Visual Abstract

Effect of protective coatings on the water absorption and mechanical properties of 3D printed PLA

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ABSTRACT. This work aims to study the influence of protective coatings on the water absorption and mechanical properties of 3D printed poly–lactic acid (PLA) parts. The PLA parts were fabricated with different levels of the 3D printing process parameters, aiming to define samples with distinct strength and ductility/toughness characteristics. Water absorption tests following the standard ASTM D570–98 were performed on uncoated and coated PLA specimens. The effectiveness of two protective coatings based on acrylic and polyurethane varnish on reducing water absorption was evaluated. Both protective coatings have shown being effective on preventing water absorption by the PLA, with polyurethane presenting the best performance reducing water absorption by 38%. Tensile tests were carried out to determine the ultimate tensile strength, elastic modulus, yield tensile strength, fracture strain and toughness of specimens, before and after the application of protective coatings. The polyurethane protective coating also benefits the tensile properties of PLA parts, increasing the strength and ductility/toughness characteristics of specimens up to 24%.

Citation: Vincente, C.M.S., Fernandes, J., Reis, L., de Deus, A. M. Vaz, M. F., Leite, M., Effect of protective coatings on the water absorption and mechanical properties of 3D printed PLA, Frattura ed Integrità Strutturale, 48 (2019) 748-756.

Received: 13.02.2019
Accepted: 13.03.2019
Published: 15.04.2019

KEYWORDS. 3D printing; PLA; mechanical properties; water absorption; printing parameters

INTRODUCTION

In recent years the use of 3D printing technologies has increased significantly and this rapid growth it is expected to remain in a near future. The 3D printers are now commonly used for the manufacture of a broad array of products, ranging from leisure articles to medical components [1]. From all available 3D printing technologies, one of the most popular to the public is fused deposition modelling (FDM) due to the vast number of companies that develop and market 3D printers based on this technology, at relatively low cost [2]. FDM printed materials include among others : acrylonitrile butadiene styrene (ABS), poly–lactic acid (PLA), polycarbonate, nylon and polyamide, which are now available for building mechanical parts with diversified characteristics, on demand [3]. From the listed materials PLA, a biodegradable thermoplastic polymerized from lactic acid from natural sources such as corn [4], was one of the materials that attracted most attention due to their unique properties for 3D printing. The main advantages of PLA are low printing temperature, smoother appearance, high geometric resolution, low warping effect and biodegradability. On the other hand, the comparative disadvantages with other thermoplastics relies on his brittleness and low thermal stability. As 3D printing technology spreads into more domestic users or at industrial level, PLA emerge as a valid sustainable thermoplastic alternative to non–biodegradable polymers, able to address the problem of residues derived from manufacturing processes, which impacts the environment of our planet. In this context, the characterization and improvement of the mechanical properties of PLA parts produced by FDM it is a topic of great interest, so that this material can be used on a reliable way.
On the past several studies stablished a relation between FDM parameters and their combinations on the mechanical properties of 3D printed PLA [5–11]. These studies allow the designer to define the 3D printing conditions (so that they can meet mechanical design requirements) by controlling the strength and ductility of materials, or assigning variable mechanical properties to parts, according to their final application. From the literature it's well know that the higher strength of PLA parts produced by FDM is linked to higher extrusion temperatures [12–14], raster angles aligned with the direction of the applied force [7,9,15] and smaller layer thicknesses [7,8,16], while the ductility follows the inverse trend. These trends of the FDM process provide guidelines for the definition of processing parameters of PLA parts regarding his application in elements of strength or ductility/toughness, depending on the project requirements.
Another important issue that also must be considered on the project of PLA components by FDM is water absorption, especially when exists direct contact with fluids, such as in the case of underwater [17], biomedical [18], or microfluidic devices [19]. Water absorption can arise due to the porosity of the printed part or from the PLA material itself. It is known that the polar bonds in PLA can make it susceptible to water absorption, which can cause partial breakdown the PLA polymer chains and change mechanical properties, turning PLA even more brittle. Moreover, after the 3D printing process PLA becomes more chemically reactive, increasing the susceptibility of PLA to water,[20] which will tend to decompose the material over time [21,22]. A classical, simple and inexpensive way to protect a water reactive material is by applying a protective coating using the painting method. This method was successful applied on surface modification of ABS reducing water absorption (from 1.6 to 0.4 % in weight) [23] and on the protection of a PLA hull with a polyurethane (PU) coating, reducing water absorption from 3.8 to 1.5% in weight [17]. Despite the significance of this topic, there is also a lack of studies evaluating the impact of surface treatments on mechanical properties of FDM parts made with PLA, with most works mainly focused on evaluating the staircase effect reduction [24–27]. The few reported works evaluating the impact of surface treatments on mechanical properties of PLA parts produced by FDM, are until now limited to blow cold vapor surface treatments [28].
On our work, PLA specimens were fabricated by FDM with different levels for the 3D printing parameters: layer thickness, raster angle and extrusion temperature, aiming to define distinct levels of strength (improved ultimate tensile stress, modulus of elasticity and yield strength) and ductility/toughness (improved fracture strain and toughness). The 3D printing conditions of specimens were replicated on cubic samples that were subjected to water absorption tests. The water absorption was estimated for uncoated and coated samples with acrylic or polyurethane (PU) protective coatings. The influence of protective coatings on mechanical properties were also evaluated trough tensile tests.
The use of protective coatings for watertight applications of PLA 3D printed parts, will enable new designs where both sealing and the preservation of mechanical properties are critical aspects. To the best of our knowledge the water absorption

and the mechanical behavior of 3D printed PLA parts (before and after protective coatings application), was not previously systematically investigated.

MATERIALS AND METHODS

3D printing and protective coatings

A Ultimaker 2™ machine was used to print all the samples from the same white PLA filament roll. The 3D models were modelled in SolidWorks® and exported in a stereolithographic (STL) file to the slicing software Cura®, to generate the g–code file. For the constant printing parameters, the recommended standard profile by Cura® was adopted. The infill density as set to 60% for all samples. The extrusion was performed with a nozzle diameter of 0.4 mm. The temperature of the platform was set to 60 ºC. The printing speed was 40 $mm.s^{-1}$ for the first layer, external layers (top and bottom) and contours. The printing speed chosen for internal contours and filling layers was 60 and 80 $mm.s^{-1}$, respectively. The thickness of the contours is 0.4 mm, while for tops and bottoms is equal to 0.8 mm. The thickness of initial layer as set to 0.3 mm. Two kind of samples (LVL1 and LVL2) were printed with the levels of printing parameters, displayed on Tab. 1. For each sample three repetitions were considered.

		PARAMETERS		
	Samples	**Layer thickness (mm)**	**Raster angle (º)**	**Extrusion temperature (ºC)**
LEVELS	**LVL1**	0.1	(0º/90º)	220
	LVL2	0.2	(–45º/45º)	200

Table 1: FDM process parameters and its levels.

The tensile tests specimens were printed on the flat direction, with the geometry and dimensions displayed on Fig. 1.

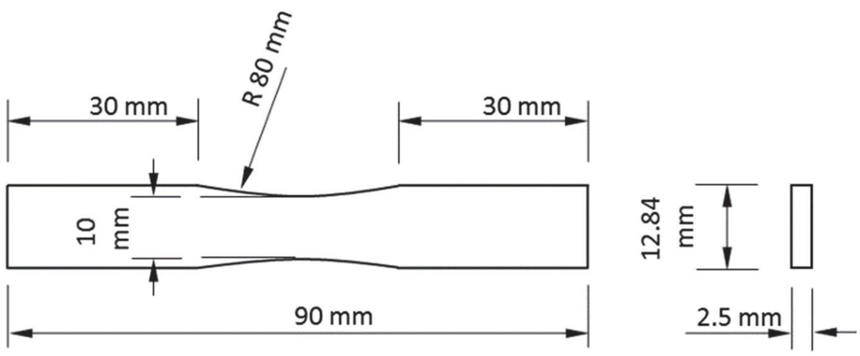

Figure 1: Geometry and dimensions of tensile tests specimens.

Water absorption samples consists on coated and uncoated cubes with dimensions of approximately 10 mm, which were fabricated with the printing parameters of Tab. 1. Two different protective coatings were selected: a PU wood sealant (Lakeone™) and an acrylic aqueous varnish (Luxens™). Before the application of protective coatings and water immersion tests, the samples were placed in an oven during 24 hours, in order to ensure that the water content was minimum and equal for all samples. After the application of coatings, the PLA coated samples were dried at room temperature, for complete solvent removal.

Tensile tests

Tensile tests were performed using a universal testing machine (INSTRON 3369) with a load cell of 50 kN and a strain rate of 2 $mm.min^{-1}$. At least three tests were made for each sample manufacturing conditions. Stress-strain curves were obtained for each test from the load vs. displacement curves. The toughness of each specimen was calculated from the integration of the stress–strain curve [29].

Water absorption tests

Water absorption was analysed according to the ASTM D570–98 standard with both uncoated and coated specimens. The specimens were weighted before water immersion and his dry mass was recorded. The number of tests repetitions for each pair sample/coating was three. The samples immersion was done by covering half of the specimen's height with water on a glass container. To guarantee that water reaches the bottom part of the samples, an absorbing filter paper was placed on the bottom of the specimens. In each defined time interval, the cubes were taken from the container, the excessive water was removed with paper and were posteriorly weighted. The weighing of the cubes was done in intervals of 30 minutes, for the first 4 hours, in intervals of 1 hour, for the next 4 hours, and in intervals of 24 hours, for the next 4 days. In total, the cubes spent 104 hours inside water. Some properties can be obtained from this type of test, namely: weight gain (*WG*) and open porosity (*P*), calculated by Eq. (1) and Eq. (2):

$$WG = \frac{m_{sat} - m_{dry}}{m_{dry}} \times 100\% \quad (1)$$

$$P = \frac{\left(m_{sat} - m_{dry}\right) / \rho_{H_2O}}{V} \times 100\% \quad (2)$$

where m_{sat} is the saturated mass (the mass at the end of the experiment), m_{dry} is the dry mass (the mass at the beginning of the experiment), ρ_{H2O} is the density of water (1 g.cm^{-3}) and V is the volume of the cube (1 cm^3). The absorption coefficient (*K*) was calculated graphically as the initial slope of the graph where *x* axis is the square root of the immersion time, and the *y* axis is the weight gain per unit of area of the cube. The weight gain per area is calculated by Eq. (3):

$$\frac{WG}{area} = \frac{m_{wet} - m_{dry}}{A \times m_{dry}} \quad (3)$$

where m_{wet} is the mass of the cube in a certain instant of time, and *A* is the area of one of the faces of the cube.

Morphological characterization

The external surface surfaces of PLA specimens were inspected by scanning electron microscopy (SEM). The SEM images were obtained with a field emission type microscope (Hitachi®–S2400) operating at 20.0 kV, in a secondary electron emission mode. The surface of specimens was fixed on the SEM sample holder with a conductive adhesive and the sample surface was cleaned with an air flux before coating process on a sputtering system (Quorum–Q150TES). The conductive coating was performed with Au–Pt alloy using a current of 40 mA and evaporation time of 60 s.

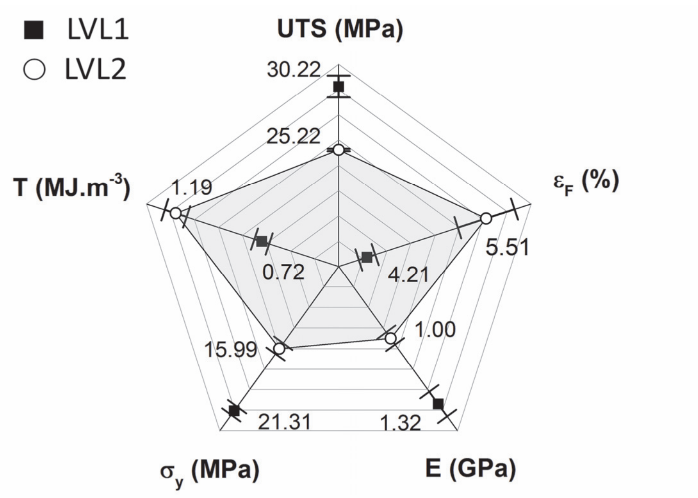

Figure 2: Mechanical properties of samples LVL1 and LVL2.

RESULTS AND DISCUSSION

Mechanical properties of PLA uncoated samples

The tensile tests average response values for each different specimen according with the experimental parameters defined on Tab. 1 are presented on Fig. 2.

The best conditions for maximizing strength correspond to sample LVL1, with a layer thickness of 0.1 mm, raster angle of (0º/90º) and extrusion temperature of 220 ºC. The level of parameters that lead to a maximum ductility they correspond to sample LVL2, with a layer thickness of 0.2 mm, raster angle of (–45º/45º) and extrusion temperature of 200 ºC. The obtained results represent a variation in strength (more than 15%) and ductility (more than 20%) between samples, by simply changing the levels of the FDM process parameters. These results are consistent with the previous reported trends of the FDM process for strength and ductility of PLA parts [7,9,12,14–16]. Taking in account the mechanical design specifications, these mechanical properties for PLA, reinforces the idea that adapting the levels of parameters could lead to a reduction of raw materials and energy costs of the FDM process.

Water absorption tests

On Fig. 3a are shown the results obtained from the experiments of water absorption tests for PLA samples LVL1 and LVL2.

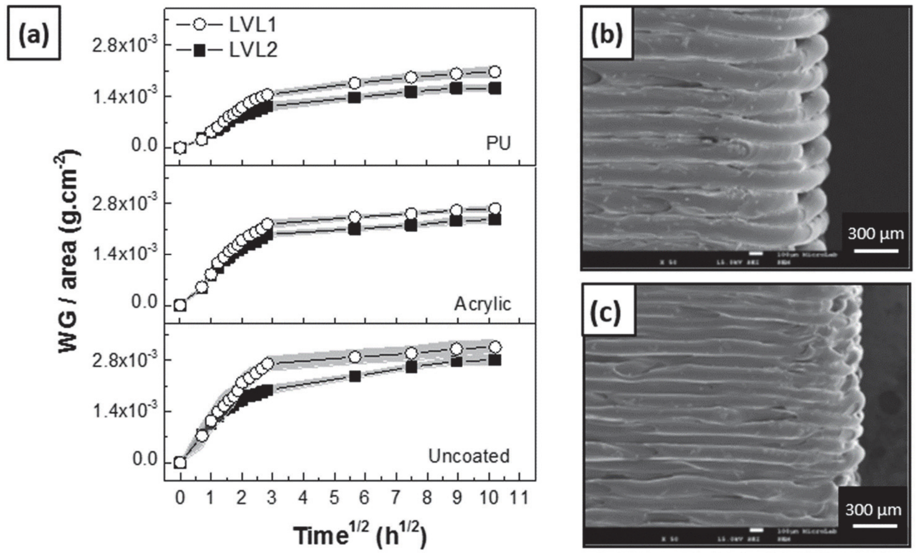

Figure 3: (a) Evolution of the WG per area of samples LVL1 and LVL2 (uncoated, coated with acrylic and coated with PU) as a function of the square root of the immersion time (grey shadowed areas correspond to the statistical errors). SEM images evidencing the roughness of samples with a layer thickness of (b) 0.2 and (c) 0.1 mm. Solid lines are auxiliary visual guidelines.

The maximum weight gain per area of the cubes was reduced in average from 2.98×10^{-3} g.cm^{-2} (uncoated) to 2.50×10^{-3} g.cm^{-2} (acrylic coated) and 1.84×10^{-3} g.cm^{-2} (PU coated), meaning that adding protective coatings of acrylic or PU on PLA helps on preventing the absorption of water by PLA, Tab. 2. The variation from uncoated to acrylic coated samples represents a reduction of water absorption of 16%, while the PU coating allow reduce water absorption by 38%. It was also observed that sample LVL1 absorbs more water per area than sample LVL2, with or without protective coatings.

The open porosity follows the same trend as the weight gain per area in all samples meaning that it is one important mechanism for water absorption in PLA parts produced by FDM. The values of water absorption coefficient also reveal that the time needed to absorb a volume unit of water (proportional to K^{-1}), increases when samples are coated, reaching its maximum with the PU coating. Sample LVL1 possess the lowest layer thickness (0.1 mm) and higher extrusion temperature (220 ºC). The lower the layer thickness, the rougher the surface is expected to be [30], as it can be verified by the comparison of Fig. 3b and 3c.

Samples		WG (%)	P (%)	K (g.cm^2.min$^{1/2}$)
LVL1	*Uncoated*	0.316 ± 0.023	0.315 ± 0.020	(1.23 ± 0.05) × 10^{-4}
	Acrylic Coated	0.266 ± 0.006	0.264 ± 0.006	(1.04 ± 0.10) × 10^{-4}
	PU Coated	0.208 ± 0.013	0.205 ± 0.013	(7.25 ± 0.08) × 10^{-5}
	Uncoated	0.281 ± 0.016	0.277 ± 0.013	(3.26 ± 0.03) × 10^{-4}
LVL2	*Acrylic Coated*	0.236 ± 0.008	0.234 ± 0.006	(8.96 ± 0.08) × 10^{-4}
	PU Coated	0.163 ± 0.011	0.161 ± 0.011	(5.34 ± 0.02) × 10^{-5}

Table 2: Weight gain, open porosity and water absorption coefficient of samples LVL1 and LVL2.

The effect of the surface roughness on wettability was described on the earlier work of Wenzel [31], concluding that the presence of roughness on a surface will contribute to enhance his natural hydrophilic or hydrophobic behaviour. PLA surfaces are described as intermediate surfaces between hydrophilic and hydrophobic, presenting a contact angle with water of approximately 73°. The effect studied by Wenzel was demonstrated on a PLA surface in the work of Jordá–Vilaplana [32], showing that the contact angle with water of PLA sheets is reduced from 73.4° to 27°, when the root mean square roughness values vary from 12 to 55 nm. From these findings we can expect that the surface of sample LVL1 will be more hydrophilic than sample LVL2, promoting thus water absorption. Another important mechanism for water absorption in polymers is the diffusion of water molecules in the micro voids between polymeric chains [33]. It is also known that the processing temperature of the polymer is responsible for the creation of these micro–cracks. An increase in micro–cracks leads to an increase of water absorption [34]. This fact is linked to the porosity of the material. In our work, the extrusion temperature of 220 °C was responsible for the highest porosity, and consequently the highest amount of water absorbed, on sample LVL1. After verifying that the application of PU coating was efficient on protecting the PLA parts from water absorption, it's now important to know which will be the impact of the PU coating on the mechanical properties of PLA parts.

Effect of the PU coating on mechanical properties

On Fig. 4 are displayed the mechanical properties of samples LVL1 and LVL2, estimated from tensile tests, before and after the application PU coating on specimens.

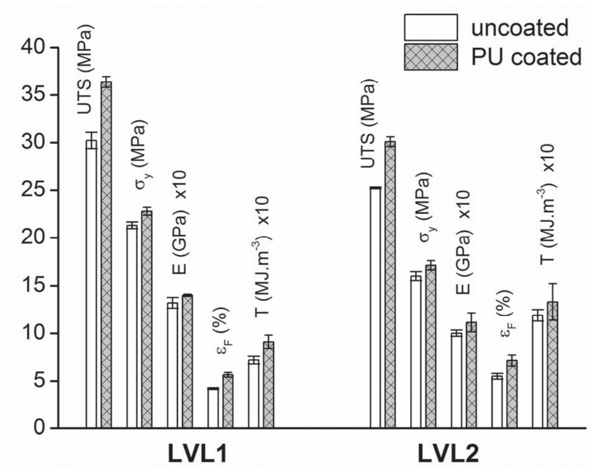

Figure 4: Mechanical properties of specimens LVL1 and LVL2, before and after the application PU coating.

From the results presented on Fig. 4, we can observe that the application of PU coating improve the strength and ductility/toughness of samples. The best mechanical properties maximizing strength (UTS=36.4 MPa, σ_y=22.8 MPa and E=1.40 GPa), were obtained for PU coated LVL1 specimens, while the best values for ductility/toughness (ε_F =7.14 % and T=1.33 J/cm^2) were found on PU coated LVL2 specimens. In average the mechanical properties related with strength were improved by 17 % (UTS), 13% (σ_y) and 8.0 % (E), while the mechanical properties related with ductility increases 24 % (ε_F) and 16 % (T), with the application of the PU coating. The beneficial effect of the PU coating on the mechanical properties can be explained by the fact that the PU coating will fill the air gaps of PLA left by the FDM process (which will reduce the roughness), helping to distribute the applied load and preventing the process of crack propagation. In fact, a similar effect was also reported on the work of *Leite et al*, where the application of an acrylic varnish on ABS parts fabricated by FDM, behind prevent water absorption was led to a strong increase of σ_y (> 30%) for samples printed in the flat direction [23]. The benefits and simplicity of the use of protective coatings on PLA parts are a potential alternative to other chemical treatments that were proposed to sealing FDM components, but with negative consequences on some mechanical properties [26,28,35] especially when thin sections are present on parts [36].

CONCLUSIONS

This work describes the fabrication and characterization of PLA parts produced by FDM, with and without protective coatings. The relationships between processing and properties that were determined allow to provide guidelines for obtain PLA parts with tailored properties depending on the final application. The 3D printing studied parameters levels and protective coatings influence strength and ductility/toughness of PLA parts. The application of acrylic and PU based coatings reduces water absorption of PLA produced by FDM. The PU protective coating demonstrated to be the most effective, reducing the water absorption by 38%, when compared with uncoated samples. The mechanical properties of PLA parts were also improved from 8 to 24 %, when samples are coated with PU. The presented results contribute to fulfill the lack of data which users of FDM printers face when projecting with PLA, contributing to stablish a better balance between strength and ductility/toughness on FDM PLA parts and broadening the scope of application of this environment–friendly material.

ACKNOWLEDGMENTS

This work was supported by Fundação para a Ciência e Tecnologia (FCT), through IDMEC, under LAETA project, UID/EMS/50022/2019. The authors also gratefully acknowledge the funding of FIBR3D project, Reference SAICTPAC/0036/2015, financed by European Structural and Investment Funds (ESIFs) through the Lisbon Regional Operational Programme 2020 and by FCT national funds, POCI-01-0145-FEDER-016414. Marco Leite acknowledges the FCT funding through UNIDEMI project UID/EMS/00667/2019. Carlos M.S. Vicente acknowledge the FIBR3D project, reference SAICTPAC/0036/2015, for the post-doctoral research grant.

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