

Article

Development of Conductive Nanocomposite Filaments from Reused Selective Laser Sintering Powder for Fused Filament Fabrication

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Abstract

In polymer selective laser sintering (SLS), powder acts as a support material during additive manufacturing, generating significant amounts of un-sintered powder exposed to prolonged thermal cycles. Although partial reuse with virgin powder is common practice, material degradation eventually renders the powder unsuitable for further SLS processing. This study investigates a sustainable approach for valorising SLS waste powder through its conversion into filament feedstock for fused filament fabrication (FFF). Poly-amide 12 filaments containing 0, 2, 3, and 4 wt.% multi-walled carbon nanotubes (MWCNTs) were produced by twin-screw extrusion to tailor the electrical conductivity of the polymer matrix. The filaments were processed by FFF to manufacture specimens for thermal, mechanical, and electrical characterization. Differential scanning calorimetry revealed the influence of reprocessing on the thermal behaviour of the reused material and resulting filaments, while thermogravimetric analysis demonstrated improved thermal stability with increasing MWCNT content. Tensile testing showed increased Young's modulus (up to 9.4%), despite an initial drop, and tensile stress at break (up to 56.3%) with increasing nanofiller concentration. In addition, distinct electrostatic-discharge (ESD) protection ranges were achieved depending on the MWCNT loading. The results demonstrate the potential of reused SLS powder as a sustainable feedstock for functional AM materials suitable for ESD-sensitive applications.

Keywords: selective laser sintering; fused filament fabrication; multi-walled carbon nanotubes; electrostatic discharge; sustainability

Academic Editors: Mohammad Khondoker, Md Ahasan Habib and Farid Ahmed

Received: 9 June 2026

Revised: 29 June 2026

Accepted: 3 July 2026

Published: 4 July 2026

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1. Introduction

The mindset of industrial manufacturers and governmental regulations is gradually changing towards environmentally friendly approaches that consider sustainability in both manufacturing and end-of-life of products [1,2]. However, industrial processes are still mostly traditional, employing either formative manufacturing that requires moulds or subtractive manufacturing that generates waste from machining or milling, impairing,

in this way, manufacturing sustainability [3,4]. Distinct from conventional manufacturing, additive manufacturing (AM) has emerged as an alternative and versatile manufacturing approach that enables creation of complex parts, that is commonly referred to as being environmentally friendly due to the controlled material usage and energy efficiency [3,5,6]. AM encompasses a series of distinct processes that share a common production approach, based on the creation of a part, through the deposition of successive layers of material, that are pre-defined by adequate software considering 3D-model data [5,7,8]. Although considered sustainable, the majority of AM processes utilize support material during building, which eventually creates waste [5,9]. With increasing application of AM technologies through the perspective of mass production, the support material waste will become a serious issue that needs addressing [5]. One example of an AM technology that is rapidly expanding and presents issues related to material waste management is selective laser sintering (SLS) [5,6,10,11]. The SLS process resources mostly comprise thermoplastic polyamide powders [5,9,11,12], such as polyamide 12 (PA12), the most frequently employed commercial material due to its inherent well-balanced properties, including good mechanical properties, durability, good dimensional stability, good chemical resistance, low water absorption, heat resistance and easy processing [6,11,13–15]. Other SLS feedstock materials include PA12 mechanically mixed with fillers, for example, aluminum particles to enhance dimensional stability and stiffness [16]. Polyamide 11 (PA11) is a bio-based alternative to PA12 that displays low moisture absorption, yielding good dimensional stability and mechanical properties [17]. However, it has a smaller processing window compared to PA12. Thermoplastic polyurethane (TPU) is another feedstock material considered for applications requiring elastomeric and rubber-like mechanical behaviour [18]. Other materials being explored include polyether ketone (PEEK) and polypropylene (PP) [18,19]. Although this study focuses specifically on PA12, these alternative materials face similar thermal degradation and waste-management challenges. Consequently, exploring their conversion from waste powder into FFF filaments represents a highly promising avenue for future research to further expand the circular economy of additive manufacturing.

In brief, the SLS process involves an initial warm-up stage to preheat the powder bed, followed by the selective sintering of successive layers of powder, by either melting or fusing powder particles based on the thermal energy supplied by a laser, and, finally, a slow cooling-down stage [6,18,20,21]. During the production process, powder layers are deposited in between each selective sintering step, covering the entire building platform area up to the production pre-defined height [20]. The deposited powder that is non-sintered during the production (~80% to ~90%) only contributes to sustaining the parts while being built and may only be reused a certain number of times before being unsuitable for the sintering process, therefore becoming waste [6,8,20,22]. This is because, even though this powder is not sintered, it stills deteriorates due to the long periods of time it is exposed to high temperatures during the entire production process [8,11,18,20,22]. Attempting to mitigate the adverse effects of the high processing temperatures, SLS processes resources in a nitrogen atmosphere; however, prolonged thermal exposure still leads to significant resulting changes to its properties [21]. Residual powders undergo physical and chemical degradation [11], which deteriorates their mechanical and thermal properties and compromises part-surface quality [8,11,22]. An increasing number of processing cycles leads to a decay in the mechanical properties of the printed parts, including a reduction in tensile strength, elongation at break, and Young's modulus [15,22]. Prolonged exposure to high temperatures causes the powder to agglomerate, creating particles with unsuitable, irregular shapes (i.e., potato-shaped or rough-edged particles). This reduces powder flowability, resulting in parts with poor surface finishing (such as an orange-peel texture), higher porosity, and lower dimensional accuracy [8,18,22]. Aged powders also

show lower melt volume-flow rate (MVR) values compared to virgin powder. This indicates an increase in zero-shear viscosity due to post-condensation phenomena, which increases the molecular weight and negatively affects the melt flow [11,21,22]. Chemical degradation changes the polymer structure mainly through thermal oxidation and post-condensation mechanisms, resulting in chain scission, branching, and cross-linking [11]. Additionally, thermal annealing of the powder alters its melting behaviour, leading to an increased melting peak and decreased onset temperatures [21,22]. These combined effects limit the direct reusability of the powders in the SLS process, creating a clear need to explore viable and profitable approaches to reuse these waste powders and ensure process sustainability [21].

The literature [5,6,12,20,22] reports the reuse of powders to improve process sustainability by mixing with non-processed powder in various ratios; for example, 30:70% and 50:50% of virgin powder and un-sintered reused powder respectively being one of the most commonly adopted ratios. However, the quality of mixing ratios is hard to control as reused powders from distinct locations of the batch are subjected to different conditions of temperature per period of time, presenting, therefore, variable properties decay [8,12,22]. The mixed powders eventually will depict a decay in properties that is visible in the dimensional accuracy and surface quality (e.g., orange-peel texture) of the parts, becoming non-viable for a new mix with virgin powders [8,12,18,22]. In addition to this issue, the recommended diameter for SLS powders varies from 45 to 90 μm [6], which is rather complex, expensive and energy-consuming to obtain [6,13,20].

The limitations and concerns regarding both the production and reuse of SLS powders create the need to research new purposes for reused powder in order to improve the sustainability and cost-effectiveness of the SLS process [5,8,11,20,22,23].

An alternative reuse for SLS powders consists of their application as feedstock for other processing technologies. However, it is important to keep in mind that, as the cost of SLS powders is high, even though it is waste material that cannot be reused in the SLS process, the application of this material for other manufacturing processes should consider a technology that enables cost recovery or at least reduces the cost loss [11,13,24]. Another AM process, i.e., material extrusion (e.g., fused filament fabrication, FFF), is a feasible possibility for reusing SLS powders to create quality filaments for the production of high-value parts. This approach will bridge distinct AM processes, enabling overall reduction in AM environmental footprint while improving economic efficiency [5,13,20,24].

Several studies may be found in the literature, in which researchers describe the development of filaments for FFF with SLS reused powder. For example, Feng et al. [13] prepared filaments for FFF and pellets for injection moulding with reused PA12 SLS powder for comparison analysis of performance and suitability of the reuse of SLS powder as feedstock for distinct technologies. Praça et al. [23] also investigated the feasibility of processing non-sintered polyamide 12 by injection moulding and FFF. Injection moulding demonstrated consistent mechanical behaviour, while in FFF, higher variability and reduced ductility were obtained. Other published studies report the inclusion of fillers to enhance specific properties of the filament produced with reused SLS powder. Kumar et al. [5] mixed PA12 powders from SLS with various weight percentages of tungsten carbide to enhance mechanical properties of filaments. With the same purpose, Wang et al. [25] created various composite filaments based on compounded PA12 powders with different contents of milled carbon fibre (mCF). In one study, Magi et al. [9] developed various filaments with PA12 powders from SLS combined with thermoplastic polyurethane (TPU) pellets and aramid fibres to alter the mechanical properties of PA12, with the final purpose of creating a material suitable for the production of hand prostheses. In another work, Rahim et al. [26] described the development of composite filaments combining PA12

powders from SLS with two bioceramic fillers to improve mechanical and bioactivity properties for safe biomedical use. Also, for the purpose of biomedical applications, Abdullah et al. [27] researched the feasibility of production of filaments with PA12 from SLS, filled with high contents of ceramic fillers for craniofacial reconstruction purposes. Finally, Uddin et al. [24] reinforced PA12 powders with various amounts of magnesium (Mg) particles to improve mechanical properties (strength and stiffness) for biomedical applications, such as casts and orthotics.

Taking into account the evident need to improve SLS-process sustainability and the possibility to expand the material portfolio for AM, the present study focused on creating a new purpose for reused PA12 SLS powders, based on the addition of various weight percentages (2, 3 and 4 wt.%) of multi-walled carbon nanotubes (MWCNT) to alter the electric properties of the polymer matrix, in order to create functional filament feedstock material for FFF that is suitable for electrostatic-discharge (ESD) applications. This target application was defined because industries that work with electronic components and assemblies (e.g., the automotive industry) have a critical need related to the use of materials, e.g., for packaging, jigs and fixtures that, when in contact with the electronics, are ESD-safe and avoid damage by electrostatic fields or discharges [28]. This paper reports the production of various nanocomposite filaments based on conventional extrusion and the comparative analysis based on the results of thermal, mechanical and electrical properties of the developed filaments and printed test specimens.

This approach to valorising aged PA12 SLS powder establishes a new utility for materials that would otherwise be discarded. PA12 powder is an energy-intensive feedstock, making it relatively expensive, typically ranging from 50 € to 100 € per kg [21]. The thermal degradation associated with powder ageing not only presents a significant sustainability challenge but also an economic one, as the material prematurely loses its SLS processability [11,29]. Therefore, developing a cost-effective strategy to repurpose this waste by modifying its properties for alternative additive manufacturing technologies, such as FFF, can significantly mitigate the environmental and economic impact of SLS-related waste.

2. Materials and Methods

2.1. Materials

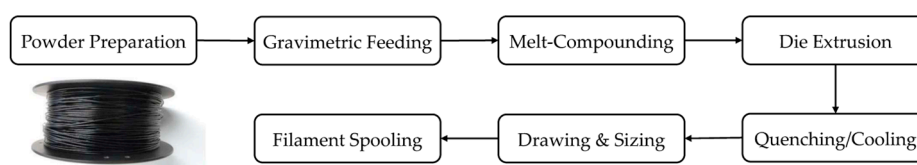
The selected polymer matrix consists of an unfilled polyamide 12 (PA12) powder with an average grain size of 56 µm, specifically developed for laser-sintering processes (i.e., PA 2200 supplied by EOS GmbH, Krailling, Germany). Parts manufactured with standard conditions of production typically present a well-balanced combination of properties, including a Young's modulus of 1650 MPa, a tensile strength of 48 MPa, a strain at break of 18% and a flexural modulus of 1500 MPa [30]. The selected powder consisted of an aged mixture (50:50% ratio of virgin and un-sintered PA2200 powder) that has endured a series of sintering processing cycles on an EOS P 396 machine (EOS GmbH, Krailling, Germany) until the SLS processing quality was hampered. To improve the electrical conductivity by forming electrically conductive paths on PA2200, a carbon-based nanofiller, MWCNT NC7000™ series supplied by NANOCYL®, Sambreville, Belgium, was selected. These multi-walled carbon nanotubes (MWCNT) are synthesized via the Catalytic Chemical Vapor Deposition (CCVD) process and present specific structural, physical, and electrical characteristics, summarized in Table 1. Depending on the type and properties of the polymer matrix and the dispersion method, loadings between 0.5 wt.% and 4.5 wt.% allow for improvement of the electrical conductivity of the polymer matrix with a low percolation threshold [31].

Table 1. Technical parameters and physical properties of the multi-walled carbon nanotubes (MWCNT NC7000™).

Property	Value and Unit	Reference
Average outer diameter	9.5 nm	[31,32]
Average length	1.5 μm	[31–33]
Carbon purity	90%	[31,33]
Specific surface area	250–300 m^2/g	[31,33]
Bulk density	66 kg/m^3	[34]
Volume resistivity	$10^{-4} \Omega\text{-cm}$	[31]

2.2. Filament Production

Several filament productions were defined considering the integration of different amounts of MWCNT, including 0 wt.% (for control), 2 wt.%, 3 wt.% and 4 wt.%, referred to in this paper as PA12 + 0 wt.%MWCNT, PA12 + 2 wt.%MWCNT, PA12 + 3 wt.%MWCNT, and PA12 + 4 wt.%MWCNT, respectively. Figure 1 presents a process flow diagram for the fabrication of the nanocomposite filaments, illustrating the sequential stages from powder preparation to feedstock spooling, and includes a photograph of one of the produced nanocomposite filaments.

**Figure 1.** Schematic overview of the step-by-step filament production procedure.

For the powder preparation, PA2200 was dried at 50 °C overnight in a laboratory convection oven prior to processing to prevent moisture-induced hydrolytic degradation during melting. To ensure precise compositional accuracy, PA2200 was gravimetrically fed to a hopper of the extruder machine with a twin-screw compact weigh feeder K-Tron K-CL-24-KT20, while the MWCNT nanofillers were fed to a secondary hopper with a twin-screw loss-in-weight feeder K-Tron K-ML-KT20. Both feeders were operating at a rate of 3 kg/h. The melt-compounding was carried out with a Coperion ZSK 26 Mc co-rotating twin-screw extruder (Stuttgart, Germany) (Figure 2a). Twin-screw systems are essential for nanocomposite processing, as they provide the high shear forces necessary to break up nanoparticle agglomerates and achieve a uniform dispersion of MWCNTs within the highly viscous PA2200 melt [35]. The melt-compounding was conducted with a uniform temperature of 200 °C, a twin-screw extruder speed of 180 rpm and a pressure of 30–31 bar at the extrusion die. The homogeneous melt was extruded through a two-hole extrusion die which allowed the simultaneous production of two monofilaments with 3 mm of nominal diameter. Because precise dimensional tolerance is critical for FFF, the extruded filaments were immediately submerged in a water-cooling system (Figure 2b) to quench the polymer and freeze its morphology. Simultaneously, the filaments were drawn by motorized pull-rollers. By precisely adjusting the speed of these pull-rollers relative to the extrusion rate, the filaments were stretched from their 3 mm-die diameter down to the targeted final FFF-compatible diameter of 1.75 ± 0.1 mm before being finally spooled for storage.

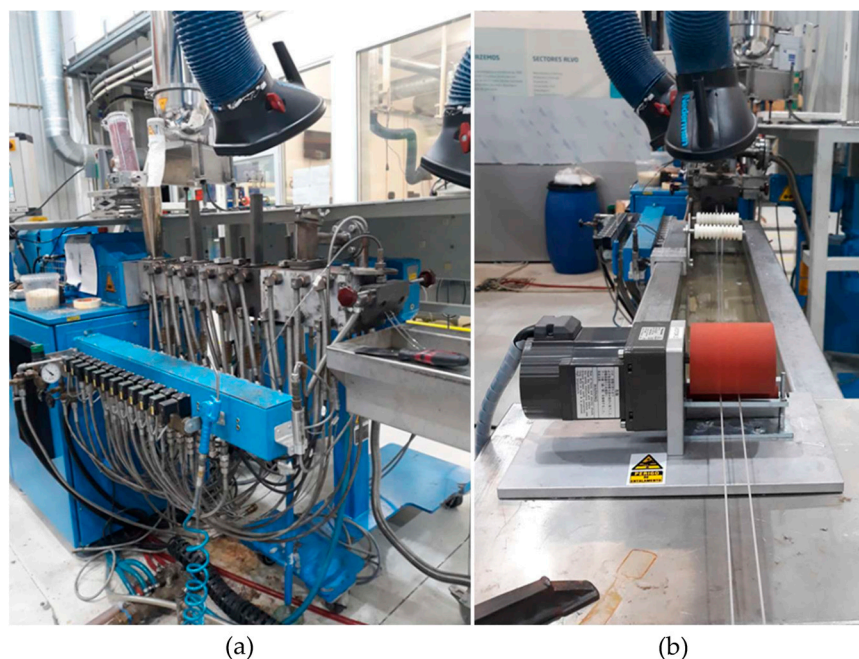


Figure 2. Photographs of the experimental setup: (a) Coperion ZSK 26 Mc co-rotating twin-screw extruder; (b) water-cooling system and motorized pull-rollers.

2.3. Test Specimen Production

Test specimens for electrical and mechanical characterization (five per test) were produced by means of FFF technology, with the open-system Roboze One + 400Xtreme (Roboze, Bari, Italy) (Figure 3a).

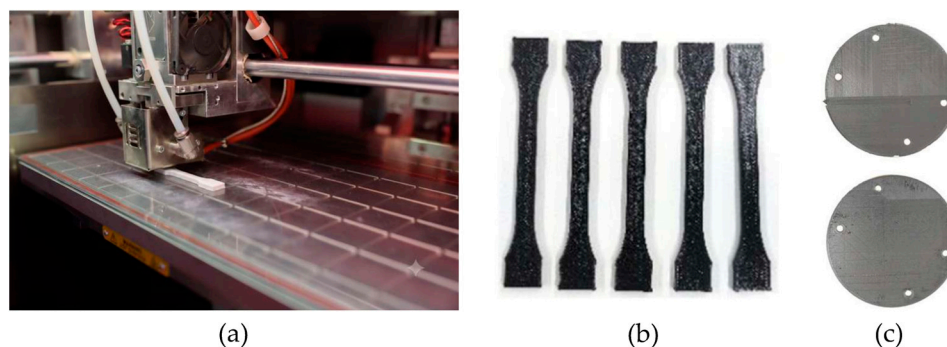


Figure 3. Photographic overview of experimental setup and test specimens: (a) internal view of the Roboze One + 400Xtreme 3D printer during operation; (b) representative FFF-printed tensile test specimens; and (c) FFF-printed circular discs for electrical characterization.

The process was optimized based on general knowledge about the material and by varying the process parameters, such as extrusion temperature, platform temperature and processing speed, to improve printing quality and reduce warping. The optimal parameters are presented in Table 2. A constant raster angle was utilized to isolate the influence of the varying MWCNT concentrations on the material performance. Specifically, an alternating $\pm 45^\circ$ layer-to-layer deposition pattern was selected, as this configuration generally provides more balanced mechanical behaviour compared to unidirectional raster orientations [36]. Following extrusion, the composite filaments were immediately stored in vacuum-sealed bags containing silica gel desiccant to prevent ambient moisture absorption. Prior to FFF production, the filaments underwent a final conditioning cycle at 50 °C for 4 h to eliminate any residual surface moisture, thereby preventing moisture-induced printing defects such as internal voids and ensuring optimal layer adhesion in the final

test specimens. The filament was maintained at the same conditions during the entire building process. Figure 3b,c show representative examples of the produced test specimens.

Table 2. Optimized FFF process parameters.

Printing Parameters		Value and Unit
Infill		100%
Deposition angle		−45°/+45°
Layer height		0.18 mm
Printing temperature	220 °C ¹	240 °C ²
Platform temperature	70 °C ¹	90 °C ²
Printing speed	3000 mm/min ¹	4200 mm/min ²

¹ Values corresponding to PA12 + 0 wt.%MWCNT. ² Values corresponding to PA12 + 2 wt.%MWCNT, PA12 + 3 wt.%MWCNT, and PA12 + 4 wt.%MWCNT.

2.4. Characterization

2.4.1. Filament Diameter

The diameter of all the produced filaments was assessed to evaluate the uniformity of the diameter along the length. A digital calliper was used for the 50 consecutive measurements taken at every 10 mm. Average and standard deviation were calculated.

2.4.2. Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was done to understand the thermal behaviour of the different filaments produced and also the thermal properties of the powder used to produce the filaments. Melting temperature and corresponding enthalpy and crystallization temperature were measured for a powder sample of PA2200 and for all the filaments produced. The degree of crystallinity (X_c) was calculated for all tested samples based on the following formula:

$$X_c = \frac{\Delta H_f}{\Delta H_f^0} \times 100\%, \quad (1)$$

where ΔH_f^0 is the value of the fusion enthalpy of 100% crystalline polyamide 12 reported in the literature [37] and ΔH_f is the fusion enthalpy measured for each sample.

The analysis was carried out with samples of approximately 10 mg sealed in aluminum pans, under a nitrogen atmosphere, and considering a first heating cycle from 20 °C to 230 °C at a heating rate of 10 °C/min followed by a cooling step at a rate of 20 °C/min. A Netzsch DSC 200 F3 Maia (Selb, Germany) differential scanning calorimeter was used, following the ASTM D3418 [38] standard test method.

2.4.3. Thermogravimetric Analysis

The thermal stability and amount of nanofiller was analyzed by means of thermogravimetric analysis (TGA) with TA Instruments Q500 equipment (New Castle, United States of America) and considering test norm ISO 11358-1 [39]. The test was performed under a nitrogen atmosphere with a flow rate of 60 mL/min for the sample and 40 mL/min for the balance and considering heating from 40 °C to 700 °C at 10 °C/min.

2.4.4. Tensile Test

Mechanical evaluation was performed by tensile testing of five 1BA tensile specimens manufactured by FFF. The uniaxial test was performed at room temperature (~25 °C) with an Instron 5969 Universal Testing System (Massachusetts, United States of America) and

in accordance with ISO-527-1 [40]. Tensile specimens were tested at 5 mm/min, with a load cell of 50 kN, and gauge length of 56 mm.

2.4.5. Electrical Properties

Volume and surface resistivity were determined for five specimens produced ($100 \times 100 \times 0.5$ mm) per developed filament. The test was performed at room temperature by using the Keithley 2635B System SourceMeter and the Model 8009 resistivity test fixture from Keithley Instruments Inc. (Ohio, United States of America), and considering the methods and calculations described in the test norm ASTM D257 [41]. Measurements of the surface resistivity were performed by applying a potential of 10 V over the test sample and measuring the corresponding current intensity. Then, the surface resistivity was calculated for the electrode geometry of the Model 8009 resistivity test fixture. Curves of voltage versus current were plotted considering measurements of the electrical current obtained from sweeping the potential from -10 V to 10 V at 0.5 V increments. Then, the volume resistivity was calculated from the slope of the plotted curves and considering the geometry of the sample.

3. Results and Discussion

3.1. Filament Diameter

The filament diameter was measured during filament-processing optimization. Once a suitable value close to 1.75 mm was achieved, the process was considered stabilized. The produced filaments' diameter was again evaluated after 48 h, showing a decrease compared to the value measured during the filament extrusion, which is related to material shrinkage. Diameter measurement results are presented in Table 3. The recommended diameter for FFF filaments is 1.75 ± 0.05 mm [24,42], still being considered acceptable at 1.75 ± 0.1 mm [42]. Considering this, all filaments presented a diameter average within the acceptable range. PA12 + 0 wt.%MWCNT exhibited a mean diameter corresponding to the lower limit of the recommended diameter for FFF filaments but fully within the acceptable range. PA12 + 2 wt.%MWCNT exhibits the best dimensional control, fully complying with the recommended diameter for FFF filaments. Increasing MWCNT content beyond 2 wt.% leads to slightly reduced filament diameter and increased variability, although still within acceptable limits.

The increased dimensional variability at higher MWCNT loadings may be attributed to fundamental changes in the melt flowability of the nanocomposite. If the nanofiller concentration exceeded the percolation threshold, the high-aspect-ratio nanofillers form a rigid, interconnected physical network that severely restricts polymer chain mobility. This network formation dramatically increases the zero-shear melt viscosity and alters the viscoelastic response of the matrix. Consequently, extrusion instabilities, including irregular swell behaviour and unstable pulling of the filament, can lead to diameter fluctuations. This increased resistance to deformation makes it physically more challenging for the pull-rollers to draw the extrudate at a perfectly constant rate, which manifests as a higher standard deviation in the final filament diameter [43,44].

Table 3. Filament diameter measurements.

Nanocomposite Filament	Value and Unit
PA12 + 0 wt.%MWCNT	1.70 ± 0.04 mm
PA12 + 2 wt.%MWCNT	1.73 ± 0.03 mm
PA12 + 3 wt.%MWCNT	1.71 ± 0.06 mm
PA12 + 4 wt.%MWCNT	1.71 ± 0.06 mm

3.2. Differential Scanning Calorimetry

The DSC curves corresponding to the heating cycle (Figure 4) present a single endotherm peak of fusion for all the samples analyzed. The value of melt temperature (i.e., T_m) measured for the reused un-sintered PA12 powder (190.3 °C) is higher than the value obtained in a previous characterization of virgin powder (186.7 °C) [22]. This change is indicative of the reprocessing effect over material properties. It is noticeable that fusion begins earlier for all the filaments (170.98 ± 0.86 °C) in comparison to the powder (181.9 °C). The peak of fusion or melting temperature is higher for the powder sample than for the filament without nanofiller content (182.3 °C), suggesting that the conventional extrusion process influenced the thermal behaviour of the material, possibly due to additional thermo-mechanical degradation and changes in the polymer structure induced during reprocessing.

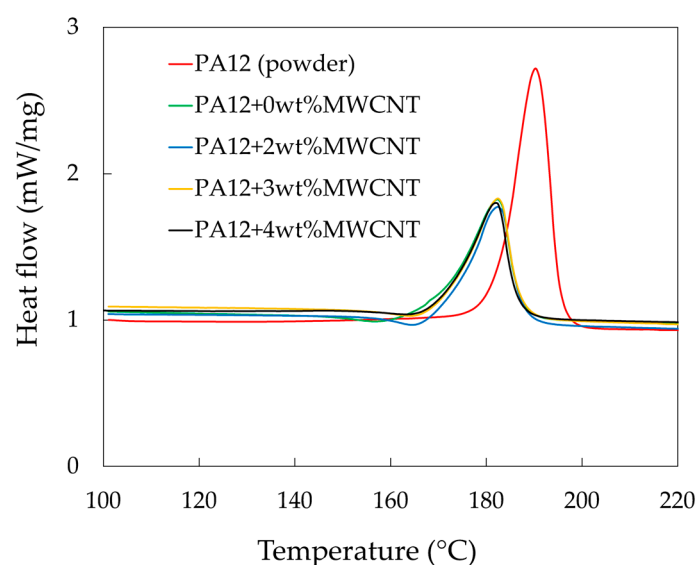


Figure 4. DSC thermograms for the PA2200 powder and all the produced filaments.

Comparing all nanocomposite filaments, the peak of fusion (Figure 5a) is quite similar for all samples, independent of the amount of nanofiller (182.3 ± 0.26 °C). The higher peak of fusion and heat of fusion of the powder sample suggests the presence of a highly arranged crystalline structure, verified also by the calculation of the degree of crystallization (Figure 5b): 45.05% for powder; 27.43% for filament without filler and $23.28 \pm 1.26\%$ for the nanocomposite filaments. For the filaments, it is possible that during the conventional extrusion process there was a relaxation of the polymeric chains and that during the immediate cooling after processing the filaments somewhat limited the formation of organized crystallites. In addition, with the increasing addition of nanofiller, a slight decrease in the degree of crystallinity was verified: 24.34%, 23.61% and 21.89% for 2, 3 and 4 wt.% of nanofiller, respectively. Regarding the crystallization temperature (i.e., T_c), it assumes the lowest value for the powder sample (138.1 °C) while for filaments, nanocomposite filaments present a higher value of ~ 7.5 °C than the filament without filler content. The increase in crystallization temperature with the integration of MWCNTs suggests that the carbon nanofillers act as effective heterogeneous nucleating agents, accelerating the onset of crystallization during cooling [45]. However, the overall degree of crystallinity of the composite filaments is lower than that of the neat powder and decreases with higher MWCNT loadings (Figure 5b). This phenomenon is attributed to the dual role of carbon nanotubes in polymer crystallization. While the high surface area of MWCNTs provides abundant nucleation sites to trigger early crystal growth, the formation of an interconnected nanotube network restricts the molecular mobility of the PA12 chains. This

physical confinement hinders the chain diffusion and reduces spherulite growth rate, thereby limiting the final degree of crystallinity [46].

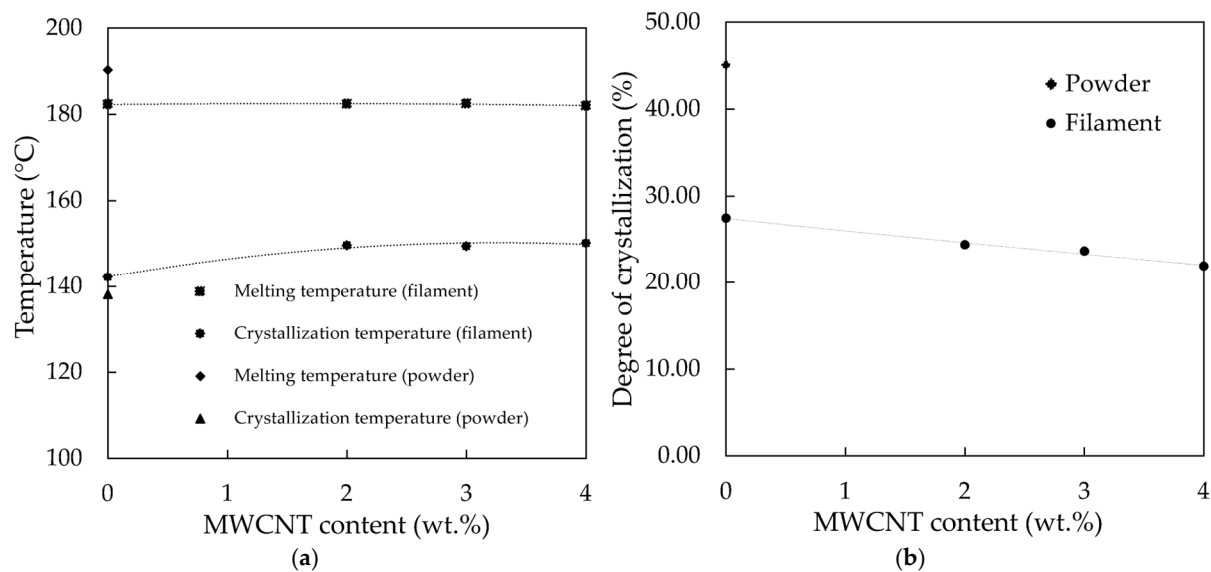


Figure 5. Effect of MWCNT content over: (a) melting and crystallization temperatures; and (b) degree of crystallization.

3.3. Thermogravimetric Analysis

The results obtained from the TGA analysis (Figure 6a) suggest that, up to a certain amount of MWCNT, the thermal stability of the polymer matrix is improved as degradation starts and reaches maximum rate at higher temperatures (above 400 °C). However, when the theoretical content of nanofiller is above 3 wt.%, the thermal stability of the polymer starts to diminish, meaning that there is a limit to which the nanofiller can improve thermal stability. The enhancement of the thermal stability can be related to the good thermal conductivity of the nanofiller, as may be confirmed by the literature [47]. Regarding the quantity of MWCNT (Figure 6b), it was verified that the real content was slightly above the expected content for all the previously defined theoretical values. A measured value of 2.3 wt.%, 3.8 wt.% and 4.5 wt.% were obtained for the filaments with set theoretical values of 2 wt.%, 3 wt.% and 4 wt.%, respectively. This may be related with processing-related effects, including difficulties in achieving homogeneous dispersion of the primary agglomerates of MWCNT, which can contribute to local composition heterogeneity [48].

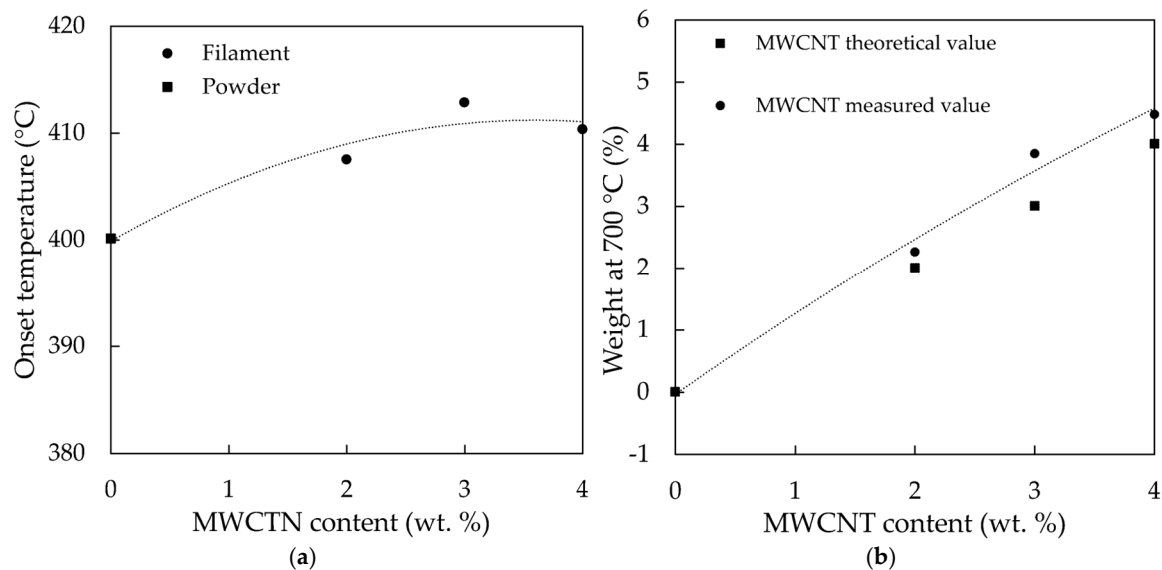


Figure 6. TGA results: (a) variation in the onset temperature according to the nanofiller quantity; and (b) comparison of theoretical and real values of MWCNT weight percent at 700 °C.

3.4. Tensile Test

Mechanical characterization based on uniaxial tensile testing enabled evaluation of the effect of the MWCNT content over certain properties of the distinct nanocomposite test specimens. Representative stress–strain curves for each material formulation are presented in Figure 7 showing that the incorporation of MWCNTs fundamentally alters the macroscopic tensile behaviour of the matrix, illustrating a clear visual transition in both stiffness and yielding behaviour as the nanofiller concentration increases.

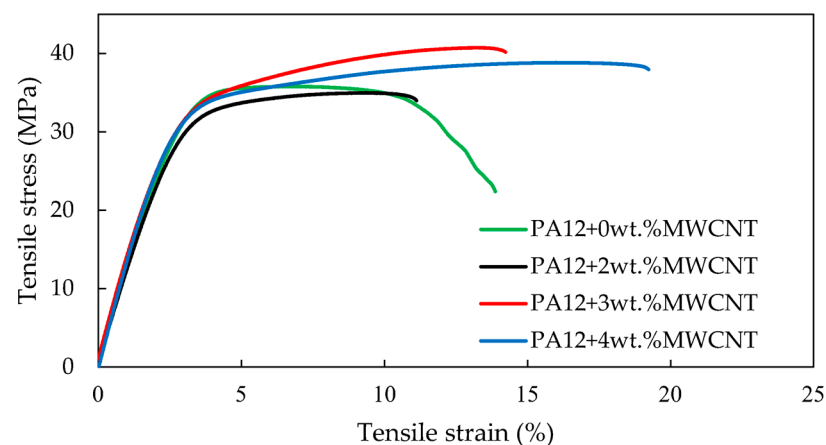


Figure 7. Representative engineering stress–strain curves of the FFF-printed specimens for the recycled neat PA12 and the PA12/MWCNT nanocomposites at varying filler concentrations.

The average and standard deviation of the Young's modulus, yield stress and strain, break stress and strain and tensile strength were determined from the stress–strain curves obtained from five test specimens, according to the calculations described in ISO527-1 [40] (Figures 8 and 9). The neat polyamide filament exhibited a Young's modulus of 1281.36 ± 36.61 MPa (Figure 8a). A slight decrease was observed for the PA12 + 2 wt.%MWCNT nanocomposite filament, which presented a value of 1115.33 ± 43.31 MPa. However, for nanofiller contents of 3 wt.% and 4 wt.%, the Young's modulus increased to 1401.98 ± 35.46 MPa and 1381.48 ± 28.27 MPa, respectively. Although the incorporation of 2 wt.% MWCNT resulted in a reduction in Young's modulus, the overall trend indicated an

increase in stiffness with increasing nanofiller content, consistent with findings reported in the literature [45,49]. As the degree of crystallinity decreased with the incorporation of nanofiller, the observed increase in Young's modulus and stiffness cannot be directly attributed to the crystallinity effects. Instead, this behaviour is likely associated with the reinforcing effect of the MWCNTs and their dispersion within the polymer matrix. Similar behaviour was observed for the tensile strength (Figure 8b). A slight decrease was verified for the PA12 + 2 wt.%MWCNT (34.75 ± 0.64 MPa) compared with the neat polyamide filament (35.66 ± 0.77 MPa), whereas increased tensile strength values were obtained for PA12 + 3 wt.%MWCNT (40.97 ± 0.69 MPa) and PA12 + 4 wt.%MWCNT (38.62 ± 1.58 MPa). The improvement in tensile strength with increasing MWCN content is consistent with results reported in the literature [49].

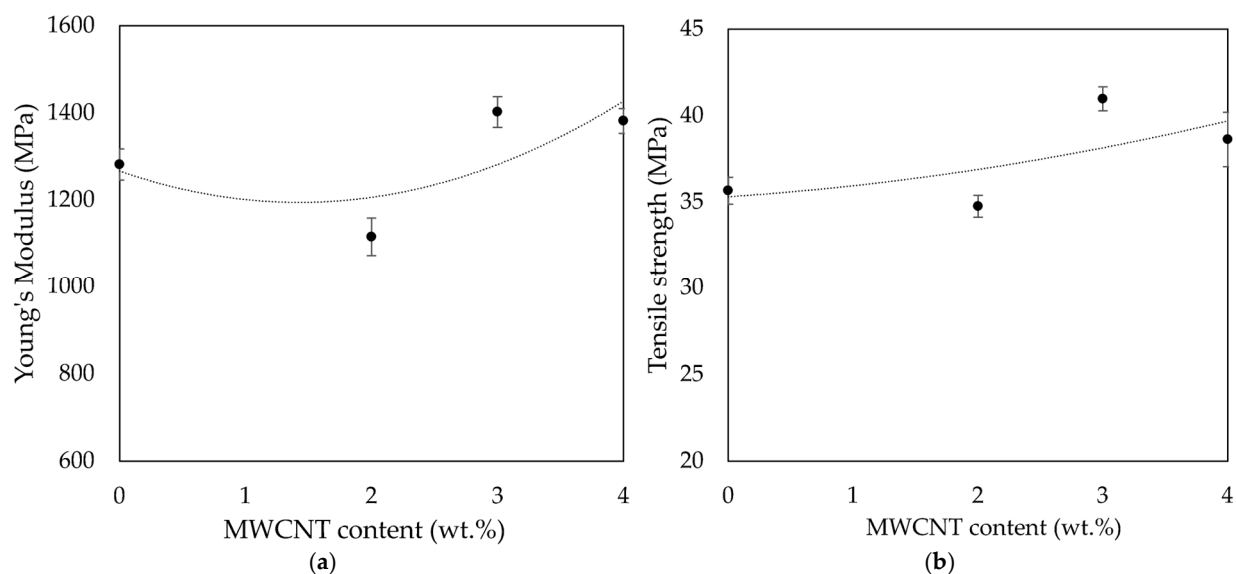


Figure 8. Tensile test results: (a) Young's modulus; and (b) tensile strength as a function of the nanofiller content.

Regarding stress (Figure 9a) and strain (Figure 9b) at yield, PA12 + 2 wt.%MWCNT exhibited an increase (28.22 ± 0.76 MPa; $2.74 \pm 0.13\%$), whereas a decrease was observed for PA12 + 3 wt.%MWCNT (25.05 ± 0.63 MPa; $1.99 \pm 0.06\%$) and PA12 + 4 wt.%MWCNT (24.23 ± 2.10 MPa; $1.95 \pm 0.16\%$), compared with PA12 + 0 wt.%MWCNT (25.18 ± 0.78 MPa; $2.17 \pm 0.08\%$). The literature reports both increasing and non-monotonic trends in yield properties with MWCNT incorporation [33,35], depending on dispersion state, processing conditions, and matrix–filler interactions.

Strain at break (Figure 9b) showed an average value of $14.13 \pm 1.17\%$ for the neat polyamide. A decrease was observed for 2 wt.% MWCNT ($11.11 \pm 0.86\%$) while higher values were obtained for 3 wt.% and 4 wt.% ($14.27 \pm 3.81\%$ and $17.82 \pm 4.60\%$, respectively). The literature generally reports a slight reduction in this property when increasing MWCNT content [35].

Finally, tensile stress at break (Figure 9a) increased for all the nanocomposite filaments (33.58 ± 0.89 MPa, 40.29 ± 0.68 MPa and 37.88 ± 1.33 MPa for 2, 3 and 4 wt.% of MWCNT, respectively) compared with the neat polyamide filament (25.78 ± 4.05 MPa). It is noticeable, for all the evaluated mechanical properties, that there is a variation in results when integrating 2 wt.% in comparison to when adding 3 and 4 wt.%. The non-monotonic behaviour observed across all mechanical properties, particularly at 2 wt.% MWCNT, may be associated with variations in filler dispersion and microstructural development during extrusion, which can influence stress transfer efficiency [45].

Polyamides are inherently hygroscopic [50]; however, PA12 exhibits a more amorphous structure compared to highly crystalline polyamides like PA6, as it is primarily its long aliphatic carbon chains that offer better dimensional stability and significantly lower moisture absorption [51]. If not addressed properly, absorbed moisture can greatly affect the macroscopic properties of polyamides, acting as a plasticizer that lowers mechanical stiffness. Furthermore, during high-temperature FFF processing, the rapid evaporation of this moisture can induce printing defects, including poor surface finish and structural voids caused by bursting vapor bubbles [29,51]. For this reason, careful vacuum storage of the extruded filaments and rigorous drying prior to processing were implemented to prevent moisture absorption [51]. Additionally, the incorporation of MWCNTs provides a physical barrier effect, which helps mitigate potential moisture uptake and preserves both the mechanical and functional integrity of the printed ESD-safe components [52].

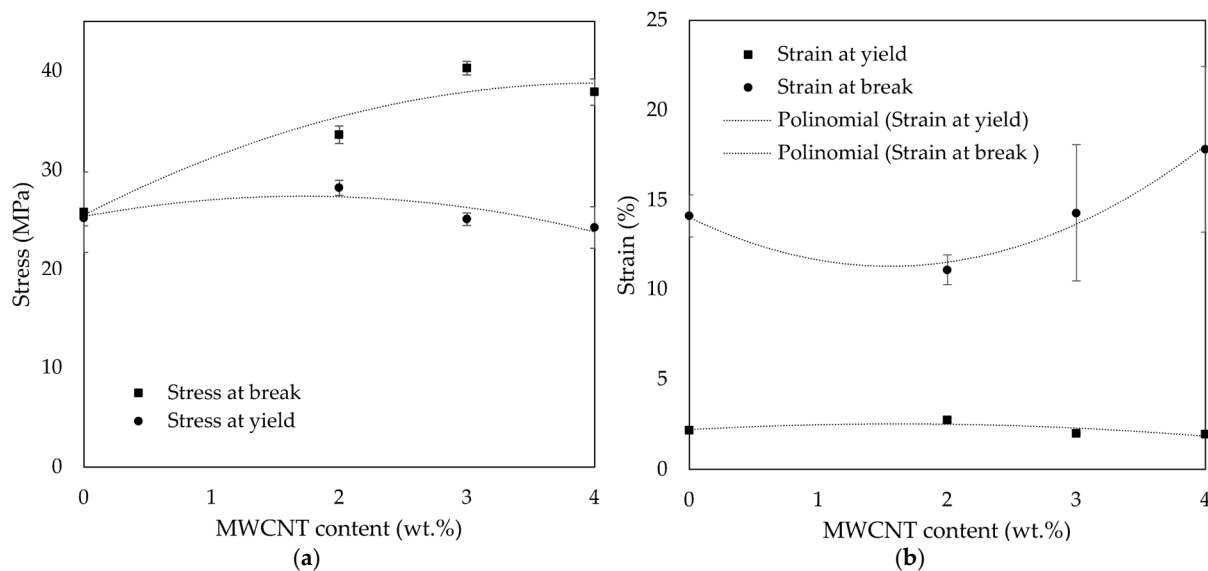


Figure 9. Tensile test results: (a) tensile stress at yield and break; and (b) tensile strain at yield and break as function of the nanofiller content.

3.5. Electrical Properties

The electrical properties of the neat polyamide and nanocomposite filaments were evaluated through the determination of surface resistivity, surface resistance, and volume resistivity. The average values obtained are presented in Figure 10 and Table 4.

Electronic systems are typically classified as electrostatic-discharge sensitive devices (ESDS), which are susceptible to damage from electrostatic fields or discharges [53]. The level of electrostatic-discharge (ESD) protection provided by a material is determined by its electrical resistance. According to the classification criteria established in IEC 61340-5-1 [54], materials are categorized into three categories: namely, insulating (surface resistance $\geq 10^{11} \Omega$), electrostatic dissipative (surface resistance between $10^5 \Omega$ and $10^{11} \Omega$) and electrostatic conductive (surface resistance between $10^2 \Omega$ and $10^5 \Omega$). For the protection of sensitive electronic components, the electrostatic dissipative range is the primary target, as it facilitates a controlled, slow dissipation of electric charges, thereby preventing the high-current sparks associated with rapid discharges [53].

Considering this, the neat polyamide exhibited insulating behaviour, with a surface resistivity on the order of $10^{12} \Omega/\text{sq}$ and correspondingly high surface resistance and volume resistivity values, consistent with the classification criteria of IEC 61340-5-1 [54].

The incorporation of MWCNTs significantly modified the electrical response of the polyamide matrix. The addition of 2 wt.% MWCNT reduced the resistivity by several orders of magnitude, shifting the material into the electrostatic dissipative range and

indicating the formation of initial conductive pathways throughout the polymer matrix. Increasing the MWCNT content to 3 wt.% resulted in a further pronounced reduction in resistivity, leading to borderline conductive behaviour and suggesting the establishment of a percolated conductive network. In contrast, increasing the filler content from 3 wt.% to 4 wt.% produced only marginal changes in the electrical properties, indicating that the electrical percolation threshold had already been reached and that further additions of nanofiller provided limited improvements in conductivity.

Overall, the results demonstrate that increasing the MWCNT content progressively enhances the electrical conductivity of the polyamide matrix, promoting a transition from insulating to electrostatic dissipative and ultimately conductive behaviour. This tunability enables the development of materials suitable for different levels of protection of electrostatic-discharge-sensitive devices [28,54]. The specific proportion of MWCNTs serves as the primary mechanism to engineer these protection levels. The electrical percolation threshold occurs between 2 wt.% and 3 wt.%, where the formation of an interconnected nanotube network leads to a sudden, significant increase in conductivity. Below this threshold (i.e., at 2 wt.%), the network is in the early stages of formation, placing the material in the electrostatic dissipative range. As the filler content increases to 3 wt.%, the nanotubes establish a fully percolated conduction path that drops the resistivity into the optimal dissipative/conductive transition zone. Increasing the loading further (e.g., to 4 wt.%) saturates this internal network, resulting in marginal changes in conductivity as the percolation threshold has been effectively surpassed [55].

It should be noted that thermogravimetric analysis revealed slight differences between the nominal and actual MWCNT contents, with measured residue values of 2.3 wt.%, 3.8 wt.%, and 4.5 wt.% for the formulations containing nominally 2 wt.%, 3 wt.%, and 4 wt.% MWCNT, respectively. These results indicate a slight enrichment of nanotube content in the final filaments. Consequently, the observed transition to dissipative and conductive behaviour should be interpreted considering the actual filler concentrations, suggesting that the effective percolation threshold occurs at approximately 3.8 wt.% MWCNT. Nevertheless, the overall trend of increasing electrical conductivity with increasing nanotube content remains unchanged.

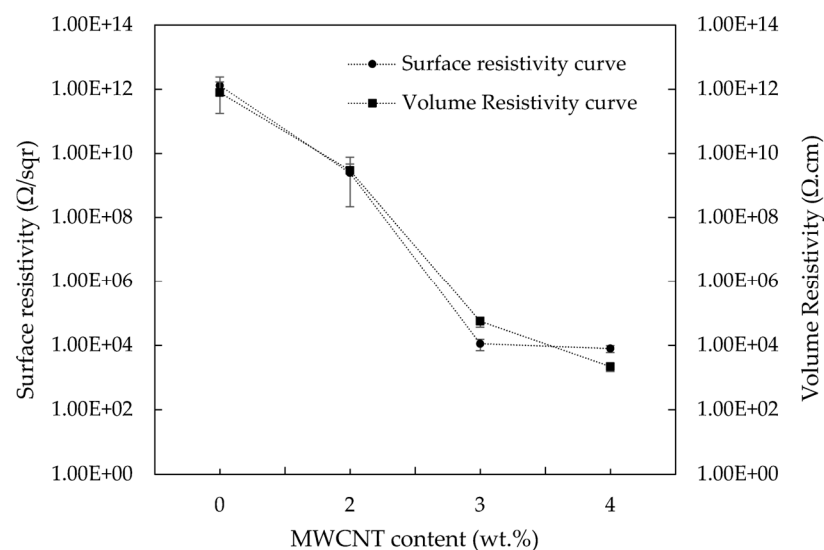


Figure 10. Influence of the nanofiller content on the value of surface and volume resistivity.

Table 4. Determined values of electrical characteristics for the test specimens produced with all the developed filaments.

Nanocomposite Filament	Surface Resistivity (Ω/sq)	Surface Resistance (Ω)	Volume Resistivity ($\Omega\cdot\text{cm}$)
PA12 + 0 wt.%MWCNT	$1.29 \times 10^{12} \pm 1.11 \times 10^{12}$	$3.54 \times 10^{10} \pm 1.36 \times 10^9$	$7.83 \times 10^{11} \pm 8.89 \times 10^{11}$
PA12 + 2 wt.%MWCNT	$2.44 \times 10^9 \pm 2.23 \times 10^9$	$4.58 \times 10^7 \pm 4.17 \times 10^7$	$2.92 \times 10^9 \pm 4.70 \times 10^9$
PA12 + 3 wt.%MWCNT	$1.11 \times 10^4 \pm 4.30 \times 10^3$	$2.07 \times 10^2 \pm 8.01 \times 10^1$	$5.51 \times 10^4 \pm 1.89 \times 10^4$
PA12 + 4 wt.%MWCNT	$7.85 \times 10^3 \pm 1.88 \times 10^3$	$1.47 \times 10^2 \pm 3.51 \times 10^1$	$2.17 \times 10^3 \pm 6.29 \times 10^2$

Having evaluated the thermal, mechanical, and electrical performance of the developed nanocomposites, it is important to critically assess the overall viability of this recycling route. While repurposing aged PA12 SLS powder into FFF filament offers significant environmental and economic benefits, this manufacturing approach presents distinct advantages and disadvantages. A primary advantage is that the twin-screw extrusion process facilitates the highly uniform dispersion of MWCNT nanofillers into the polymer matrix, a critical requirement for consistent ESD performance [56]. Furthermore, it successfully transitions a waste material into a format compatible with widely accessible and cost-effective FFF systems [57].

However, this approach also introduces notable challenges. The transition requires an intermediate manufacturing step (filament extrusion) where maintaining strict dimensional tolerances (e.g., consistent filament diameter) is difficult when processing thermally degraded powders [24]. The intermediate melt-extrusion step subjects the already-aged PA12 to an additional thermal cycle that can compromise material properties [58]. Additionally, unlike the self-supporting and thermally controlled powder bed of the SLS process, FFF processing of PA12 requires careful thermal management to mitigate the inherent shrinkage, warpage, and bed-adhesion issues characteristic of semi-crystalline polymers [59].

4. Conclusions

This study demonstrated an innovative circular economy pathway by upcycling aged, residual polyamide 12 (PA12) powder from selective laser sintering (SLS) into functional nanocomposite filaments for fused filament fabrication (FFF). Incorporating multi-walled carbon nanotubes (MWCNTs) at 2, 3, and 4 wt.% served as an effective method to alter matrix properties while imparting target functional traits into the recycled feedstock. Thermally, the integrated MWCNTs exhibited a dual crystallization role, acting as heterogeneous nucleation sites that successfully shifted the crystallization temperature upward, while concurrently establishing a rigid physical network that confined polymer chain mobility and restricted the final degree of crystallinity. Mechanical characterization of the printed parts revealed contrasting performance profiles. Increasing the filler loading enhanced the overall stiffness and significantly reinforced the flexural performance, culminating in a progressive increase in both flexural strength and modulus up to 4 wt.%. Conversely, a trade-off was observed in tensile performance, where the addition of MWCNTs led to a gradual reduction in tensile strength. Additionally, crossing the electrical percolation threshold between 2 and 3 wt.% MWCNT successfully altered the electrical volume resistivity from an insulating state down to the electrostatic dissipative range. This structural-functional optimization confirms that waste SLS powder can be reliably repurposed into engineering feedstock, yielding additive manufacturing components perfectly tailored for demanding electrostatic-discharge (ESD) sensitive applications.

Future work should focus on the fabrication and validation of ESD-safe components using the developed materials, investigating the influence of printing parameters and build orientation, and evaluating the thermo-mechanical stability of these nanocomposites across multiple subsequent recycling cycles. Furthermore, while the baseline properties of FFF-printed virgin PA12 are well-documented in the literature, future studies comparing these established virgin benchmarks directly against the MWCNT-enhanced valorised filaments could provide further insights into the absolute mechanical trade-offs of this circular manufacturing approach.

Author Contributions: Conceptualization, C.S.S. and A.J.P.; methodology, C.S.S., A.C.L. and A.J.P.; software, C.S.S.; validation, C.S.S., A.C.L. and A.J.P.; formal analysis, C.S.S., A.C.L. and A.J.P.; investigation, C.S.S.; resources, C.S.S. and A.J.P.; data curation, C.S.S. and A.C.L.; writing—original draft preparation, C.S.S.; writing—review and editing, C.S.S., A.C.L., Á.M.S. and A.J.P.; visualization, C.S.S., A.C.L.; supervision, Á.M.S. and A.J.P.; project administration, A.J.P.; funding acquisition, A.J.P. All authors have read and agreed to the published version of the manuscript.

Funding: This work is carried out within the framework of the “Agendas para a Inovação Empresarial” (Project nº 49, acronym “INOV.AM”, with reference PRR/49/INOV.AM/EE, operation code 02/C05-i01.01/2022.PC644865234-00000004), supported by the RRP—Recovery and Resilience Plan and by the European Funds NextGeneration EU. <http://www.recuperarportugal.gov.pt/>. The publication of this article was financially supported by the Foundation for Science and Technology (FCT), multi-annual funding 2025–2029, UID/05256: Institute of Polymers and Composites (IPC/UM), of its R&D activities, through national funds (OE) and, when eligible, co-financed by community funds.

Data Availability Statement: Data is contained within the article.

Conflicts of Interest: The authors declare no conflicts of interest.

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