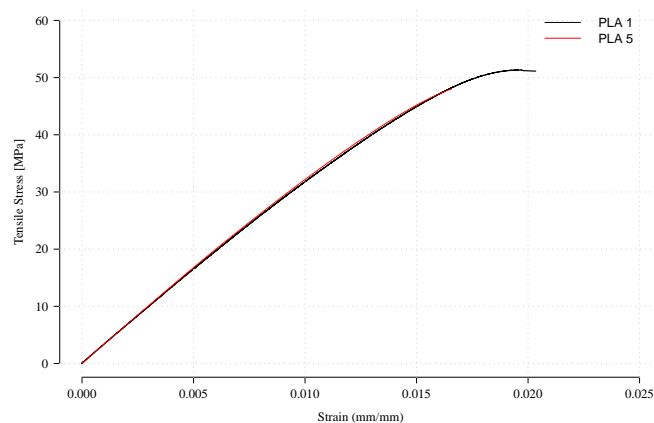


Figure 3: Stress-strain curves for samples of the extrusion 1 and 5.

The stress-strain curves for samples PLA_1 and PLA_5 are presented in the figure 3. It is shown that the tensile modulus is slightly influenced by recycling process. This trend is also observed in the study made by [37, 38]. One possible of the reason for this behavior is that the potential decrease of the molecular weight of PLA is balanced by an increase of crystallinity. Therefore, the strength of the material in the elastic domain is not affected by the recycling. However, it is observed a decrease of strain at break from 1.88% to 1.68% as illustrated in figure 4. The reason for

this can be attributed to the increase in the crystallinity of the material which it is a factor that stimulates crack propagation in the elastic zone of the material.

Table 2: Tensile properties of PLA recycled

| Extrusion number | Tensile modulus (MPa) | Stress at break (MPa) | Strain at Break (%) |
|------------------|-----------------------|-----------------------|---------------------|
| 1 | 3093.65 ± 194.99 | 46.44 ± 2.69 | 1.88 ± 0.20 |
| 2 | 3556.13 ± 56.86 | 51.79 ± 0.85 | 2.25 ± 0.40 |
| 3 | 3356.23 ± 161.70 | 46.16 ± 3.08 | 1.85 ± 0.27 |
| 4 | 3441.58 ± 119.05 | 48.94 ± 1.48 | 1.98 ± 0.28 |
| 5 | 3491.60 ± 98.14 | 48.81 ± 1.47 | 1.68 ± 0.02 |

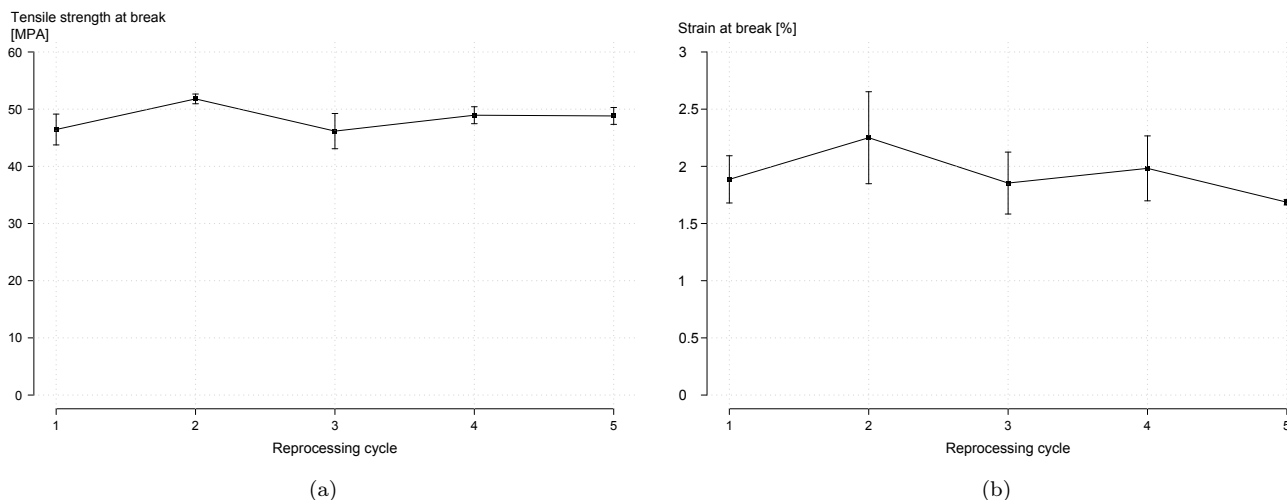


Figure 4: (a) Tensile strength at break σ_B and (b) Strain at break ϵ_B as function of the extrusion number

3.2 Influence of recycling on molecular weight

Molecular weights of recycled samples was obtained by means of SEC measurements. According to the manufacturer, the number-average molecular weight M_n is about 85,000 g/mol and the weight-average molecular weight M_w is 110,000 g/mol. From these values, the polydispersity index I_p is 1.29. Table 3 summarizes the strongly decrease of molecular weight of recycled material after the recycling process.

Table 3: Molecular weight of recycled PLA samples after extrusion process

| Extrusion number | M_n [g/mol] | M_w [g/mol] | I_p |
|------------------|---------------|---------------|-------|
| Virgin | 85000 | 110000 | 1.29 |
| 1 | 75000 | 104000 | 1.39 |
| 2 | 66600 | 88200 | 1.32 |
| 3 | 57500 | 80600 | 1.402 |
| 4 | 45800 | 68600 | 1.50 |
| 5 | 37300 | 58400 | 1.55 |

It can be pointed out that a reduction of about 26.73% is achieved after 3 cycles with respect to virgin polymer, and 46.91% is achieved after the 5 cycles. Pillin et al. [38] highlights that a decrease of 70% of the M_w can be considered as a severe degradation of PLA. These decrease in molecular weight may be due to hydrolytic, radical degradation and/or to residual catalyst that can promote transesterification during processing at high temperature [35, 45]

3.3 Rheological properties

Figure 5 presents the evolution of the recycled samples zero-shear viscosity η_o .

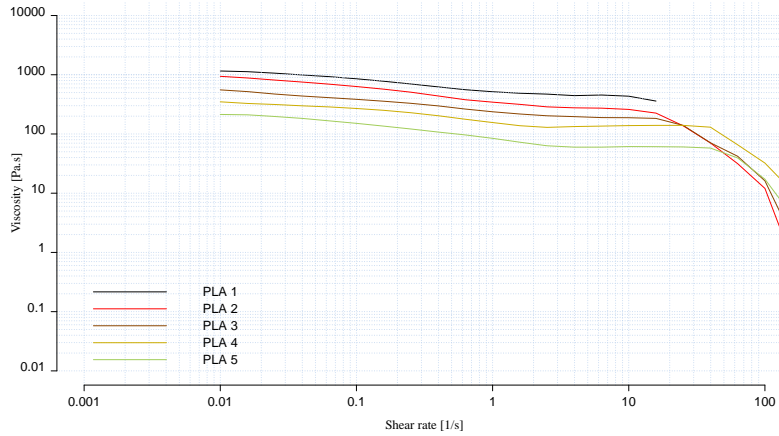


Figure 5: Viscosity profile en fonction of the shear rate for the recycled samples

Experimental data has been adjusted according to the Carreau model (equation 1) in order to obtain the zero-shear viscosity η

$$\eta = \eta_{\alpha} + (\eta_0 - \eta_{\alpha}) [1 + (\lambda_c \dot{\gamma})^2]^{\frac{n-1}{2}} \quad (1)$$

where, λ_c is time constants related to the relaxation times of the polymer in solution and n is a dimensionless exponent. Figure 6) shows the reduction of the zero-shear viscosity η_0 from 2729.21 Pa.s in extrusion 1 to 219.85 Pa.s in extrusion 5. These results confirm the hypothesis of an important decrease in molecular weight of the recycled PLA. Drumright, Gruber, and Henton [34] describes the degradation process where the high molecular chains hydrolyze to a lower molecular weight oligomers. Agents such as temperature and moisture levels, acids or basis can further accelerate the degradation process. One of the main consequences is the embrittlement of the plastic affecting the structural integrity of PLA articles. At about a M_n inferior to 40.000, microorganisms in the environment can continue the degradation process by converting the lower molecular weight components to carbon dioxide, water and humus.

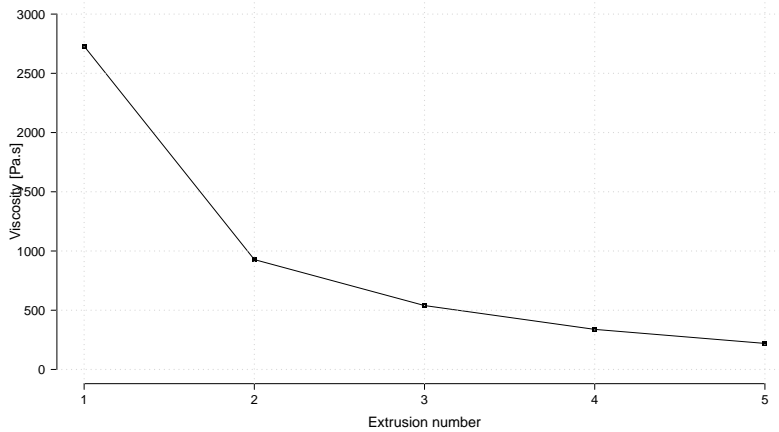


Figure 6: Zero-shear viscosity η_0 calculated from Carreau model as a function of extrusion number

Filament's rheological and mechanical properties are the most important considerations in order to fabricate feedstock for fused deposition with optimal set of properties [46–49]. There are important characteristics such as: (1) high flexural modulus and strength to enable continuous spooling and unspooling operations, (2) high compressive strength to not break after passing through the rollers, and (3) sufficient filament stiffness or low viscosity in order to avoid "buckling" of the filament, which it interferes with the correct process of printing.

3.4 Melt Flow Index

Figure 7 show the values of MFI as a function of the PLA extrusion number. In this case, it is only shown the evolution from virgin value (extrusion 0) until extrusion 4. They are approximated by a second-order polynomial ($R \approx 0.9693$). MFI value significantly increases with successive PLA extrusion process. The Snedeco's F-test indicates that the values of two consecutive values of MFI (MFI_i and MFI_{i+1}) are significantly differ from each other. The increase of MFI value of about 6.05 times ($MFI_4/MFI_0 = 6.05$). The range of deviation between mean values of MFI are $1.152 - 9.356 \text{ gr}/\text{min}$. The larger the melt index value, the lower is its viscosity, and therefore, the average molecular weight of the polymer is lower.

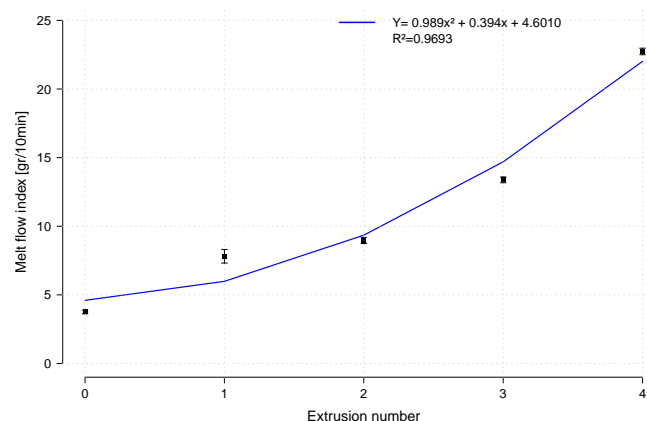


Figure 7: Melt Flow Index (MFI) as function of the extrusion number from virgin to extrusion 4

4 Conclusions and future work

In this study, it is aimed to investigate the mechanical, rheological and molecular properties of recycled PLA using open-source 3D printing. The feedstock material was prepared using a counter-rotating twin-screw extruder. Results show that mechanical properties of the recycled material are slightly affected by the recycling process. Elastic modulus is remains constant. However, the strain at break show a reduction of 10.63% after 5 cycles. SEC experiments confirm an important reduction of molecular weights of about 26.73% after 3 extrusions with respect to virgin polymer, and 46.91% after the 5 extrusions. Using the Carreau model, rheological experiments show a reduction of the zero-shear viscosity η_0 from $2729.21 \text{ Pa}\cdot\text{s}$ in extrusion 1 to $219.85 \text{ Pa}\cdot\text{s}$ in extrusion 5. MFI results show an important increase of about 6.05 times in reference to the virgin value after the recycling process. The present results indicate the viability for PLA recycling using the technology of open-source 3D printing

This is an ongoing research project, and it is intended to further study the impact of nozzle extrusion of the 3D printer on the mechanical and rheological properties of the recycled material. Different studies [46–49] have developed mathematical models in order to understand the liquefier dynamics in order to establish control strategies in the extrusion phase of 3D printing. It is very important to link the parameters of the 3D printing process with properties of the material. Hence, it is possible to guarantee optimal properties of recycled materials for a distributed recycling model using opens-source 3D printers.

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