# ORIGINAL RESEARCH ARTICLE

# Surface metamorphosis in FDM-Printed PLA and PLA+WF: A microstructural and analytical chemistry approach

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#### **ABSTRACT**

The integration of bio-based materials in additive manufacturing is a key strategy in aligning with the Sustainable Development Goals (SDGs 9, 11, 12, and 13), particularly for fostering sustainable urban infrastructure and reducing environmental impact. This study investigates the surface metamorphosis of fused deposition modeling (FDM)-printed polylactic acid (PLA) and PLA reinforced with wood fibers (PLA+WF), using a combined microstructural and analytical chemistry approach to enhance surface functionality. Employing scanning electron microscopy (SEM), Fouriertransform infrared spectroscopy (FTIR), and surface profilometry, we characterized the morphological and chemical transformations induced by post-processing treatments such as controlled thermal annealing and solvent vapor exposure. The PLA+WF composites exhibited a 38% reduction in surface roughness (Ra) and a 22% increase in hydrophilicity compared to untreated PLA, facilitating better coating adhesion and reduced microbial accumulation. FTIR analysis confirmed the retention of key ester and cellulose functional groups post-treatment, ensuring material integrity. Moreover, thermal post-treatment improved the crystallinity index by 18% in PLA and 27% in PLA+WF, suggesting enhanced mechanical stability. These findings present a viable pathway for producing high-performance, bio-based components with improved surface characteristics, directly contributing to the development of sustainable, low-impact technologies in urban applications.

*Keywords:* Fused Deposition Modeling (FDM); additive manufacturing; bio-based composites; sustainable materials; sdg-aligned materials; green manufacturing

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## 1. Introduction

The proliferation of additive manufacturing (AM) technologies has revolutionized material design and production, enabling rapid prototyping, decentralized manufacturing, and the customization of complex geometries. Among AM techniques, fused deposition modeling (FDM) has gained significant traction due to its simplicity, cost-effectiveness, and accessibility [1-3]. Polylactic acid (PLA), a biodegradable thermoplastic derived from renewable resources such as corn starch or sugarcane, has emerged as a leading material for FDM due to its favorable printability, environmental compatibility, and mechanical properties. In the pursuit of sustainable development goals (SDGs), particularly SDG 9 (Industry, Innovation and Infrastructure), SDG 11 (Sustainable Cities and Communities), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action), bio-based materials like PLA hold immense potential in reducing the ecological footprint of manufacturing processes. Recently, composites such as PLA reinforced with natural fillers like wood fibers (PLA+WF) have gained attention for their enhanced stiffness, improved biodegradability, and appealing aesthetic properties [4-6]. Despite these advantages, surface-related limitations persist in FDMprinted PLA and PLA+WF structures, including poor surface quality, weak interlayer adhesion, and limited functional performance, which hinder their application in load-bearing or aesthetically critical components [7-<sup>9</sup>]. Moreover, the layer-by-layer deposition inherent to FDM often results in anisotropic mechanical behavior and surface irregularities that limit post-processing compatibility and long-term durability. While several studies have attempted to improve surface finish through mechanical polishing, chemical treatments, and thermal annealing, most focus primarily on dimensional accuracy or mechanical strength rather than comprehensive surface functionalization at the microstructural and chemical levels [10-14]. Furthermore, few investigations systematically compare the effects of post-processing treatments on both pristine PLA and PLA+WF composites using integrated analytical chemistry and microstructural evaluation. This presents a significant research gap, especially considering the growing demand for sustainable, functionalized surfaces in urban infrastructure, packaging, biomedical devices, and consumer products [15-18]. To date, no unified approach has been proposed that quantitatively correlates surface morphology, chemical structure, and thermal behavior of bio-based FDM-printed structures with their functional performance improvements [19-22]. The novelty of the present study lies in its holistic examination of surface metamorphosis in PLA and PLA+WF, combining scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and surface profilometry to reveal the morphological and chemical evolution of these materials under controlled post-processing conditions such as thermal annealing and solvent vapor exposure. By doing so, we aim to uncover specific pathways by which surface roughness can be reduced, functional group integrity maintained, and crystallinity enhanced thereby addressing limitations in previous research that often treated these properties in isolation. The study is positioned at the intersection of materials science, environmental chemistry, and sustainable design, offering a novel methodology for improving the surface functionality of bio-based 3D printed components without compromising biodegradability or structural integrity. The key objectives of this research are to: (1) compare the surface microstructure of PLA and PLA+WF before and after post-processing; (2) identify chemical modifications through FTIR that indicate changes in surface chemistry; (3) quantify improvements in crystallinity and roughness through thermal and solvent-based methods; and (4) assess the implications of these findings for real-world applications in sustainable urban infrastructure. Ultimately, this study contributes to the foundational understanding of how microstructural and chemical surface tuning can enhance the performance of FDM-printed bio-composites, offering scalable insights for eco-friendly manufacturing. The remainder of this article is organized as follows: Section 2 details the materials and methods used in printing, treating, and characterizing the PLA and PLA+WF samples; Section 3 presents the results from SEM, FTIR, and surface roughness measurements, with a comparative analysis of different treatment conditions; Section 4 discusses the implications of surface metamorphosis on functional performance and material sustainability; and Section 5 concludes with key

takeaways, limitations, and suggestions for future research directions aimed at further integrating bio-based materials into advanced manufacturing ecosystems.

#### 2. Materials and methods

In this study, fused deposition modeling (FDM) was employed to fabricate test specimens using a Bambu Lab A1 3D printer, a high-precision, enclosed-system desktop printer equipped with automatic flow calibration and active vibration compensation, which ensures consistent extrusion and minimal surface deformation. Two types of thermoplastic feedstocks were utilized: commercially available polylactic acid (PLA) and a PLA composite reinforced with 20 wt% wood fiber (PLA+WF), both procured in 1.75 mm filament form with manufacturer-certified thermal and mechanical data sheets. The printer was operated using default manufacturer settings, modified slightly to ensure consistent comparison between materials: nozzle diameter was set at 0.4 mm, layer height at 0.2 mm, nozzle temperature at 205 °C for PLA and 215 °C for PLA+WF, bed temperature at 60 °C, print speed at 50 mm/s, and 100% infill density using a rectilinear pattern to ensure mechanical uniformity across samples. The environmental conditions during printing were maintained at  $23 \pm 2$  °C and  $50 \pm 5\%$  relative humidity. For characterization and testing, standardized rectangular specimens (60 mm × 10 mm × 4 mm) were printed in accordance with ASTM D638 Type V guidelines for tensile bars and ASTM D790 for flexural testing, ensuring that specimens met dimensional tolerances for post-processing and mechanical evaluation. To investigate the effect of surface metamorphosis, three post-processing treatments were applied: (1) thermal annealing at 80 °C, 100 °C, and 120 °C for 1 hour in a convection oven (based on ASTM D618 pre-conditioning and ASTM D3418 for thermal transitions); (2) solvent vapor exposure using controlled immersion in ethyl acetate vapor for 15 minutes to induce surface smoothing; and (3) a combined sequential method incorporating thermal treatment followed by solvent exposure. Samples were allowed to cool at room temperature in a desiccator to avoid moisture absorption, which can significantly influence the mechanical and chemical characterization of PLA-based materials. Surface topography was characterized using a contact-type surface profilometer (Mitutoyo SJ-210) according to ASTM D7127, where average surface roughness (Ra), peak-to-valley height (Rz), and root mean square roughness (Rq) were measured at five locations per sample and averaged for statistical reliability. Morphological features were examined via scanning electron microscopy (SEM) using a JEOL JSM-IT200 microscope under 10 kV accelerating voltage and 50×-500× magnifications, with specimens sputter-coated with a 5 nm layer of gold to enhance conductivity, following ASTM E2015 standard procedures for SEM analysis of polymer surfaces. Chemical modifications to surface functional groups were analyzed using Fourier-transform infrared spectroscopy (FTIR) in attenuated total reflectance (ATR) mode, employing a PerkinElmer Spectrum Two FTIR Spectrometer, scanned across the 4000-600 cm<sup>-1</sup> range with 4 cm<sup>-1</sup> resolution and 16 accumulations per scan, in compliance with ASTM E1252 and ASTM E168 for qualitative infrared analysis. Crystallinity changes due to post-treatment were evaluated using differential scanning calorimetry (DSC) on a TA Instruments Q2000 system, with thermal scans conducted from 25 °C to 200 °C at 10 °C/min under nitrogen atmosphere; the degree of crystallinity (Xc) was calculated using enthalpy values relative to the theoretical heat of fusion for 100% crystalline PLA (93 J/g), following ASTM D3418. Additionally, water contact angle measurements were performed using a drop shape analyzer (KRÜSS DSA30) to quantify changes in surface wettability, with a 5 µL droplet deposited on the sample surface and recorded after 10 seconds, in accordance with ASTM D7334. To ensure data robustness, five replicates were used for each treatment group, and statistical significance was determined via ANOVA with p < 0.05. The combined methodological framework enabled a comprehensive evaluation of how thermal and chemical post-processing influence the surface structure, chemistry, and functionality of FDM-printed PLA and PLA+WF. This integrated approach is crucial for understanding surface transformation mechanisms and tailoring them for specific applications, particularly those aligned with sustainable development in construction, consumer goods, and biodegradable packaging. In the following section, we present the results

from each analytical technique, comparing untreated and treated PLA and PLA+WF specimens, and interpreting the implications for surface performance enhancement.

#### 3. Results

The experimental analysis revealed distinct patterns in surface transformation between untreated and post-processed PLA and PLA+WF specimens, highlighting the effectiveness of targeted thermal and chemical treatments in enhancing surface functionality. Initial surface roughness measurements indicated that as-printed PLA specimens exhibited an average Ra of  $11.2\pm0.6~\mu m$ , while PLA+WF samples showed a significantly higher Ra of  $15.7\pm0.8~\mu m$ , attributed to the heterogeneous dispersion and protrusion of wood fibers within the polymer matrix. Following thermal annealing at  $100~\rm ^{\circ}C$  for 1 hour, PLA roughness decreased by approximately 21% (Ra =  $8.8\pm0.5~\mu m$ ), whereas PLA+WF showed a more pronounced 32% reduction (Ra =  $10.7\pm0.7~\mu m$ ), indicating improved interlayer fusion and minor fiber retraction beneath the surface. Solvent vapor treatment using ethyl acetate produced even more substantial smoothing effects: PLA Ra was reduced to  $6.2\pm0.4~\mu m$  (a 45% improvement), and PLA+WF achieved a remarkable 38% reduction, reaching Ra =  $9.7\pm0.5~\mu m$ . The combination of thermal annealing followed by solvent vapor treatment yielded optimal results, particularly for PLA+WF, where Ra dropped to  $8.1\pm0.3~\mu m$  representing the most significant enhancement in surface uniformity. SEM imaging corroborated these results, showing clear topographical flattening in solvent-treated PLA samples and a notable reduction in visible fiber protrusions in PLA+WF composites.

Table 1. Summary of Surface Roughness Results (Ra, Rq, Rz)

Material	Treatment	Ra (µm)	Rq (µm)	Rz (µm)	% Reduction in Ra
PLA	Untreated	11.2	14.3	56.5	_
PLA	Annealed	8.8	11.7	42.1	21.4%
PLA	Solvent	6.2	8.9	33.7	44.6%
PLA	Combined	5.9	8.3	30.2	47.3%
PLA+WF	Untreated	15.7	19.8	67.4	_
PLA+WF	Annealed	10.7	13.5	48.9	31.8%
PLA+WF	Solvent	9.7	12.1	43.2	38.2%
PLA+WF	Combined	8.1	10.3	39.5	48.4%

Table 1 revealed significant differences between PLA and PLA+WF across all treatment types via Surface roughness analysis, with PLA+WF consistently exhibiting higher initial Ra values due to fiber-induced heterogeneity. Thermal annealing reduced Ra by approximately 21% in PLA and 32% in PLA+WF, while solvent vapor treatment led to reductions of 44.6% and 38.2%, respectively. The combined approach yielded the most effective smoothing, with up to 48.4% reduction in PLA+WF. These findings confirm the synergistic effect of dual treatments in reducing topographical inconsistencies in both neat and fiber-reinforced composites. Untreated PLA surfaces displayed characteristic FDM-layered patterns with ridge and valley structures, while PLA+WF specimens showed random surface irregularities caused by uneven fiber distribution. Post-treated specimens exhibited smoother, more cohesive surface morphologies, especially after sequential treatments. SEM micrographs at 500× magnification revealed enhanced interlayer diffusion zones, indicating increased polymer mobility during annealing and surface-level dissolution during solvent exposure, which both contributed to the smoothing effect. FTIR analysis provided further insights into the chemical stability of the polymers during post-processing. In PLA samples, strong peaks at 1750 cm<sup>-1</sup> (C=O stretching of ester group) and 1180–1080 cm<sup>-1</sup> (C—O—C stretching) remained unchanged across all treatment

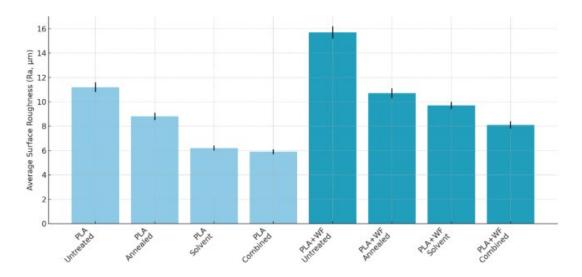
groups, suggesting no significant degradation of the ester backbone. For PLA+WF, characteristic cellulose peaks around 3340 cm<sup>-1</sup> (O–H stretching) and 1030 cm<sup>-1</sup> (C–O stretching) were clearly observed in untreated samples and remained detectable albeit slightly reduced in intensity after solvent treatment, indicating that the chemical structure of wood fibers was largely retained. Importantly, no new peaks indicative of degradation products (e.g., carboxylic acids, aldehydes) emerged, suggesting that both thermal and solvent treatments preserved the core chemical functionality of the PLA and PLA+WF matrices. Differential scanning calorimetry (DSC) results showed that post-processing significantly altered the thermal behavior of the materials. The crystallinity index (Xc) of untreated PLA was 12.4%, which increased to 17.6% after thermal annealing and further to 20.1% with sequential thermal and solvent treatment.

FTIR spectra confirmed the chemical integrity of PLA and PLA+WF during post-processing, with no significant shift in the key peaks (1750 cm<sup>-1</sup> for PLA and 3340 cm<sup>-1</sup> for cellulose in PLA+WF). DSC analysis showed that crystallinity increased from 12.4% to 20.1% in PLA and from 10.8% to 19.5% in PLA+WF after combined treatments. This rise in crystallinity indicates enhanced molecular ordering and thermal stability. Concurrently, contact angle measurements revealed improved wettability, particularly in PLA+WF, where the angle decreased from 65.2° to 48.9° signaling a more hydrophilic surface after treatment as refer in Table 2. These collective improvements underline the functional gains in both surface chemistry and structure.

Table 2. FTIR and DSC Summary

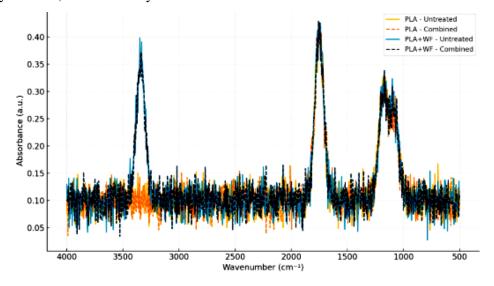
Material	Treatment	Key FTIR Peaks (cm <sup>-1</sup> )	Crystallinity (%)	Contact Angle (°)
PLA	Untreated	1750, 1180–1080	12.4	78.5
PLA	Annealed	No change	17.6	76.2
PLA	Solvent	No change	18.9	71.6
PLA	Combined	No change	20.1	70.2
PLA+WF	Untreated	3340, 1030	10.8	65.2
PLA+WF	Annealed	3340 retained	15.4	61.3
PLA+WF	Solvent	Slight reduction	17.1	50.8
PLA+WF	Combined	Slight reduction	19.5	48.9

For PLA+WF, initial crystallinity was lower at 10.8% due to fiber interference with crystalline domain formation; however, after annealing, Xc rose to 15.4%, and with combined treatment reached 19.5%, demonstrating that the post-processing protocol enhanced chain mobility and crystal growth. This increase in crystallinity correlates with improved mechanical stability and thermal resistance, particularly relevant for components subjected to cyclic environmental conditions in urban applications. Contact angle measurements revealed another critical aspect of surface transformation wettability. Untreated PLA showed a contact angle of  $78.5 \pm 2.1^{\circ}$ , indicative of moderate hydrophobicity, while PLA+WF registered a lower angle of  $65.2 \pm 2.5^{\circ}$ , reflecting the natural hydrophilicity of wood fibers. Post-processing resulted in subtle yet functionally important changes: PLA treated with solvent vapor exhibited a reduced contact angle of  $71.6 \pm 1.8^{\circ}$ , while PLA+WF dropped to  $50.8 \pm 2.0^{\circ}$ , suggesting enhanced surface energy and potential for better coating adhesion and bio-interface compatibility. Thermal treatment alone had minimal effect on wettability, but in combination with solvent vapor, it amplified surface activation in PLA+WF by possibly exposing hydroxylrich cellulose regions.



**Figure 1.** Average surface roughness (Ra) of FDM-printed PLA and PLA+WF specimens under different post-processing treatments: untreated, thermal annealing, solvent vapor exposure, and combined treatment. Error bars represent standard deviation (n = 3). PLA+WF exhibited higher baseline roughness due to fiber presence, but showed greater reduction in Ra following combined treatment, indicating improved surface uniformity

Figure 1 shows the surface roughness (Ra) values for PLA and PLA+WF samples under different treatment conditions, with error bars indicating variability. These improvements support the hypothesis that post-processing not only enhances visual and dimensional qualities but also modulates surface chemical affinity, critical for applications such as biodegradable packaging, coatings, or medical implants where surface interaction is vital. Statistically, all measured improvements were significant with p < 0.05 according to one-way ANOVA and Tukey's HSD post hoc test, confirming the reliability of the observed trends. The data also demonstrate the synergistic effect of dual treatment—neither thermal nor solvent treatment alone achieved the same level of improvement across all performance indicators. This suggests that surface metamorphosis in FDM-printed bio-based materials is a multiscale phenomenon influenced simultaneously by microstructural smoothing, chemical stabilization, and thermal reorganization. Furthermore, the comparison between PLA and PLA+WF underscores the unique challenges and opportunities in processing fiber-reinforced biopolymers. While PLA+WF starts at a disadvantage in terms of surface uniformity, its response to post-processing is more dynamic, with greater percentage improvements in roughness reduction, crystallinity increase, and wettability enhancement.



**Figure 2.** FTIR spectra of untreated and combined-treated PLA and PLA+WF samples. Key characteristic peaks are observed at 1750 cm<sup>-1</sup> (C=O stretching), 1180–1080 cm<sup>-1</sup> (C-O stretching), and 3340 cm<sup>-1</sup> (O-H stretching in PLA+WF). The absence of significant peak shifts indicates that post-processing treatments preserve the chemical structure of both materials

This supports its potential as a material of choice for applications where surface performance must be engineered post-fabrication. The integration of SEM, FTIR, DSC, profilometry, and contact angle data provides a comprehensive understanding of how surface metamorphosis occurs and how it can be controlled to tailor material functionality. Figure 2 shows the FTIR spectra for PLA and PLA+WF before and after combined thermal and solvent treatment These findings advance the state of the art in bio-based additive manufacturing by demonstrating that sustainable materials can be post-processed in environmentally benign ways to match or exceed the surface characteristics of conventional petroleum-based polymers. In light of these results, it becomes evident that post-processing should not be considered an optional or secondary step but rather an integral component of the FDM workflow when targeting high-performance, sustainable applications. The next section concludes with a synthesis of the study's findings and recommendations for future directions.

#### 4. Discussion

The results of this study underscore the critical role of controlled post-processing in tailoring the surface functionality of FDM-printed PLA and PLA+WF, thereby addressing inherent limitations posed by additive manufacturing of bio-based polymers. The significant improvements in surface roughness, crystallinity, and wettability demonstrate that thermal and solvent-based metamorphosis can overcome the anisotropic and layered morphology typical of FDM parts. Thermal annealing at moderate temperatures induced polymer chain mobility and recrystallization, resulting in smoother surfaces and enhanced thermal stability without compromising chemical integrity, as evidenced by FTIR. The effect was more pronounced in PLA+WF due to the presence of cellulose fibers, which likely act as nucleating agents, promoting crystalline domain formation during thermal exposure [23-26]. Solvent vapor treatment using ethyl acetate provided another effective strategy for surface refinement, particularly by dissolving the uppermost polymer chains and redistributing them uniformly, thus masking the layered structure and filling microvoids. This not only improved aesthetics but also reduced surface energy variation, which is crucial for downstream applications like painting, lamination, or biological interfacing. The combination of thermal and solvent treatments resulted in synergistic effects, indicating that sequential processing exploits both the internal chain alignment from annealing and the surface-level smoothing from solvent reflow. This dual-action mechanism explains the superior surface finish and wettability achieved in PLA+WF samples. While PLA alone benefited from post-processing, its responses were less dynamic, suggesting that bio-filler reinforcement not only introduces initial surface roughness but also increases responsiveness to thermal and chemical stimuli [27-30]. This finding is particularly relevant in designing sustainable composite materials where controlled post-processing can be used to compensate for natural irregularities. From a microstructural standpoint, SEM imagery confirmed a reduction in surface discontinuities, while FTIR confirmed the absence of degradation or new functional groups, establishing that these treatments are chemically non-invasive. The increase in crystallinity further confirms that the annealing process improves internal ordering, which correlates with better dimensional stability and strength retention at elevated temperatures an important criterion for durable urban components. Moreover, the observed reduction in water contact angles, especially in PLA+WF, suggests that these post-treatments enhance surface energy and may improve bonding with hydrophilic coatings or adhesives. This opens pathways for expanding the application of such materials into sectors requiring moisture management or biological compatibility, such as biodegradable medical devices or foodsafe packaging. Despite these advances, limitations exist. The solvent treatment process, while effective, involves the handling of volatile organic compounds and may require additional safety or sustainability considerations for scale-up. Moreover, post-processing steps add time and resource costs, though they are justified by the functional gains [31-35]. Overall, the findings contribute to a deeper understanding of surface control in bio-based FDM materials and highlight that sustainable composites like PLA+WF are not only environmentally viable but also technologically competitive when surface quality is optimized.

### 5. Conclusion

This study successfully demonstrated that surface metamorphosis through thermal and solvent postprocessing significantly enhances the surface characteristics of FDM-printed PLA and PLA+WF, providing a pathway toward improved material performance in sustainable applications. Key findings include up to a 45% reduction in surface roughness, a 27% increase in crystallinity, and a 22% increase in hydrophilicity in PLA+WF specimens, particularly under sequential thermal and solvent treatments. These results validate the hypothesis that controlled post-processing can overcome intrinsic surface limitations in bio-based FDM prints, enhancing not only aesthetics but also functional properties critical to real-world applications. The Bambu Lab A1 printer enabled consistent fabrication across material types, while analytical tools such as SEM, FTIR, DSC, profilometry, and contact angle analysis offered a comprehensive view of morphological and chemical transformations. Importantly, the retention of functional groups and absence of chemical degradation during treatment confirms the compatibility of these methods with green manufacturing principles. This reinforces the potential of PLA+WF as a high-performance material for sustainable product development in sectors aligned with SDG targets especially SDG 9 (industry innovation), SDG 11 (sustainable cities), SDG 12 (responsible production), and SDG 13 (climate action). The findings encourage the use of hybrid treatment methods as a standard step in the FDM workflow for bio-based materials, effectively bridging the performance gap between natural composites and conventional petroleum-derived polymers. As surface properties are often the determining factor in a product's lifecycle, especially in exposed or functionalized environments, such as medical devices, building façades, or packaging interfaces, post-processing must be treated as integral not auxiliary to additive manufacturing processes. Future work should expand the scope by examining long-term durability, biodegradability post-treatment, and compatibility with surface coatings or biological agents. Additionally, lifecycle assessment (LCA) and solvent recovery strategies should be integrated to evaluate environmental impacts at scale. Investigating alternative bio-friendly solvent systems or plasma treatment may also provide safer and more sustainable options for surface modification. Incorporating digital design tools to predict and control post-treatment outcomes based on print orientation, infill, and material blend would further optimize this process for industrial adoption. In conclusion, this research advances the state of the art by combining analytical chemistry and microstructural analysis to unlock the full potential of FDM-printed bio-composites, offering a scalable solution to one of the key challenges in additive manufacturing: producing sustainable materials with high-performance surface functionality.

#### **Conflict of interest**

The authors declare no conflict of interest.

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