Thermomechanical properties of coated PLA-3D-printed orthopedic plate with PCL/Akermanite nano-fibers: Experimental procedure and AI optimization

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Date: 2024-04-02T00:00:00+00:00

Abstract

In this study, first, an orthopedic plate was 3D printed with Polylactic acid (PLA) and coated with polycaprolactone (PCL)/Akermanite (AKT) nano-fibers. The composition included 8 wt.% of PCL and 3 wt.% of nAKT, while diameter of the PCL/AKT nano-fibers was approximately 253 nm ± 33 nm. Thermomechanical properties such as pressure, three-point bending flexural, and thermal conductivity of coated and non-coated specimens were examined and compared. In the next step, the bioactivity of the coated samples was evaluated following a 28-day immersion in simulated body fluid (SBF). Further, scanning electron microscope (SEM) images were taken to assess morphology of nanofibers and apatite formation on samples. By adding PCL to PLA, the maximum pressure force is enhanced by 16.83%. Further by adding nAKT to PLA+PCL sample, the maximum pressure force is enhanced by 4.72%. Further, by adding PCL to PLA, the maximum bending flexural force is enhanced by 21.06%. Further by adding nAKT to PLA+PCL sample, the maximum bending flexural force is enhanced by 21.39%. The results of this study are used to improve modeling of the orthopedic plates.

Full Text

Preamble

Thermomechanical Properties of PCL/Akermanite Nanofiber-Coated 3D-Printed PLA Orthopedic Plates: Experimental Procedure and AI Optimization

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Abstract

Three-dimensional printing has become increasingly popular among surgeons for orthopedic applications due to its ability to fabricate patient-specific implants. In this additive manufacturing process, polymeric biomaterials are deposited layer-by-layer to create orthopedic plates and implants. However, 3D-printed devices often lack the necessary surface properties for effective bonding with host tissue. Coating these plates with nanofibers offers a promising solution to overcome this limitation. In this study, orthopedic plates were first 3D-printed using polylactic acid (PLA) and subsequently coated with polycaprolactone (PCL)/akermanite (AKT) nanofibers. The coating composition consisted of 8 wt.% PCL and 3 wt.% nAKT, yielding nanofibers with an average diameter of 253 nm ± 33 nm. The thermomechanical properties—including compressive strength, three-point bending flexural strength, and thermal conductivity—of both coated and uncoated specimens were examined and compared. Bioactivity was evaluated following 28 days of immersion in simulated body fluid (SBF), while scanning electron microscopy (SEM) was used to assess nanofiber morphology and apatite formation. The results demonstrated that adding PCL to PLA enhanced the maximum compressive force by 16.83%, with a further 4.72% increase upon nAKT addition. Similarly, the maximum bending flexural force increased by 21.06% with PCL addition and an additional 21.39% with nAKT incorporation. These findings contribute to improved modeling and design of

orthopedic plates.

Keywords: Thermomechanical properties; 3D printing; Nanofibers; Orthopedic plate; Bioactivity evaluation; Optimization

1. Introduction

Orthopedic plates have revolutionized fracture stabilization and deformity correction by providing effective mechanical support that facilitates natural bone healing and restores skeletal integrity [1-3]. However, conventional manufacturing techniques such as casting and machining struggle to produce plates with complex geometries required for patient-specific applications [4]. This limitation necessitates alternative fabrication methods capable of creating intricate designs [5, 6].

Three-dimensional printing, or additive manufacturing, has emerged as a transformative technology for medical implants [7, 8]. The layer-by-layer deposition process enables precise control over geometry and internal architecture, allowing fabrication of patient-specific orthopedic plates tailored to individual anatomies [9]. This flexibility optimizes mechanical performance while accommodating diverse biomaterials including biopolymers, bioceramics, and bio-based metals [10-13]. Schulze et al. [14] utilized selective laser melting to fabricate Ti-6Al-4V orthopedic implants, demonstrating improved mechanical properties suitable for clinical applications. Despite these advantages, 3D-printed implants often exhibit suboptimal biological and mechanical surface properties, requiring additional processing techniques to address these challenges.

Coating technologies play a crucial role in enhancing implant surface properties by improving biocompatibility, osseointegration, mechanical strength, corrosion resistance, and drug delivery capabilities [15-22]. Various methods including dip coating, chemical vapor deposition, and electrospinning have been employed to deposit functional coatings [23-27]. When combined with 3D-printed orthopedic plates, these techniques enable fabrication of multifunctional implants with superior performance. Robertson et al. [28] demonstrated that titania nanotube interfaces enhance hydroxyapatite coating adhesion on metallic implants, showing improved coating strength and bioactive dopant incorporation.

Polymeric nanofibers have attracted significant attention due to their high surface-area-to-volume ratio, manufacturability, and potential for controlled drug release [29-34]. With diameters typically ranging from tens to hundreds of nanometers, these fibers can mimic the native extracellular matrix to promote cell adhesion and proliferation. In orthopedic applications, nanofiber coatings improve surface characteristics and enhance tissue integration. Saniei et al. [35] combined 3D printing with electrospinning to create multifunctional implants, using polyvinyl alcohol/hydroxyapatite nanofibers on PLA surfaces to improve biocompatibility and bioactivity.

The selection of polymeric biomaterials critically influences coating performance, with biocompatibility, biodegradability, mechanical strength, and processability being key considerations. Polycaprolactone (PCL) has gained prominence in orthopedics due to its biocompatibility and tunable degradation rates [36-38]. As a coating material, PCL provides a bioactive interface for cell interaction and tissue regeneration, enhancing long-term implant stability [39, 40]. Its slow degradation profile makes PCL particularly suitable for sustained drug release and tissue engineering applications [41, 42]. Radhakrishnan et al. [43] investigated PCL/AgNP scaffolds, reporting improved stiffness, cytocompatibility, and antibacterial properties, while Razmjooee et al. [44] enhanced PCL nanofiber anti-thrombogenicity through surface modification.

Bio-based nanoceramics such as hydroxyapatite and akermanite have emerged as promising coating materials due to their nanoscale dimensions and unique biological properties [45]. Akermanite ($Ca_2MgSi_2O_7$) has been widely utilized in biomedical applications because of its biocompatibility, bioactivity, and similarity to natural bone minerals, providing an ideal environment for cell attachment and bone integration. Incorporating akermanite into nanofiber coatings enhances osteoconductivity and biomineralization. Zare-Harofteh et al. [46] used akermanite nanoparticles in gelatin scaffolds, demonstrating bioactivity and favorable mechanical properties, while Dong et al. [47] showed that akermanite-magnetic nanoparticle composite coatings improved mechanical properties and enabled controlled drug release. Karamian et al. [48] similarly demonstrated enhanced bioactivity in hydroxyapatite-baghdadite scaffolds coated with PCL/bioglass.

Artificial intelligence has become an invaluable tool in orthopedic implant design, enabling rapid exploration of coating parameters to identify optimal configurations for enhanced thermomechanical performance [49, 50]. AI-driven predictive modeling also facilitates estimation of long-term implant behavior under various physiological conditions. Nasiri et al. [51] demonstrated that data-driven approaches can predict mechanical properties of metallic, composite, and 3D-printed implants, proving extremely useful for design optimization and cost reduction.

This study investigates the thermomechanical properties of PCL/AKT nanofiber-coated 3D-printed PLA orthopedic plates through experimental procedures. Bioactivity evaluation in SBF and SEM analysis of nanofiber morphology and apatite formation are performed. Additionally, AI analysis demonstrates how advanced technologies can enhance medical device development. The results provide valuable insights into the mechanical and biological performance of coated orthopedic plates and contribute to improved modeling approaches.

2. Materials and Methods

2.1. Materials

Polycaprolactone (PCL, Mw: 80,000 g/mol), dichloromethane (CH_2Cl_2), fetal bovine serum (FBS), and reagents for nano-akermanite synthesis were purchased from Sigma-Aldrich. Deionized water was used for all solution preparations. Commercial PLA filament (1.75 mm diameter) was obtained from Esun Company.

2.2. 3D Printing of Orthopedic Plate

The plate design was converted to an STL file using Ultimaker Cura software with a layer height of 200 m and 0-90 infill pattern. The file was transferred to a customized 3D printer equipped with a 200 m extrusion nozzle. Printing was performed at a nozzle temperature of 200°C and a speed of 20 mm/sec.

2.3. Synthesis of Nano-Akermanite Powder

Akermanite (AKT, $Ca_2MgSi_2O_7$) powders were synthesized via a sol-gel method [52]. Tetraethyl orthosilicate ((TEOS; $C_2H_5O)_4Si$), magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$), calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$), and nitric acid (HNO_3) as precipitant were stirred for 4 hours at 25°C. The solution was then heated at 70°C for 24 hours and dried at 130°C for 48 hours. The resulting AKT powder was milled to produce nano-sized AKT (nAKT) and sintered at 1400°C for 3 hours.

2.4. Electrospinning of PCL Nanofibers

PCL was dissolved in dichloromethane and stirred magnetically for 4 hours to prepare solutions of 4.0, 6.0, and 8 wt.%. One milliliter of each solution was loaded into a standard syringe. Electrospinning was performed using a high-voltage power supply, metal collector, and syringe infusion pump.

2.5. Electrospinning of PCL-nAKT Nanofibers

The 8 wt.% PCL solution was selected for its uniform, bead-free nanofiber morphology. Three weight percent of nAKT powder was added to the PCL solution and sonicated for 1 hour to prepare homogeneous suspensions. Circular orthopedic plate samples (10 mm diameter, 3 mm thickness) were 3D-printed and positioned on the collector. The PCL-nAKT suspensions were directly electrospun onto the sample surfaces using the following parameters: injection rate of 0.2 mL/h, needle-to-collector distance of 100 mm, and applied voltage of 12 kV.

2.6. Bioactivity Evaluation

Bioactivity was assessed according to ISO 23317. Coated samples were immersed in polyethylene containers filled with simulated body fluid (SBF) and incubated at 37°C for 28 days. The SBF volume in each container was calculated using Equation (1). After incubation, specimens were removed, washed with deionized water, and dried at 25°C.

2.7. Characterization Methods

Field emission scanning electron microscopy (FESEM-NOVA, 10 kV) was used to observe electrospun PCL and PCL-nAKT nanofiber morphologies. Fiber dimensions were characterized using ImageJ software. All experiments were performed in triplicate. X-ray diffraction (XRD-D8 Bruker) was conducted to verify nAKT formation during synthesis.

2.8. Thermal Conductivity

Thermal conductivity of coated and uncoated 3D-printed samples was measured using a KD2 Pro device with a stainless steel KS1 sensor at temperatures of 30, 35, 40, and 45°C. Each test was repeated on four samples.

3. Results and Discussion

3.1. Characterization of 3D-Printed Plate and Electrospun PCL Nanofibers

The orthopedic plate was successfully 3D-printed with dimensions matching the design specifications and a smooth, uniform surface [Figure 1: see original paper]. Three PCL concentrations were evaluated for electrospinning. As the polymer concentration increased from 4.0 to 8.0 wt.%, uniform, bead-free nanofibers with a mean diameter of 296 nm ± 48 nm were obtained. Consequently, the 8 wt.% PCL solution was selected for subsequent experiments.

The morphology of electrospun PCL nanofibers from the 8.0 wt.% solution is shown in [Figure 2: see original paper]. Bead formation during electrospinning is influenced by three parameters: net charge density, solution surface tension, and viscosity [53]. Eliminating beads requires increasing solution viscosity and net charge while decreasing surface tension [53]. Higher polymer concentrations enable the charged electrospinning jet to withstand Coulombic stretching forces, resulting in smooth, thick fibers [54].

3.2. Characterization of Electrospun PCL-nAKT Nanofibers and Coated Plate

[Figure 3: see original paper] illustrates the morphology of PCL-nAKT electrospun nanofibers containing 8 wt.% PCL and 3 wt.% nAKT. SEM images reveal uniform, bead-free nanofibers with nAKT particles uniformly dispersed within the PCL matrix. Incorporating ionic materials into polymer solutions typically reduces nanofiber diameter [55-58]. ImageJ analysis confirmed that nAKT addition decreased the average fiber diameter from 296 nm ± 48 nm to 253 nm

± 33 nm. Energy-dispersive X-ray spectroscopy (EDX) verified nAKT presence within the nanofiber matrix.

XRD analysis confirmed nAKT formation through the synthesis process. [Figure 4a: see original paper] shows sharp characteristic peaks at 2 = 28.904°, 31.169°, and 33.434° with d-spacings of 3.0865 Å, 2.8672 Å, and 2.6781 Å, respectively, along with lower-intensity peaks. [Figure 4b: see original paper] verifies the PCL-nAKT composition through a characteristic peak at 2 = 19.619° with a d-spacing of 4.5213 Å.

3.3. In-Vitro Bioactivity Evaluation

Implant bioactivity—the ability to bond with host tissue—can be evaluated in vitro by assessing apatite formation on implant surfaces after SBF immersion [59]. This study utilized nAKT particles to accelerate apatite layer formation and enhance orthopedic plate bioactivity.

SEM images of coated and uncoated samples after 28 days in SBF are presented in [Figure 5: see original paper]. Spherical apatite crystals formed on all specimen surfaces during incubation, confirming apatite layer formation and in vitro bioactivity. [Figure 5a: see original paper] shows the surface of an uncoated PLA specimen, where the apatite layer verified bioactivity but failed to form a porous extracellular matrix (ECM). Surface nano-structuring is crucial for implant functionality [60].

[Figure 5b: see original paper] displays coated PLA specimens with PCL-nAKT nanofibers after SBF immersion. After 28 days, apatite crystals nucleated and grew within the PCL nanofibers, with AKT nanoparticles acting as nucleation sites that accelerated apatite formation. Mineralization of PCL-nAKT nanofibers increased surface roughness, which benefits cell attachment and proliferation. Furthermore, nAKT precipitation and apatite formation within the nanofibers created a porous surface structure on the PLA orthopedic plate, enhancing water and nutrient transport for improved cell growth.

3.4. Compressive Strength

[Figure 6: see original paper] presents compressive force-displacement curves for uncoated and coated samples. Coating significantly improved pressure resistance. The maximum compressive force was 7028.86 N (at 10 mm displacement) for PLA alone, 8211.78 N for PLA+PCL, and 8599.08 N for PLA+PCL+nAKT. This represents a 16.83% improvement with PCL addition and a further 4.72% enhancement with nAKT incorporation.

3.5. Three-Point Bending Flexural Strength

[Figure 7: see original paper] shows three-point bending flexural results for all sample types. Coating improved bending properties substantially. The maximum bending flexural force was 70.99 N (at 2 mm displacement) for PLA, 85.94 N for PLA+PCL, and 104.33 N for PLA+PCL+nAKT. PCL addition enhanced the maximum bending flexural force by 21.06%, with nAKT providing an additional 21.39% improvement.

3.6. Thermal Conductivity

[Figure 8: see original paper] displays thermal conductivity measurements of 3D-printed samples with and without coating in SBF solution. Increasing nAKT volume fraction decreased thermal conductivity due to the insulating effect of nAKT particles. Conversely, elevated temperature increased thermal conductivity by enhancing particle movement and inter-particle interactions.

3.7. Optimization

[Figure 9: see original paper] presents 3D heat transfer data for coated and uncoated samples in SBF solution. A curve was fitted to the experimental data using the Levenberg-Marquardt algorithm. The optimization model is expressed in Equation (2). The fitted paraboloid model achieved a reduced chi-square of 2.01119E-6, R-square (COD) of 0.99774, and adjusted R-square of 0.99692, where S represents the sample and T represents temperature (30, 35, 40, and 45°C).

Using this correlation, optimized curves were generated and compared with empirical data [Figure 10: see original paper]. The primary limitation in training accuracy was the limited number of experimental test results; additional data would improve model precision.

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4. Conclusion

This study investigated the thermomechanical properties of PCL/AKT nanofiber-coated 3D-printed PLA orthopedic plates through experimental procedures. Key findings include:

- XRD and SEM analyses verified nanofiber composition and confirmed homogeneous particle distribution within the nanofiber matrix.
- In vitro bioactivity evaluation demonstrated spherical apatite crystal formation on all specimen surfaces after SBF immersion, confirming bioactivity. The coated samples exhibited enhanced apatite nucleation and growth within the PCL-nAKT nanofibers.
- Mechanical testing revealed significant improvements in both compressive and flexural properties. Adding PCL to PLA enhanced maximum compressive force by 16.83% and maximum bending flexural force by 21.06%. Further nAKT addition to PLA+PCL provided additional improvements of 4.72% in compressive strength and 21.39% in flexural strength.

- Thermal conductivity decreased with increasing nAKT volume fraction due to particle insulation effects, while temperature elevation increased conductivity through enhanced particle movement and interactions.
- Machine learning algorithms and curve fitting methods optimized the experimental data, yielding a calculated equation with $R^2 = 0.99774$, indicating excellent fit to the 3D data.

Future research should include animal studies to further validate the bioactivity, biocompatibility, and biodegradability of PCL/akermanite nanofiber-coated PLA 3D-printed orthopedic plates, which represents a current limitation of this study.

Acknowledgements

This research received funding from the Norwegian Financial Mechanism 2014-2021 under Project Contract No. 2020/37/K/ST8/02748.

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Note: Figure translations are in progress. See original paper for figures.

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