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RESEARCH ARTICLE

EFFECT OF HYDROXYAPATITE FILLER SIZE ON HIGH STRESS FATIGUE LIFE OF SELF-REINFORCED POLYLACTIC ACID COMPOSITES

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Abstract. In this study, the fatigue behaviour of self-reinforced polylactic acid (sr-PLA) biomaterials at high stress levels was investigated, and the influence of different filler sizes (2.99 µm and 20 nm) was evaluated using Weibull distribution analysis to assess their potential for bone fixation applications. The composite was produced by drawing PLA fibres in a PLA matrix containing hydroxyapatite (HA) particles of micrometre and nanometre-sized particles to produce HA/PLA/PLA prepreg. The pre-impregnated sheets were then compression moulded and tested under quasi-static bending and flexural fatigue at an 80 % stress level, 2 Hz until failure. The addition of the filler enhances the bending properties of the composite. At the same time, the bending strength and modulus increase with a decrease in the filler size. Under the specific cyclic loading conditions tested, the initial data indicate that the Weibull median fatigue life of sr-PLA reduces with the presence of the fillers. However, the fatigue resistance of the HA-filled composite improved with a smaller filler size. The Weibull median fatigue life of HA/sr-PLA increases from 104,514 cycles to 254,884 cycles for µm-HA/PLA/PLA and nm-HA/PLA/PLA composites, respectively. The Weibull statistical model indicates that the scale parameters improve by 140 % when smaller filler particles (nm-HA) are used. During fatigue testing, the modulus of the composite decreased due to material damage. SEM analysis indicated that failure at the HA/matrix interface was a major contributing factor. Furthermore, fracture behaviour suggests that the materials are more susceptible to tensile stress, which may be attributed to limited bonding at the HA/matrix interface.

Keywords: Fatigue testing, polylactic acid, hydroxyapatite, filler size, Weibull distribution, bone fixation.

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1. INTRODUCTION

The treatment of bone fractures, especially hand fractures, has shifted from conservative methods using cast and splint immobilisation to fixation via surgical intervention [1]. This new method improved clinical outcomes by providing stable fixation, enabling early mobilisation, and minimising soft tissue damage. Metallic implants such as Kirschner wires, plates, screws, intraosseous wires or intramedullary devices were used by surgeons to fix the unstable hand fractures. However, once bone consolidation is achieved, these osteosynthesis devices are no longer needed and indeed can be harmful. They can irritate soft tissues, disrupt tendon gliding and impair joint movement. Metallic implants such as Kirschner wires, screws or plates may lead to tendon rupture, implant migration and patient discomfort [2]. Thus, it underscores the need for safer alternatives, especially in anatomically delicate regions like the hand. As a result, bioabsorbable osteofixation devices are increasingly favoured for trauma, orthopaedic and craniomaxillofacial surgery. In the hand, bioabsorbable implants are advantageous as the biomechanical stresses are relatively low, and they eliminate the need for secondary surgical removal, which carries the risk of damaging delicate structures in the hand [3].

The most intensively studied group of bioabsorbable polymers for osteo-fixation devices is poly-α-hydroxy acids, especially polyglycolide (PGA) and polylactide (PLA). Lactic acid exists in two stereoisomeric forms, L- and D-lactic acid. The L-isomeric form of polylactide (PLLA) is relatively hydrophobic and semi-crystalline; thus, it will take several years for complete biodegradation and bioabsorption [4]. During the degradation process, the polymer produces lactic acid as a by-product, which can cause fluid accumulation and inflammation at the implant site.

To overcome these limitations, the incorporation of bioactive fillers into bioabsorbable polymers has garnered tremendous interest [5]. Beyond biomedical applications, recent advancements in sustainable polymer composites demonstrate that the inclusion of inorganic nanofillers significantly influences the stiffness, thermal behaviour, and morphology of the polymer matrices, primarily through optimised filler-matrix interface interactions [6]. In bioabsorbable orthopaedic implants, bioactive calcium phosphate such as hydroxyapatite (HA) or tricalcium phosphate (TCP) is used to enhance the mechanical properties of the bioabsorbable polymers while promoting bone bonding and accelerating fracture healing [7-9]. The particle size of the HA filler plays a critical role in these interactions. Smaller particles typically yield greater adhesion strength, thereby improving the overall mechanical performance of the composite [10, 11]. Furthermore, most potential clinical applications subject implants to significant cyclic loading. For instance, a patient's hand undergoes approximately 12,600 cycles of rehabilitation exercises during the initial six weeks of the healing process [12]. Moreover, cyclic overloading contributes to approximately 20 % of the failure force in osteosynthesis devices, highlighting the critical importance of fatigue resistance [13]. Recent studies on the cyclic fatigue of biodegradable polymers [14] and specifically HA/PLA systems [15] have highlighted that while filler incorporation can modulate static mechanical properties, it often introduces complex, premature failure mechanisms under dynamic stress.

While the quasi-static mechanical properties and biological advantages of HA-filled PLA composites are well-documented, a crucial research gap remains in their dynamic fatigue behaviour under high stress amplitudes. Self-reinforced PLA (sr-PLA) possesses a distinct fibre-matrix architecture that provides superior baseline strength; however, the mechanism by which different filler sizes (micro versus nanoscale) disrupt or enhance interfacial bonding under cyclic stress in this specific self-reinforced system has not yet been defined. In dynamic cyclic loading, the filler-matrix interface often acts as the primary site for crack initiation. Micro-scale particles can create significant localised stress concentration and larger voids upon debonding, acting as critical flaws that accelerate fatigue failure. Conversely, nanoscale fillers are hypothesised to improve fatigue resistance not only by offering a higher surface-to-volume ratio for load transfer, but also by significantly reducing interparticle spacing. This denser packing at the nano-scale is thought to interfere with stress fields and effectively arrest or deflect propagating micro-cracks during continuous cyclic loading.

Therefore, this study aims to address this gap by investigating the initial flexural properties and high-stress-amplitude fatigue behaviour of self-reinforced polylactide composites containing HA. By evaluating the influence of filler size on fatigue life and utilising Weibull analysis to predict survival probability, this study shall contribute to a better understanding of filler-matrix failure behaviour under cyclic conditions, which is essential for designing reliable bioabsorbable implants for bone fixation.

2. MATERIALS AND METHODS

2.1 Materials

Polylactide with L:D, L ratio of 70/30 (PLA70), that is 70 % PLLA and 30 % a racemic mixture of PLLA and PDLA (Boehringer Ingelheim, Ingelheim am Rhine, Germany) and intrinsic viscosity of 6.1 dlg^{-1} was used as matrix. Polylactic with an L:D ratio of 96:4 (PLA96, Purac Biochem b.v., The Netherlands) in the form of fibres had a final diameter of 60-90 µm. Two different particle sizes of HA were used as the filler: d_{50} = 2.99 µm (Plasma Biotal Ltd, Tideswell, UK) and 20 nm (manufactured in-house).

2.2 Composite Production

PLA70 was dissolved in acetone using the solvent casting method. Initially, HA particles were added to the matrix solution at a concentration of 40 wt.%. To ensure uniform filler dispersion before the pre-pregging process, the solution was magnetically stirred for 1 hour. The PLA96 fibres are subsequently drawn through a solution of PLDLLA in acetone with or without HA particles, thus coating the fibres and producing the pre-preg using a pre-pregger machine. Due to the continuous nature of the pre-pregging process, the final HA filler content in the produced HA/PLA-PLA pre-preg was determined to be in the range of 20-25 wt.%, as verified by the ashing method. The pre-preg of HA/PLA-PLA was compression moulded into rods at 150 °C using a hydraulic press (Bradley & Turton Ltd, Kidderminster, UK). A pressure of 8 MPa was applied gradually while the mould was heated. Once the temperature of 150 °C had been reached, a 20 MPa load was applied for 1 min. The mould was then allowed to cool to room temperature using water quenching. The sample size was 4 mm in diameter and 60 mm in length. Prior to testing, all samples were gamma irradiated at a dose of 2.5 Mrad by Isotron plc for the purpose of terminal sterilisation, mimicking standard medical implant protocols. As all the sample groups were subjected to the identical sterilisation process, any irradiation effect on the polymer system is negligible and relative comparisons of their mechanical and fatigue properties remain valid.

2.3 Quasi-static Flexural Test

Three-point static flexural tests were conducted using a modified ASTM D790-17 standard adapted for cylindrical specimens. Testing was performed on an MTS 858TM Mini Bionix servo-hydraulic testing machine (MTS, Minnesota, USA) equipped with a 2 kN load cell, using a crosshead speed of 2 mm/min at a span length of 45 mm. Six samples for each variable (unfilled SR-PLA, and composites containing micro (µm-HA) and nano (nm-HA) millimetre sizes of HA filler) were tested until fracture to determine the ultimate flexural strength (UFS), modulus and strain at break.

2.4 Flexural Fatigue Test

The flexural fatigue testing was performed in three-point bending using MTS 858TM Mini Bionix servo-hydraulic testing machines (MTS, Minnesota, USA) at room temperature with a sinusoidal waveform, at a frequency of 2Hz and a load ratio (P_{min} /P_{max}) of 0.1. The maximum load was 80 % of the composite ultimate strength (UFS). Due to the time-intensive nature of the test, a sample size of n = 6 was used for each group, which was tested under fatigue until fracture. Load and displacement data were recorded at 2Hz, capturing 10 data points per load cycle of every 100 load cycles up to 1000 cycles, afterwards every 1000 cycles up to 10,000 cycles, and continuing in this logarithmic progression until failure. The acquired load and displacement data were used to calculate stress, strain, secant modulus, and energy absorbed per load cycle.

2.5 Morphology Analysis

The fractured surface morphology and polished sections of the specimens were examined using a Scanning Electron Microscope (6300^{TM} JEOL, Japan). The samples were mounted onto stubs using a conductive adhesive tab and then coated with gold. An accelerating voltage of 10 kV was used for the examinations.

2.6 Weibull Distribution

The results obtained from the fatigue test were analysed using a two-parameter Weibull model to establish fatigue life survival probability as shown in Equation 1[16].

$$P_s(x) = 1 - P_f(x) = e^{-\left(\frac{x}{\alpha}\right)^{\beta}} \tag{1}$$

where $P_f(x)$ is the failure probability, x is the number of cycles, and α and β are the scale and shape parameters of the Weibull distribution, i.e., α is the Weibull characteristic of life, and β is the Weibull modulus. A linear regression was performed by plotting $ln(ln(1/1-P_f(x)))$ given by Equation 2 [16]. The β was determined directly from the slope of the fitted line, while the α was calculated from the y-intercept.

$$\ln\left(\ln\left(\frac{1}{1-P_f(x)}\right)\right) = \beta\ln(x) - \beta\ln(\alpha) \tag{2}$$

The failure probability can be determined from Equation 3 [16].

$$P_f(x) = \frac{i-0.3}{n+0.4} \tag{3}$$

The Weibull median fatigue life, which represents the specific number of cycles at which 50 % of the composite is expected to fail ($P_{f(x)} = 0.5$), was determined from the generated survival probability curves.

3. RESULTS AND DISCUSSION

3.1 Quasi-static Flexural Analysis

Figure 1 shows the effect of particle size filler on the flexural properties of the composite. The initial flexural strength of the sr-PLA is 91.83 (±2.60) MPa. It can be observed that the addition of micro and nanosized fillers has increased their strength by 6 and 20 %, respectively. The presence of filler in the composite may have a positive influence on its mechanical properties, as seen in previous research [17]. The increase in flexural strength is believed to be due to increases in the interface area with a decrease in particle size, which leads to both lower stress concentrations around the smaller particles and smaller voids being formed when debonding of the particle occurs.

The composite in this study is subjected to shear loadings. While direct interfacial measurements were not conducted, the presence of the HA filler may aid in load transfer between the PLLA fibre and PLA matrix, potentially delaying the crack propagation under the flexural stress. Smaller filler size (nm-HA) provides a higher surface-to-volume ratio than µm-HA. Subsequently, this greater surface area contact is proposed to facilitate a more effective interfacial interaction, thus contributing to the improved mechanical properties observed. This finding is similar to that reported by

Kryszak et al. [18]. The flexural strengths for filled samples were within the range of values found in cortical bone [19], suggesting that the composite will be mechanically suitable for use in bone fracture repair. A similar outcome is observed with the flexural modulus of the composite. The presence of the HA filler contributes to the increase of the initial modulus of the sr-PLA from 2.95 (± 0.91) GPa to 3.35 (± 0.06) GPa and 3.80 (± 0.05) GPa for the micron and nanosized HA fillers, respectively. Because HA has a high intrinsic stiffness, its incorporation into the PLA matrix is thought to restrict the mobility of the polymer chains, resulting in stiffer composite materials.

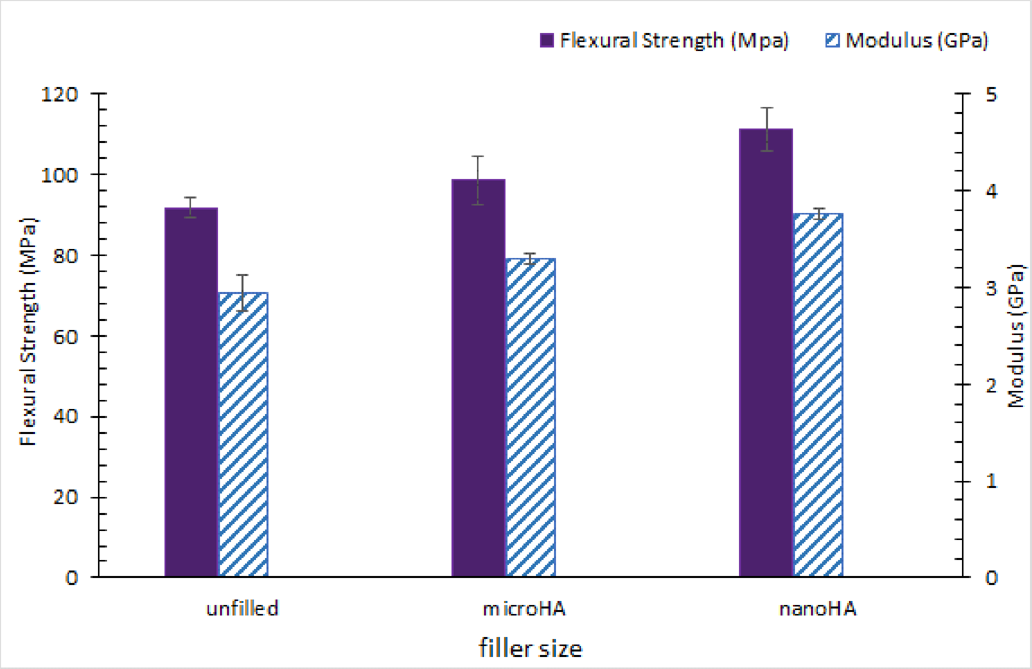

Figure 1: Effect of filler size on flexural strength and modulus of sr-PLA composites

3.2 Fatigue Analysis

Table 1 shows the effect of the filler size on the fatigue life of the sr-PLA at high stress levels. The flexural strength was used to calculate the upper stress used, and the lower stress was 10 % of the upper stress used in each case. The displacements at failure are given in Table 1, which was obtained from the last complete cycle before failure. The mean of each set of fatigue tests is given along with the Weibull median. The Weibull median was obtained from the curve as the number of cycles to failure corresponding to the 50 % probability of failure (Figure 2). Based on the tested samples, the unfilled PLA composite exhibited the highest fatigue life, with an estimated Weibull median life exceeding 400,000 cycles at 80 % of its ultimate flexural strength. Although the incorporation of HA particles increases the quasi-static stiffness of the composite by restricting polymer chain mobility, this compromises their fatigue life. The Weibull median fatigue life of nm-HA/sr-PLA and µm-HA/sr-PLA composite was 254,884 cycles and 104,514 cycles, respectively. Under cyclic loading, the lack of chemical coupling between the PLA matrix and HA particles may transform the rigid filler into a localised stress concentrator [20]. Consequently, micro-cracks initiate more readily at these poorly bonded interfaces, leading to a shorter fatigue life compared to the unfilled sr-PLA. The unfilled matrix, conversely, can dissipate cyclic energy more uniformly through plastic deformation without interruption of rigid non-bonded particles.

The distribution of the fatigue lives is quite crucial in designing materials for biomedical implants. A narrow spread of fatigue life is preferable as it suggests the material behaviour is more predictable during application [19]. Through Weibull analysis, the slope (β), which corresponds to the Weibull modulus as well as the scale parameter (α) (Figure 2), can be obtained by fitting the linear regression to the experimental data of the sr-PLA composite studied (Table 1). It can be observed that the confidence index (R^2) of the unfilled sr-PLA and HA-filled sr-PLA composites consistently exceeds 0.89. In accordance with standard practices for a limited dataset, this indicates a strong correlation with the experimental data and confirms the adequacy of the two-parameter Weibull distribution fit. The composite produced an α value > 1, indicating that the damage occurs in the composite system due to the fatigue load [16]. The shape parameter (β) represents the dispersion of the fatigue data, which physically correlates to the predictability of the failure mechanism. A higher β value, such as seen in the unfilled sr-PLA (β =3.57) and nm-HA composites (β = 3.50), implies a predictable 'wear-out' failure mechanism where damage accumulates steadily. Conversely, the much lower β of the µm-HA composite (β = 1.91) indicates a wider scatter in the data. This finding suggests that failure in the m-HA composite samples is driven by unpredictable, random macroscopic defects such as poor dispersion, larger voids, or severe stress concentration around the larger micron-sized particles. The scale parameter (α), which signifies the material's characteristic endurance capacity, was reduced by 77 % (from 514,659 to 114,191 cycles) with the addition of µm-HA particles. Nevertheless, this capacity rebounded approximately by 140 % (to 282,983 cycles) when substituting the micron filler for nano-sized HA particles. This confirms that minimising filler size is crucial to mitigating defect-driven premature fatigue failure in these composite systems.

Table 1: Effect of HA filler size on the high-stress flexural fatigue life and Weibull parameters of sr-PLA composites

Filler size	**Upper stress, (%)**	**Cycle to failure (N_f)**		**Final displacement (mm)**	**Weibull Parameters**		
		Range	mean		Weibull median	α	β
Unfilled	80	290, 013 - 573,513	496, 201	5.03	441, 802	514, 659	3.57
µm-HA	80	44,156 - 148,664	97, 551	4.40	104, 514	114, 191	1.91
nm-HA	80	229,529 - 328,775	275, 269	4.19	254, 884	282, 983	3.50

Figure 3 shows the survival probability of the sr-PLA composite when incorporated with different sizes of HA at high stress levels (80 % UFS). This information is crucial to determine the fatigue life at any survival probability. For example, the fatigue life for a survival probability of 50 % under high stress levels can be determined by drawing a horizontal line from the vertical line axis to intersect with the distribution curve. At a high stress amplitude, the probability of 50 % survival of the unfilled sr-PLA composite is 441,802 cycles. At the same survival rate, the presence of smaller HA filler increases the fatigue life of the composites from 104,514 cycles (µm-HA/sr-PLA) to 254,884 cycles (nm-HA/sr-PLA), respectively. The enhancement of the fatigue resistance by incorporating a smaller size filler using the Weibull method is 140 %. The characteristic life (α) value can be determined when $x = \alpha$ in Equation 2, corresponding to a survival probability of $Ps(x)= 0.368$ (or 36.8 %). On the other hand, at this point, 63.2 % of the tested population is expected to fail. Reaching this value indicates that the composite has survived up to its baseline characteristic endurance.

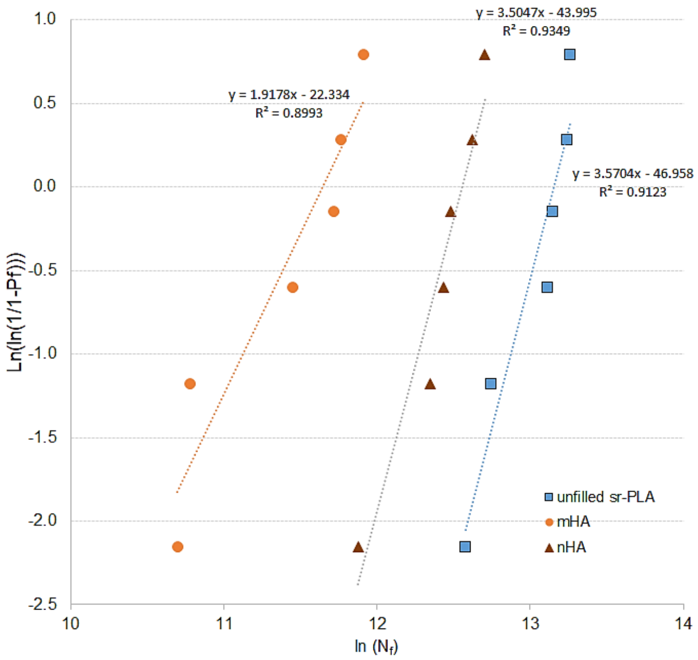

Figure 2: Two-parameter Weibull plot for fatigue life of SR-PLA composite with different filler sizes

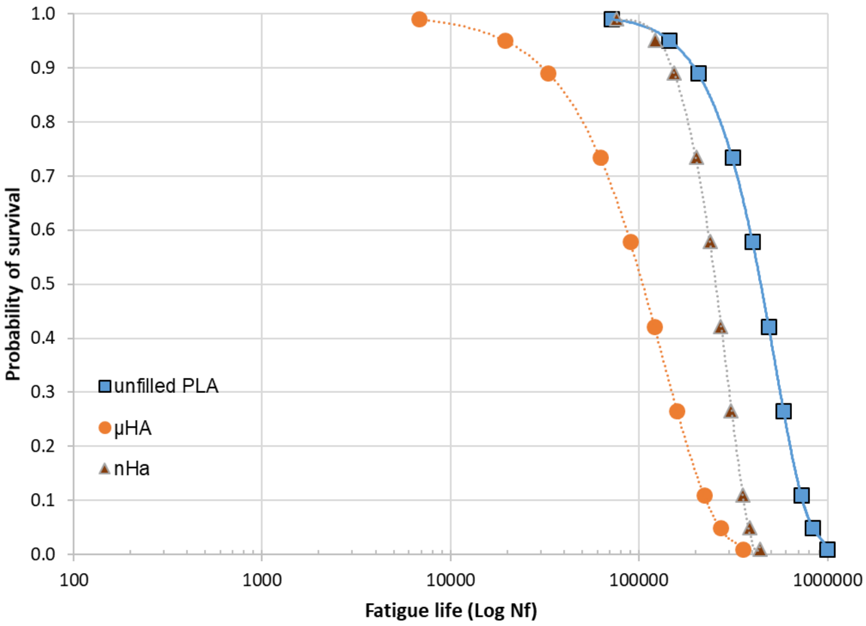

Figure 3: Survival probability graphs for sr-PLA composite with different filler sizes at 80 % stress levels

It is important to note that these fatigue trends are based on a limited sample size subjected to a single high-stress amplitude (80 % UFS). While Weibull analysis provides a robust statistical framework for predicting survival probabilities from small datasets, further investigation across multiple stress levels is necessary to establish comprehensive stress-life (S-N) curves. Nevertheless, the observed behaviour at this high stress level highlights a critical trade-off commonly encountered in polymer nanocomposites. The incorporation of inorganic fillers often enhances quasi-static properties such as stiffness and flexural strength, but simultaneously introduces microstructure and discontinuities as suggested by Habib et al. [21]. In this present HA- filled sr-PLA system, these rigid particles act as localised stress concentrators under cyclic loading. Consequently, while the composite is statically stronger, the uncoupled filler-matrix interface becomes a limiting factor for dynamic fatigue resistance, emphasising the need for optimised interfacial engineering in future implant designs.

3.3 Composite Fracture Morphology

The fracture morphology due to the flexural fatigue of the sr-PLA is shown in Figure 4. Closer inspection of the cross-section of the specimens indicated that failure occurred predominantly at the matrix-fibre interfaces (Figure 4 (b)). In HA-filled composites, as no chemical coupling was utilised during composite fabrication, the interaction at the HA/sr-PLA interface is expected to rely primarily on mechanical interlocking rather than chemical adhesion. Consequently, the fatigue fracture surfaces contained evidence of HA particles residing at the bottom of wells, alongside drawn polymer chains (Figures 5(a) and 6)). This evidence of particle pull-out and interfacial cracking suggests that limited interfacial bonding likely played a significant role in the failure mechanism, acting as sites for void initiation and crack propagation under cyclic loading.

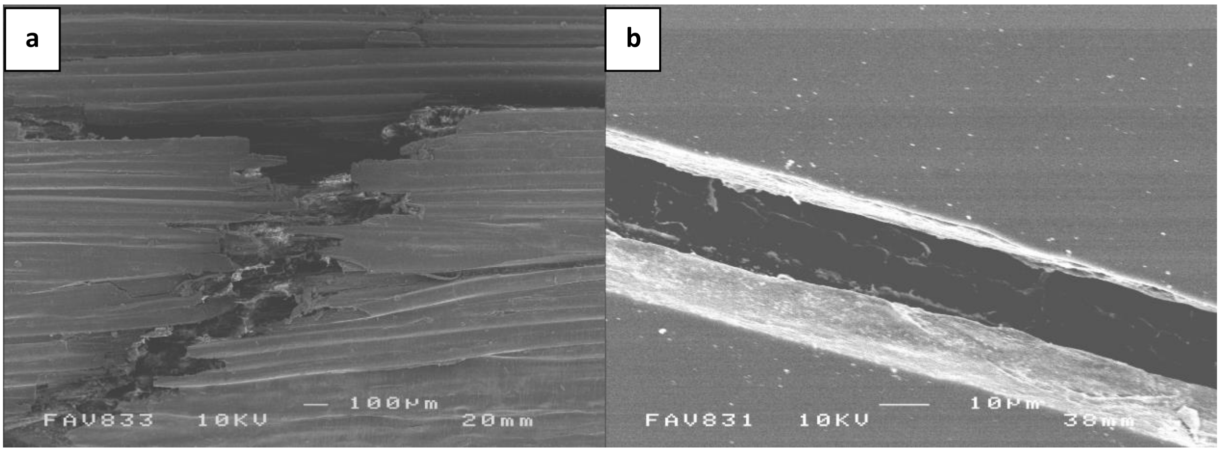

Figure 4: Fatigue damage observed on the specimen surface for unfilled PLA, observed on (a) surface fracture of the unfilled sr-PLA composite, Marker bar =100 µm and (b) cross section showing the crack propagation observed along the interface fibre–matrix. Scale bar =10 µm

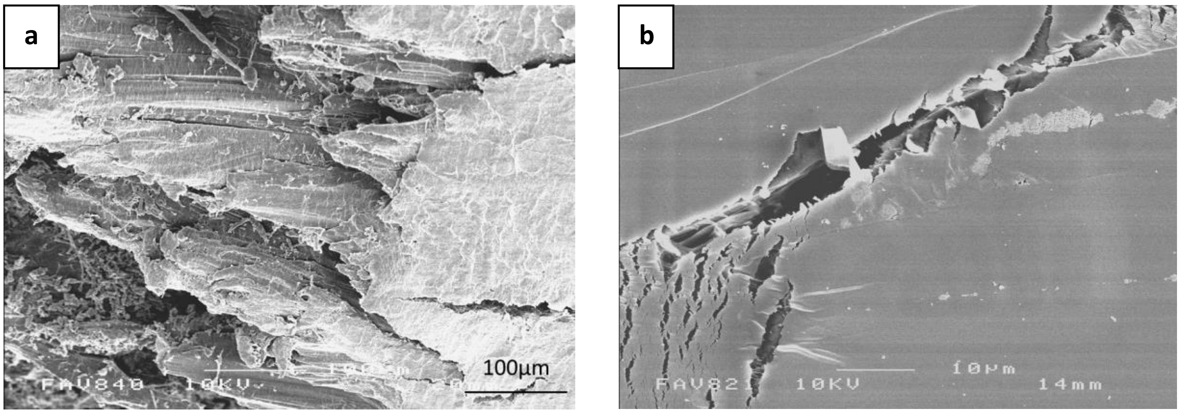

Figure 5: Fatigue damage observed in µm-HA/sr-PLA composite observed on (a) specimen surface. Marker bar =100 µm and (b) cross-section showing fatigue crack propagation observed along the interface fibre–matrix. Scale bar =10 µm

At the same time, it was observed that fatigue resistance is reduced in the presence of the filler. Even though energy was absorbed by the ductile matrix region through plastic deformation during the fatigue loadings, the resultant energy from the cyclic loading is sufficient for void initiation and crack propagation at the PLA-HA interfaces. This observation is consistent with the findings of this study, where fatigue fracture path in the composites predominantly followed the interfaces between PLA matrix and HA particles (Figure 5(b)), indicating the composite failed due to weak interfacial bonding [22, 23]. The composites are also more susceptible to tensile stress than compressive stress, where all the samples in this study experienced failure on the surface subjected to tension during bending fatigue.

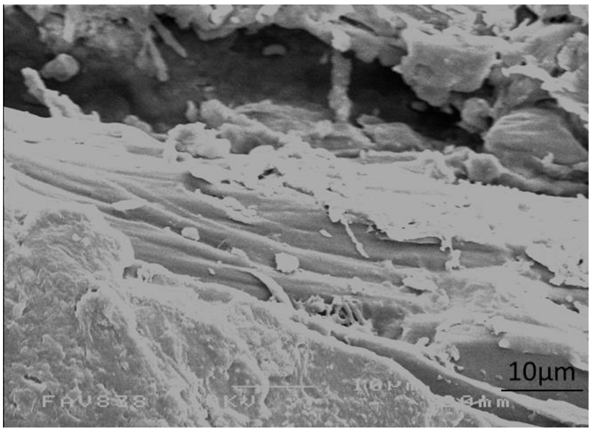

Figure 6: Fatigue damage observed on the specimen surface of nm-HA/ sr-PLA. Scale bar =10 µm

Although the morphological evidence is qualitative, it aligns well with quantitative Weibull analysis, where larger interfacial defects and broader data scatter (lower β value) are observed in the µm-HA composites, further supporting the conclusion that failure is largely driven by interaction at the filler/matrix interface. While both µm-HA and nm-HA systems rely on this limited mechanical interlocking, the higher surface-to-volume ratio of the nm-HA particles specifically delays crack initiation. Nanoparticles are vastly smaller and more densely distributed. Therefore, the applied cyclic load is dispersed over a significantly larger total interfacial area. This widespread dispersion minimizes the localized stress concentration at any single, poorly bonded interface. In contrast, the µm-HA particles present larger, continuous surface areas of weak bonding that act as critical macroscopic flaws where stress is easily concentrated, leading to earlier void formation and premature crack initiation. Furthermore, Chanda et al. [24] point out that fatigue strength increases with decreasing particle size. As the distance between particles decreases, the stress concentrations set up around them will interfere, and adjacent fields will interact, thereby hindering crack propagation. This sensitivity to filler size under cyclic loading is highly consistent with the investigation reported in the literature [18].

4. CONCLUSIONS

The study evaluates the quasi-static and high-stress flexural fatigue behaviour of HA/sr-PLA composites. The incorporation of HA particles increased the initial flexural strength and modulus of the sr-PLA composite, and utilising smaller nano-scale HA further enhanced their properties to be within the range of cortical bone. The fatigue life of the composite at an 80 % stress amplitude was statistically analysed using two-parameter Weibull distribution functions. While limited to this high-stress regime, the Weibull median fatigue life of HA/sr-PLA increased significantly with the use of smaller nano-scale HA particles compared to micro-scale particles. This indicated that minimising filler size is a critical factor in mitigating premature fatigue failure in these sr-PLA composites. Morphology analysis suggests the composite primarily failed due to a combination of the fibre fracture and damage initiated at the matrix/filler interface. Ultimately, these findings indicate that while HA fillers improve static strength, weak interfacial bonding limits high stress fatigue life. Minimising filler size is a critical factor in mitigating premature failure, which must be considered when designing bioabsorbable implants for bone fixation.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors declare no potential conflict of interest in the publication of this work.

Compliance with Ethical Standards

The work is compliant with ethical standards.

References

[1] Mustafa, Z. & Tanner, K. E. (2011). Composites for hard tissue repair. *Wiley Encyclopedia of Composites*, 1-14.

[2] Egerci, O. F., Dogruoz, F., Cetin, H., Ertan, M. B., Yapar, A. & Kose, O. (2025). High rate of failure after magnesium bioabsorbable compression screw fixation for scaphoid fractures. *Journal of Orthopaedic Surgery and Research,* 20, 1-11.

[3] Kim, T., See, C. W., Li, X. & Zhu, D. (2020). Orthopedic implants and devices for bone fractures and defects: past, present and perspective. *Engineering Regeneration,* 1, 6–18.

[4] Li, J. W., Du, C. F., Yuchi, C. X. & Zhang, C. Q. (2019). Application of biodegradable materials in orthopedics. *Journal of Medical and Biological Engineering,* 39(5), 633-645.

[5] Hussin, M. S. F., Idris, M. I., Abdullah, H. Z., Mukhtar, M. F., Roslan, M. F. & Ahmad, M. N. (2025). Preparation and characterization of PLA/Hap composite for biomedical applications. *Malaysian Journal of Microscopy,* 21(2), 234-244.

[6] Khan, Z. I., Habib, U., Mohamad, Z. B. & Khan, I. (2024). Mechanical and thermal properties of a newly developed sepiolite filler-filled rPET/PA11 thermoplastic nanocomposites. *Results in Engineering,* 21, 101731.

[7] Zhao, R., Meng, X., Pan, Z., Li, Y., Qian, H., Zhu, X., Yang, X. & Zhang, X. (2025). Advancements in nanohydroxyapatite: synthesis, biomedical applications and composite developments. *Regenerative Biomaterials,* 12, 1-31.

[8] Zhang, Z., Chen, G., Wang, J., Hu, X., Hou, P., Xiong, C. & Zhang, L. (2025). A novel ternary polymer/β-TCP composite microspheres with polydopamine-encapsulated nano zinc oxide coating for bone tissue engineering. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 709, 136219.

[9] Selvam, S. P., Ayyappan, S., Jamir, S. I., Sellappan, L. K. & Manoharan, S. (2024). Recent advancements of hydroxyapatite and polyethylene glycol (PEG) composites for tissue engineering applications–A comprehensive review. *European Polymer Journal*, 215, 113226.

[10] Rodrigues, I. C. P., Llanos, J. H. R., Mendonça, L. H. P., Pereira, K. D., Luchessi, A. D., Lopes, É. S. N. & Gabriel, L. P. (2025). Rotary jet spun semicrystalline polymers containing nanohydroxyapatite increase bioactivity and cell adhesion for bone applications. *Journal of Bionic Engineering*, 1-14.

[11] Li, S., Wang, T., Hu, J., Wang, B., Li, Y., Wang, L., Li, G., Zhang, J. & Zhou, Z. (2021). Effect of hydroxyapatite content and particle size on the mechanical behaviors and osteogenesis in vitro of polyetheretherketone–hydroxyapatite composite. *Polymer Composites*, 42(12), 6512-6522.

[12] Cameron, P. M., Hutchinson, D. J., Malkoch, M., Varga, P. & Schwarzenberg, P. (2025). Cyclic testing reliability analysis on a novel light-curable bone fixation technique. *Frontiers in Bioengineering and Biotechnology*, 13, 1-9.

[13] Silva-Henao, J. D., Schober, S., Pahr, D. H. & Reisinger, A. G. (2024). Critical loss of primary implant stability in osteosynthesis locking screws under cyclic overloading. *Medical Engineering & Physics,* 126(1), 104143.

[14] Gautam, K., Jimenez, H. G., Dikici, Y., Bensusan, J., Wnek, G. E., Rimnac, C. & Akkus, O. (2026). Optimization of 3D printing parameters for PLGA/HA scaffolds using the Taguchi method. *Journal of the Mechanical Behavior of Biomedical Materials*, 178, 107385.

[15] Pazhamannil, R. V. & Alkhedher, M. (2026). Structure-property relationships in 3D printed bone scaffolds: Role of TPMS architecture and porosity in PLA and nanohydroxyapatite reinforced composites. *Journal of Thermoplastic Composite Materials*, 39(3), 984-1010.

[16] Mustafa, Z., Nawi, T. I., Fadzullah, S. H. S. M., Shamsudin, Z., Malingam, S. D. & Ratanawilai, T. (2021). Fatigue characteristic and Weibull analysis of sustainable rubberwood flour/recycled polypropylene composites. *International Journal of Automotive and Mechanical Engineering*, 18(4), 9179-9187.

[17] Oosterbeek, R. N., Zhang, X. C., Best, S. M. & Cameron, R. E. (2021). A technique for improving dispersion within polymer–glass composites using polymer precipitation. *Journal of the Mechanical Behavior of Biomedical Materials*, 123, 104767.

[18] Kryszak, B., Ujčić, A., Gajdošová, V., Šlouf, M. & Szustakiewicz, K. (2025). Micro vs nano: influence of filler size on the rheological and mechanical properties of highly-filled PLA/HAp and PCL/HAp composites. *Journal of Materials Research and Technology*, 37, 2919-2934.

[19] Harper, E. J. & Bonfield, W. (2000). Tensile characteristics of ten commercial acrylic bone cements. *Journal of Biomedical Materials Research*, 53, 605-616.

[20] Tazibt, N., Kaci, M., Dehouche, N., Ragoubi, M. & Atanase, L. I. (2023). Effect of filler content on the morphology and physical properties of poly (lactic acid)-hydroxyapatite composites. *Materials*, 16(2), 809.

[21] Habib, U., Mohsin, M. E. A., Khan, Z. I., Mohamad, Z., Othman, N., Mousa, S., Hossain, S.K.S. & Ali, S. S. (2025). Mechanical, thermal, and flammability properties of eco-friendly nanocomposites from recycled PET/PA-11 blends reinforced with graphene nanoplatelets. *Polymers,* 17(8), 1038.

[22] Arora, A., Sharma, A., Singh, M., Mahajan, D. K. & Kushvaha, V. (2023). Fatigue response of glass-filled epoxy composites: A crack initiation and propagation study. *International Journal of Fatigue*, 170, 107542.

[23] Pan, Y., Mao, J. & Ding, J. (2018). Fatigue performance of hydroxyapatite-filled polyetheretherketone functional gradient biocomposites. *Materials Technology*, 33(12), 761-768.

[24] Chanda, J., Mishra, N., Dolui, T., Ghosh, P. & Mukhopadhyay, R. (2022). Fatigue crack growth behavior and morphological analysis of natural rubber compounds with varying particle size and structure of carbon black. *Polymer Engineering & Science*, 62(3), 743-757.