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Post-processing in multi-material 3D printing

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ABSTRACT

Purpose: This study aims to investigate the adhesion of combining two materials with different properties (PLA-TPU and TPU-PLA) printed in FFF (fused filament fabrication) with post-processing treatments.

Design/methodology/approach: The scope of the study includes making variants of samples and subjecting them to three different post-printing treatments. After processes, shear tests were conducted to determine the adhesion.

Findings: The post-printing treatment results in a stronger inter-material bond and increased adhesion strength; the best average shear strength results were achieved for annealing without acetone and for PLA/TPU samples for treatment in cold acetone vapour.

Research limitations/implications: In the study, adhesion was considered in the circular pattern of surface development.

Practical implications: Reinforcement of the biopolymer broadens the possibilities of using polylactide. Examples of applications include personalised printing items, where the elastomer will strengthen the polylactide.

Originality/value: These studies aim to promote the use and expand the possibilities of using PLA biopolymer. The strength properties of printouts from different materials are often insufficient, hence the proposal to use post-printing processing.

Keywords: 3D printing, Post-processing, PLA, TPU, Multi-material

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PROPERTIES

1. Introduction

3D printing is a revolutionary additive manufacturing technique that enables rapid prototyping of complex geometric structures and design flexibility [1-16]. 3D printing responds to market demands, especially sudden and unpredictable ones. It supported the industry during the Covid-19 pandemic by enabling the rapid production of filters, visors, and much personal protective equipment. During the war in Ukraine, it supported the production of tourniquets for the wounded and missing military and paramilitary parts. These applications point to 3D printing's main advantage of producing any object from available raw materials, with fast supply chains in the local market.

Applications of additive technologies include biomedical [17-24], dental [9], automotive [25,26], aerospace [27], optics [11,28], and also textile and daily life sectors [29-32]. Developments in printing are leaning toward more and more applications, including the use of conductive, electrically functional, insulating, or semiconducting materials [1]. The wide range of polymers available for 3D printing enables the creation of geometries with shape memory, where structures under the influence of certain external stimuli such as temperature, light, or water, for example, change their shape [1,33].

FDM (Fused Deposition Modelling) printing technology, also known as FFF (Fused Filament Fabrication), is an extrusion-based additive manufacturing process in which a thermoplastic material as a filament is melted and selectively deposited through a printing nozzle [9, 34-38]. This way, a model according to the computer design is produced layer by layer. Fused deposition technology is one of the world's most widely used incremental methods.

The biggest challenge in creating models is achieving proper adhesion between the printed layers. Thermoplastic material is extruded through a nozzle onto a previously deposited layer. The high printing temperature and pressure accompanying the extrusion cause the previously deposited material to melt and form a permanent bond with the new layer. Deposition of layers of the same polymer is usually trouble-free, and the quality of the connections is satisfactory (the bond strength between layers is always lower than that of the building material). Printing from two nozzles with different materials means that material boundaries can weaken the strength of models. This is due to the need to use various printing parameters for each filament: temperature, printing speed, and cooling. In addition, objects produced with FDM technology are characterised by a wavy outer structure resulting from the path of layer application. Some precision parts (e.g., threads, overlays) require additional post-printing treatment to smooth the outer layers (e.g., using acetone) [39,40].

Many researchers around the world are studying multi-material printing from polymers. The number of printing materials is virtually unlimited [41], especially since it is becoming increasingly accessible and achievable for research units to produce or modify filaments [42-45]. One popular and economical [46] polymer-based multi-material printing technique is FDM/FFF [46-50].

Multi-material 3D printing can modify objects' properties using different polymers [51] and additives in a single printing process [7]. To stereo-complex 3D samples made of PLA, annealing can be used, which involves placing the pieces in a vacuum oven at 50°C for 24 hours, then subjecting the samples to temperatures between 160 and 210°C for one hour. The results show [52] that the acetone vapour treatment process significantly improves the surface finish of the parts, with minimal deviations in the geometric accuracy of the details after treatment. Acetone is a solvent adhesive to bond PLA prints [53]. The contact surfaces are diluted with acetone and connected, then allowed to dry.

Manipulation of printing process parameters and annealing can lead to specific properties and structures of objects [54]. A paper [55] investigated the effect of material contact surfaces in multi-material printing. It proposed a solution in the form of mechanical interlocking systems on bonded layers-three different geometries (T-shape, U-shape, and dovetail shape) were tested. The positive effect of using mechanical interconnections of TPU (thermoplastic polyurethane) and PLA on the flexibility of the samples was demonstrated. The interlocking of material boundaries influenced the achievement of higher Young's modulus values.

Interest in the use of bioplastics and biocomposites is growing. The insufficient strength parameters of this polymer limit the use of PLA. For the polylactide to compete with petroleum-derived plastics, it must be modified.

2. Materials and methodology

This study aims to investigate the adhesion of combining two materials with different properties (PLA-TPU and TPU-PLA) printed in FFF technology with post-processing treatments. The scope of the study includes making four variants of samples (shown to be the best for a given surface development in the study [56]) and subjecting them to three different post-printing treatments, viz:

1) Annealing at 50°C,
2) Annealing at 50°C in acetone vapour,
3) Holding in cold acetone vapour at room temperature.

After the processes mentioned above, shear tests were conducted to determine the bond strength between PLA and TPU materials after the applied treatments.

2.1. Materials

PLA – polylactide is an aliphatic linear polyester, fully biodegradable [57]; it decomposes by biochemical transformations. PLA materials exhibit the properties of traditional plastics, being biocompatible and thermoplastic. Due to its strength properties (bending strength 64-106 MPa, tensile strength about 59 MPa) for many industrial applications, polylactide may be insufficient and should be reinforced or modified [58]. Material modifications are needed to take advantage of the environmental potential of biodegradable polymers, such as the creation of PLA-based composites. Films for printing with PLA are gaining popularity, characterised by good stability during model printing [56], relatively low melting point (180-220°C), and glass transition temperature of 60-65°C [59]. Fused deposition printing allows modification of materials, so manufacturers strengthen the filaments, for example, by adding other polymers, carbon fibres, glass fibres, or metal powders. PLA *Ultimaker 2.85* was used for the research.

TPU – thermoplastic polyurethane exhibits: a high modulus of elasticity, resistance to cracking and abrasion, but also resistance to oils, fats, and organic solvents. At room temperature, TPU behaves like cross-linked elastomers. Still, unlike conventional elastomers, when heated above their softening point, they can be reprocessed with techniques characteristic of thermoplastics, such as injection or extrusion [60].

TPU printing filaments allow the production of flexible models, resistant to abrasion, vibration, and shock [61,62]. An essential feature of the material is its hygroscopicity- the printing process requires pre-preparation of the filament: it needs to be dried and stored in desiccators (water hinders the printing process, the nozzle clogs or unwanted material gaps are formed, weakening models). TPU *Ultimaker 2.85* was used for the research.

2.2. Methods

From a previous study [56], the best configurations for each surface development studied emerged:

- TPU/PLA circles/circles – circular pattern on both planes,
- TPU/PLA lines0°/lines45° – linear pattern 0°/lines 45°,
- PLA/TPU circles/circles – circular pattern on both planes,
- PLA/TPU lines/circles – linear/circular pattern.

Table 1 shows the parameters of printing samples for each material.

Table 1.
Printing parameters (PLA – polylactic acid, TPU – thermo-plastic polyurethane)

Print setting	Nozzle 1: PLA	Nozzle 2: TPU
Top/bottom line width	0.35 mm	0.35 mm
Layer height	0.2 mm	0.2 mm
Wall line court	3	4
Top/bottom layers	5	6
Infill density	70%	100%

The sample was divided into two parts, each entered separately into the Cura system, given the appropriate parameters for each element. Then, using the *Merge Models* function, the sample was merged into one whole object. The Cura software allows different surface development patterns for each material, meaning that a different pattern can be used for the lower and upper cylinders of the sample. Due to the model's geometry, two surface patterns were chosen: linear and circular (*line* and *circles*) and different printing directions. These were applied in various configurations for the two sequences of printed materials.

For the above configurations, an additional 60 samples (15 pieces for each design) were printed and divided into three series for experimental testing depending on the treatment used.

- Series 1: Annealing at 50°C;

  The samples were placed in a GOLDBRUNN Therm vacuum dryer for 24h at 50°C.
- Series 2: Annealing at 50°C in acetone vapor;

  Samples were placed on racks in plastic containers (5 pieces per 500 ml container) along with 20 ml of technical acetone. The shelves inside the containers protected the samples from direct contact with the liquid acetone. The leaking containers were placed in an oven at 50°C for 24 hours. The acetone completely evaporated after about 2 hours from the start of the process.
- Series 3: Holding in cold acetone vapour at room temperature.

  Samples were placed on racks in plastic containers (5 samples in a 500 ml container) along with 20 ml of technical acetone. Shelves inside the containers protected the samples from direct contact with the liquid acetone. The leaking containers were placed in a drying oven at room temperature for 24 hours. Figure 1 shows the prepared container used for acetone vapour treatment and the arrangement of the sample containers in the dryer.

Fig. 1. The arrangement of the containers in the dryer

2.3. Samples

A cylindrical specimen geometry was used for the shear tests. The 3D model was created using Ansys Workbench software in the Space-Claim module (Ansys Inc., Canonsburg, PA, USA). The sample geometry consists of two cylindrical structures superimposed on each other: Figure 2 shows a model of the prepared sample, how to apply the filament (according to the pattern) and materials combinations (PLA/TPU and TPU/PLA).

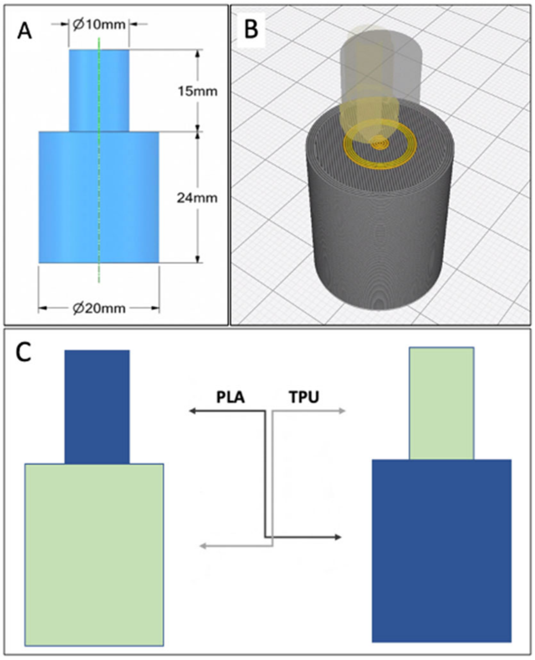

Fig. 2. A) Specimen model used for shear strength tests, B) way of applying the filament (according to the *circles'* pattern), C) materials combinations

2.4. Experiments

To determine the adhesion strength, a shear strength test was carried out on a Zwick/Roell universal testing machine by ISO 29022:2013. Figure 3 shows the position of the knife relative to the specimen for which a displacement of 1 mm/min was set.

3. Results

Analysis of the impact of post-printing treatment included a comparison of the maximum force applied to the sample. Treatment in hot acetone vapour led to most of the pieces disintegrating: all PLA/TPU-based samples, regardless

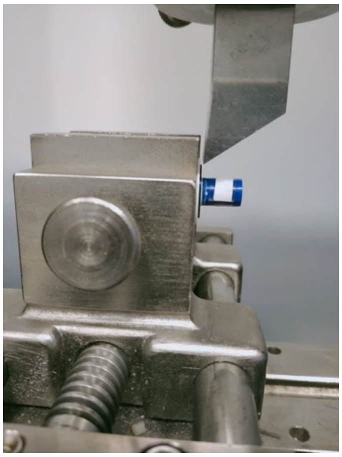

Fig. 3. Shear strength test – Zwick/Roell testing machine

of surface development, disintegrated. In the case of TPU-based models, only a few deteriorated after removal from the dryer and cooling. Finally, four PLA/TPU samples with a linear pattern printed in the 0° and 45° directions (lines0°/lines45°) and three with a circular pattern on both contact surfaces (circles/circles) were tested. The results of the tests are presented in graphs (Figs. 4-7), separately for each configuration of materials and patterns, to compare the properties of the specimens after and without treatment.

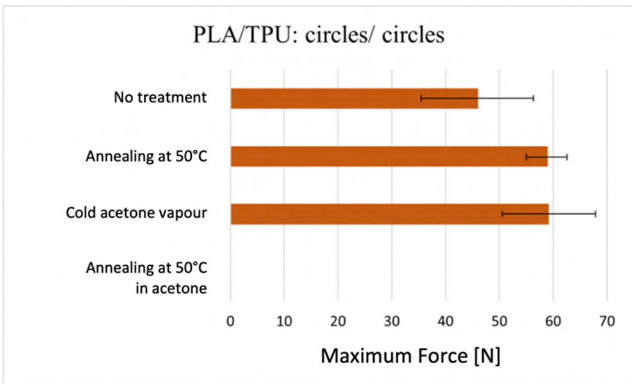

Fig. 4. Maximum applied force: impact of post-print processing. Samples PLA/TPU with pattern circles/circles

As seen in Figure 4, the treatment application positively influenced the specimens' bond strength properties: values of 59.15N and 58.94N were achieved for cold acetone vapour treatment and annealing, respectively. On the other hand, hot acetone vapour led to the disconnection of the layers at the material interface. Annealing and applying cold acetone vapour improved the strength by more than 10 N on average for the two series. When analysing the results and standard deviation, some results in the series overlapped with other series, so the results were subjected to two-sided significance analysis for a level of p=0.05.

The statistical significance for no post-printing treatment versus annealing at 50° is 0.269, while that for cold acetone vapour treatment is 0.379. The importance of the two types of treatment analysed is 0.986. The results are, therefore, statistically insignificant; the averages obtained for the treatments show an improvement in strength when force is applied. Still, the selected samples in the series achieved similar values for each type of treatment.

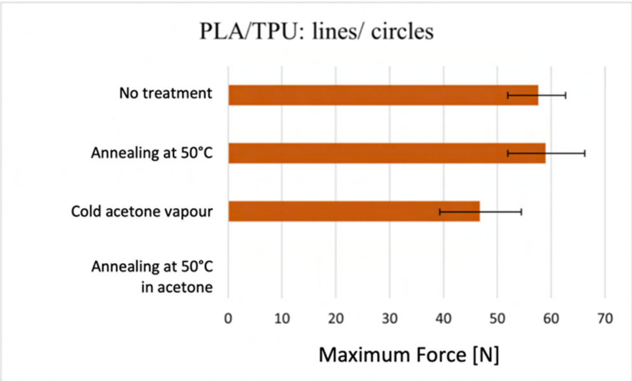

Fig. 5. Maximum applied force: impact of post-print processing. Samples PLA/TPU with pattern lines/circles

From the results presented in Figure 5, it can be seen that treatment in cold acetone vapour of samples with a linear and circular pattern (lines/circles), as opposed to a circular pattern on both surfaces (Fig. 4), worsened the quality of the adhesive grafting and less force was required to shear the top cylinder. Annealing at 50°C positively affected the bonding of the two polymers. The maximum average force applied was 58.96 N, whereas the average for untreated samples was 57.6 N. Cold acetone vapour weakened the bond by almost 10 N. Analysing the results and standard deviation, some development in the s in the series overlapped with other series.

The statistical significance for no post-print treatment versus annealing at 50° is 0.895, while that for cold acetone vapour treatment is 0.202. The importance of the two types of treatment is 0.297. Therefore, the difference in results is statistically insignificant, and tests would need to be carried out on more samples for each series to determine the effect of post-print treatment and its validity.

Analysing the test results for TPU-based specimens (Fig. 6), it can be seen that the models survived annealing in hot acetone vapour. Still, the quality of the bond was significantly impaired. Untreated samples reached 48.74 N, and hot acetone vapour reduced the adhesion: only 5.35 N was applied until the top cylinder was sheared. Heating at 50°C for 24 h translated into a higher maximum force result: 53.5 N. The result is, therefore, ten times higher; the applied temperature improved the quality of the polymer bond, but when analysing the statistical significance for the untreated samples and those annealed at 50°C, it was found to be 0.587, so the results should be considered statistically insignificant. Thus the annealing treatment may improve the strength properties, but a larger sample would need to be tested. Treatment in cold acetone vapour led to a result of 32.24 N. Treatment with acetone is ineffective for TPU/PLA samples with a circles/circles pattern.

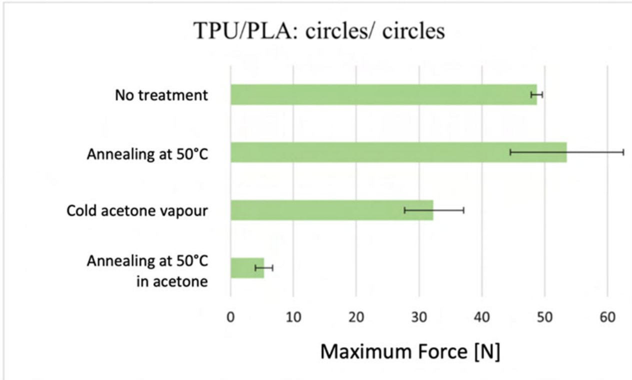

Fig. 6. Maximum applied force: impact of post-print processing. Samples TPU/PLA with pattern circles/circles

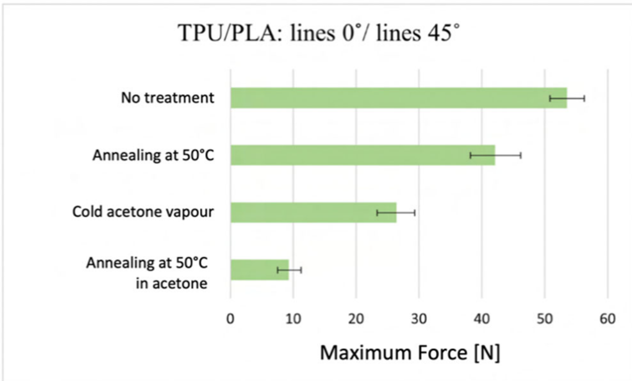

Fig. 7. Maximum applied force: impact of post-print processing. Samples TPU/PLA with pattern lines0°/lines45°

Samples with a TPU base with a linear pattern at 0°/45° (lines0°/lines45°) (Fig. 7) untreated reached 53.57 N, and hot acetone vapour reduced adhesion: only 9.35 N was applied until the top cylinder was sheared. Heating at 50°C for 24 h translated into a higher maximum force result: 42.10 N. The result is, therefore, ten times higher. Treatment in cold acetone vapour led to a consequence of 26.42 N. Treatment with acetone for TPU/PLA samples with this linear pattern proved ineffective. The shear strength results in MPa are shown in Figures 8-11.

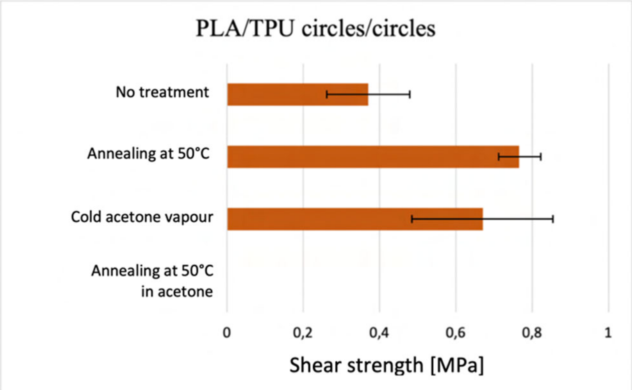

Fig. 8. Shear strength: impact of post-print processing. Samples PLA/TPU with pattern circles/circles

The treatment application positively affected the specimens' strength properties (Fig. 8); values of 0.671 MPa and 0.765 MPa were achieved for cold acetone vapour treatment and annealing, respectively. Annealing in an acetone environment (hot acetone vapour) led to the dissolution of the polymer on the outer parts of the specimens, weakening the bond and disconnection of the layers at the interface of the materials, which made it impossible to test the models. Warming up and applying cold acetone vapour improved the shear strength. When comparing the results and standard deviations, statistical significance should be tested for the untreated and cold acetone vapour-treated samples – this is 0.034, so the result is considered significant, and the cold acetone vapour treatment improves the shear strength for all the samples tested.

The shear strength for the specimen with linear and circular patterns (lines/circles) (Figure 9) was highest for the annealing treatment. Cold acetone vapour also improved the quality of the joint: 0.574MPa was achieved for cold acetone vapour, where untreated specimens- 0.432MPa. The statistical significance for the untreated and annealed samples is 0.043 (significant result), while against cold acetone vapour treatment, 0.185 (non-significant effect). The annealing treatment proved effective for the lines/circles pattern and material order: PLA/TPU and improved adhesive bond quality.

TPU-based specimens (Fig. 10) survived annealing in hot acetone vapour, but the quality of the joint was

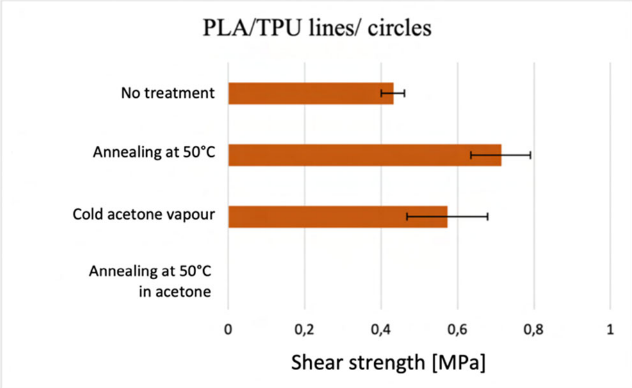

Fig. 9. Shear strength: impact of post-print processing. Samples PLA/TPU with pattern lines/circles

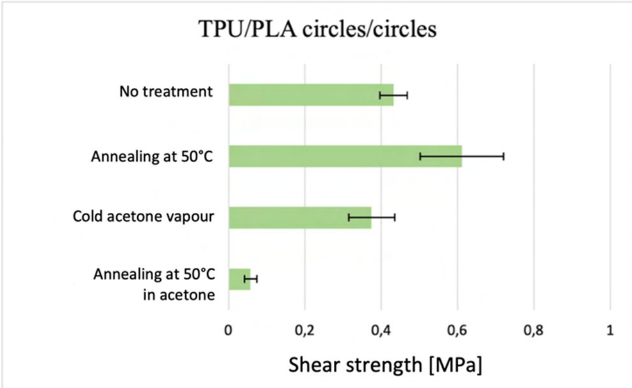

Fig. 10. Shear strength: impact of post-print processing. Samples PLA/TPU with pattern circles/circles

significantly impaired: the shear strength was 0.058 MPa. The annealing treatment increased the shear strength. The highest result for this pattern combination (TPU/PLA circles/circles) was achieved; 0.61 MPa. The statistical significance for the untreated and the annealed samples is 0.027, so the results are significant. Cold acetone vapour reduced the strength from 0.43 MPa (untreated) to 0.37 MPa; the statistical significance against untreated samples is 0.252, so treatment with cold acetone vapour did not lead to conclusive results related to weakening or strengthening of adhesion bonds.

TPU base specimens with a 0°/45° linear pattern (lines0°/lines45°) (Fig. 11) untreated reached 0.629 MPa, and hot acetone vapour reduced the shear strength: 0.102 MPa. Annealing at 50°C for 24 h translated into a shear strength result: 0.493 MPa. The post-printing treatment proved ineffective for TPU/PLA samples with this linear pattern (lines0°/lines45°).

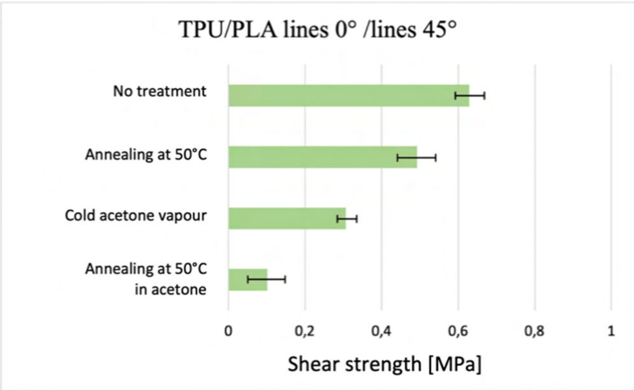

Fig. 11. Shear strength: impact of post-print processing. Samples PLA/TPU with pattern lines0°/lines45°

4. Conclusions

The post-printing treatment results in a stronger inter-material bond and increased adhesion strength; the best average shear strength results were achieved for annealing without acetone and for PLA/TPU samples for treatment in cold acetone vapour. Compared to the untreated samples, all results achieved for the acetone-free annealing are statistically significant ($p<0.05$).

Cold acetone vapour increased the shear strength for PLA/TPU circles/circles samples by 86.4% ($p=0.034$) and for PLA/TPU lines/circles samples by 32.69% ($p=0.185$).

Annealing increased the shear strength for TPU/PLA circles/circles samples by 41.35% ($p=0.027$) and for PLA/TPU circles/circles by 106.5% ($p=0.023$) and for PLA/TPU lines/processes by 65.47% ($p=0.043$).

Hot acetone vapour contributed to the spontaneous disintegration of most of the samples – the use of this treatment could be used to decompose composites and recover individual raw materials after the life of the objects.

Multi-material printing offers the possibility of combining different filaments in a single process, but to achieve satisfactory print quality, it is necessary to study the adhesion between materials. In these considerations, adhesion was compared for different surface developments for two polymers – polylactide and thermoplastic polyurethane – but with the same printing parameters.

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