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Mechanical Characterisation of Nano-Hydroxyapatite Reinforced PLA Composites Prepared by Dry Blending for Biomedical Applications

Omar A. Al Rumhi, Reem S. Al Mamari, Nassr A. Al Kiyumi, Issa M. Al Sibani, Elyas H. Al Abbadi, Mohammed S. Al Owiemri and Moosa S.M. Al Kharusi
Department of Mechanical and Industrial Engineering
Sultan Qaboos University, Muscat, Oman

Abstract

This study focuses on fabrication and mechanical characterisations of biocompatible composites to be as a promising material for biomedical applications. Polylactic Acid (PLA) was reinforced with HA of average particle size 200 nm to enhance its mechanical performance by varying the HA loadings of 0 wt.%,0.25 wt.%, 0.5 wt.%, 1wt.%, and 2 wt.%. The composites were fabricated by blending the PLA pellets and nHA powder in their dry states, followed by compression molding to be ready for mechanical characterisation (compression, tensile, and flexural) according to ASTM D695, ASTM638, and ASTM D790 standards, respectively. Notably, the experimental results indicated that the addition of nHA to PLA matrix affected the mechanical behavior of the overall composites. The composite with 1 wt. % HA content achieved the optimal performance among all loading, with 7.8% improvement in tensile strength as well as a significant improvement in both compressive and flexural properties. Conversely, the strength declined at higher HA contents owing to the agglomeration, interfacial bonding within the matrix, and voids formation that acts as stress concentration points leading to the specimen fracture.

Keywords

PLA composites, Hydroxyapatite, Biomedical implants, Compression molding, Mechanical properties

1. Introduction

The growing demand for advanced composites in biomedical implants comes from the need for reliable options that can handle both mechanical loads and biological interactions inside the body. Among the various polymer composites developed in medical implant applications, the combination of Polylactic Acid (PLA) and Hydroxyapatite (HA) has gained special attention. Polylactic Acid (PLA) is a bio based, and sustainable polymer produced from renewable and natural sources such as corn and sugarcane. It is popular in medical implants applications because of its thermoelectricity, biocompatibility, and lightweight. When reinforced with Hydroxyapatite (HA), the composite shows notable improvements in mechanical properties and in biological response, which makes the composite (PLA/HA) suitable for the medical implant applications (Hussain et al., 2024). Notably, Polylactic Acid (PLA) possesses several distinguished features compared to other polymers including sustainability, biodegradability, and no-toxic biocompatibility. It has been used in medical implants applications such as scaffolds, implant fabricates, and bone tissue engineering. PLA has the ability to dissolve and degrade naturally over a specific period and without causing negative effects inside the body (Custodio et al., 2021). When reinforced with HA, PLA's bioactivity and mechanical properties are significantly enhanced (Ko et al., 2021). Studies, such as those by (Nguyen et al., 2023) have demonstrated that nano sized HA enhances the mechanical strength, thermal and anti-corrosive features. The overall properties of the PLA/HA composites vary depending on the amount of HA percentage added. One major limitation of using pure ceramics such as HA is their brittleness. However, mixing the ceramic particles (HA) with a PLA matrix increases the overall composite toughness. This combination takes advantage of the flexibility of PLA and the hardness of the HA, producing a strong composite that can withstand repeated loading without cracking or failure (Omigbodun et al., 2024). These findings show the importance of PLA/HA composites in advanced medical implant technology that can provide a combination of strength and biological performance.

Ensuring uniform dispersion of hydroxyapatite (HA) within the polylactic acid (PLA) matrix is critical for developing composites with reliable mechanical strength and biocompatibility for biomedicals implant applications. (Ahmadi et al.,2024) have effectively demonstrated a method for enhancing the homogeneity of polymer blends through high-pressure and shear deformation, providing a viable strategy for achieving uniform dispersion in composite materials. Melt consolidation techniques with a prior blending stage have been shown to blend homogeneity and stability (Li et al.,2024).

PLA/HA composite mechanical properties vary based on the particle size of the HA. Nano-scale reinforcements have been observed to provide more desirable mechanical properties compared to composites containing micrometric fillers (Kryszak, Bartłomiej, et al, 2025). The nano-HA provides a high surface-area-to-volume ratio resulting in strong interfacial bonding with the PLA and effective stress transfer, improving mechanical properties like compressive strength. Such improvements in mechanical properties make PLA/nHA composites suitable for load-bearing implants in bone and tooth defects due to their biocompatibility with bone tissues, and strength. (Farkas, Noémi-Izabella, et al.2022).

The fabrication of PLA/nHA composites for biomedical use typically employs melt-based processing techniques such as twin-screw extrusion followed by compression molding, producing mechanically stable samples for load-bearing implants. Mechanical testing such as tensile, compressive, and flexural assessments are conducted to evaluate load-bearing capacity under simulated physiological conditions. In vitro studies revealed that 3D-printed PLA/nHA composite scaffolds promoted cell adhesion, proliferation compared to pure PLA scaffolds (Wang, Wenzhao, et al.,2021). These evaluations contribute to the development of PLA/nHA composite implants that can endure physiological loading while encouraging strong bone tissue integration, thereby improving the performance and success of biomedical implant applications.

The incorporation of 3D printing creates a strong link between digital design (e.g., patient scan information) and real-world clinical use. This approach improves the overall dependability and efficiency of PLA/HA implants by ensuring that each part is customized to meet the specific needs of the individual patient. The real strength of 3D printing is found in its capacity to control the PLA/HA composite with exceptional accuracy. The polymer (PLA) offers foundational structural support and is intended to degrade harmlessly over time, whereas the ceramic (HA) supplies the bioactive sites that promote the formation of new bone (Raziyan, 2024). The ability to customize on demand establishes 3D printing as the primary route for the future enhancement of these advanced bone repair composites.
This research addresses the effects of nano-HA fillers on the mechanical properties of PLA composites. By examining HA's influence on mechanical properties of the composite, this study contributes valuable insights into the development of high-performance PLA composites for biomedical applications, such as orthopedic implants and bone scaffolds. These findings underscore the potential of PLA/HA composites as sustainable and durable materials suitable for applications that require both structural integrity and biological compatibility.

1.1 Objectives

This study was designed to systematically investigate the fabrication and mechanical performance of the PLA/nHA composites to be used in a biomedical scope. According to that, the objectives below are formulated to ensure a comprehensive assessment of the development and performance enhancement of the material.

- To fabricate PLA/nHA composite specimens with different filler loadings using a compression molding method.
- To characterize the prepared composite specimens in terms of mechanical properties through tensile, compression, and flexural tests.
- To analyze and compare the mechanical behavior of PLA/nHA composites with the findings of the existing literatures.

2. Materials and Methods

This section exhibits the fabrication and mechanical characterisation of Polylactic acid (PLA) composites reinforced with nano-hydroxyapatite (nHA). The workflow was intentionally designed to enhance mechanical performance and be appropriate for biomedical applications. The following flowchart represents the methodology which begins with composite fabrication and ends with testing and results analysis as shown in Figure 1.

Figure 1. Experimental procedure for the fabrication and mechanical characterisation of PLA/nHA composites.

2.1 Fabrication of PLA/HA composite

In this study, Hydroxyapatite-reinforced Polylactic acid (PLA/HA) composites were prepared using dry blending technique. This process involves blending the polymer in pellet form with the filler in powder form in their dry state, followed by compression molding (Figure 2). The HA filler was added with various loadings of 0 wt.%, 0.25 wt%, 0.5 wt.%, 1 wt.%, and 2 wt.% as shown in Table 1. The total 120 g for three tests of composite material is uniformly filled with 30 g for compression mold, and 90 g for tensile and flexural plate mold. The variation of HA percentages shown provides an in-depth assessment of the nano fillers' effect on mechanical compressive, tensile, and flexural strength and their suitability in the biomedical scope. Fig. 2. Clearly shows dispersion of HA particles at various loadings of 0.25wt.%, 0.5wt.%, 1wt.%, and 2wt.%, in fabricated tensile specimens. The PLA/ HA formulations are shown in the Table 1 below:

Table 1. Formulations of PLA reinforced with nano Hydroxyapatite (HA).

Sample No.	Formulation	PLA content	HA content (wt.%)
1	Pure PLA	100%	0%
2	0.25wt.% HA	99.75%	0.25%
3	0.5wt.% HA	99.5%	0.5%
4	1wt.% HA	99%	1%
5	2wt.%. HA	98%	2%

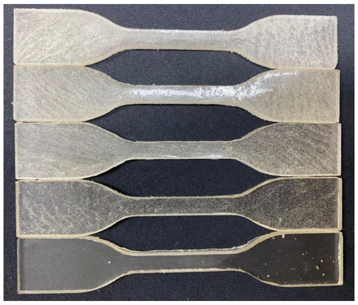

Figure 2. Dispersion of HA particles at various loadings of 0.25wt.%, 0.5wt.%, 1 wt.%, and 2 wt.%.

2.2 Mechanical characterisation

The mechanical performance of the PLA/nHA composites specimens was evaluated through compression, tensile, and flexural testing following the ASTM D695, ASTM D638, and ASTM D790 standards. Prior to testing, the required dimensions of specimens were precisely measured via a digital caliper to ensure dimensional accuracy. Compression and three-point flexural tests were conducted using a Tinius Olsen H50KT universal testing machine (Tinius Olsen Ltd., Redhill UK; Serial No. H50KT-0167). Tensile tests were carried out using a Tinius Olsen H5KT universal testing machine (Tinius Olsen Ltd., Redhill UK; Serial No. H5KT-0807). Compression testing was applied at 50 % of sample's height, with constant crosshead speed of 1.3 mm/min, and under load range of 5000N. Additionally, tensile test was conducted on the dog bone-shaped specimens at 1.5 mm/min, while the flexural test was applied using a three-point bending setup at a speed of 5 mm/min. The resulting extension- force and strain stress curves data were recorded to determine the compressive strength, tensile strength, flexural strength, and deformation characteristics of the composite. This analysis provides a comprehensive understanding of how the nano HA filler influences the mechanical behavior of PLA matrix under compressive load.

2.3 Polylactic acid (PLA)

The PLA material shown in Fig. 3 was used in the form of pellets and was commercially sourced from PLAMORE company, a local supplier. This PLA possesses several characteristics, such as its excellent processability and biodegradability (Figure 3).

Figure 3. PLA Pellets.

2.4 Hydroxyapatite (HA)

The hydroxyapatite (HA) used in this study had an average size for the particles of 200 nm and obtained from Xi'an Bos Biotech Co., Ltd. (China). HA is employed in several medical applications such as orthopedics and dental implants due to its bioactive and biocompatible nature. Additionally, HA exhibits chemical compositions similar to that of the natural bone, enamel, and dentine, as presented in Table 2 *(Al-Sanabani et al., 2013)*. The calcium and phosphorus contents of these biological tissues closely match those of hydroxyapatite (HA), supporting its extensive use as a biomaterial in medical and dental applications.

Despite the size used in this study is 200 nanometers, which exceeds the usual size of <100 nanometers, it is still considered to be used in the field of biomedical research, as materials in the range of 100 - 300 nanometers are considered nanomaterials because they have a high surface area and distinctive biological activity (Rezić, 2022; Lee and Moon, 2020). Therefore, the 200 nm particles used in this work influence the crystallization and interfacial bonding of PLA/nHA composites (Owiemri *et al.*, 2025).

The SEM micrograph (Figure 4) of hydroxyapatite particles with averaging around 200 nm shown in Fig. 4., providing the surface morphology and crystal structure. These particles contribute to enhancing the mechanical performance of the composite. The tested samples were coated with a thin layer of gold (Au) by sputtering, and the surface microstructure was observed using a Field Emission Scanning Electron Microscope (FE-SEM, SUPRA 40 VP) operated at an accelerating voltage of 20 KV (Doostmohammadi *et al.*, 2011).

Table 2. Chemical and structural comparison of teeth, bone and hydroxyapatite (HA).

Compositions, wt.%	Enamel	Dentine	Bone	HA
Calcium	36.5	35.1	34.8	39.6
Phosphorus	17.1	16.9	15.2	18.5
Ca/Pa ratio	1.63	1.61	1.71	1.67
Total inorganic (%)	97	70	65	100
Total organic (%)	1.5	20	25	-
Water (%)	1.5	10	10	-

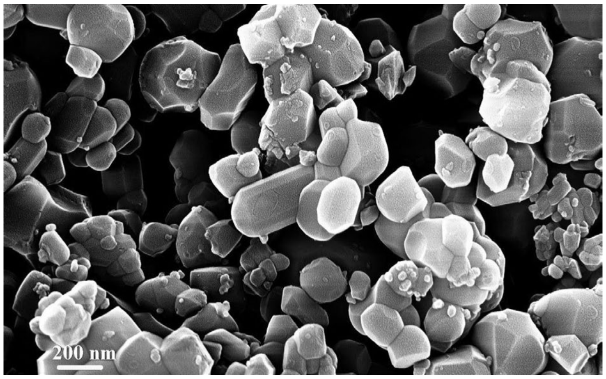

Figure 4. SEM Micrographs of Hydroxyapatite Particles

3. Results and Discussion

3.1. Overview of Mechanical performance

The mechanical performance of the PLA/HA composite was evaluated through compression, tensile, and flexural tests to evaluate and determine the effect of changing the reinforcement (HA) percentage of the composite. All specimens were fabricated using the compression molding technique to ensure test quality and consistent density. The results show some fluctuation in results but in general an enhancement in mechanical properties with increasing HA content up to 1 w%, followed by decline in higher concentration. This indicates that the additional HA can effectively improve the load transfer within the composite by enhancing bonding inside the composite and reducing the internal defects. However, beyond optimum, particle agglomeration might affect and reduce stress distribution within the matrix, causing a notable drop in mechanical strength.

3.2. Compression Test

Compression testing is an essential technique to evaluate mechanical performance and deformation behavior between materials. It gives an impression of the materials' strength, stiffness, and energy absorption capacity. The data presented in fig. 5 clearly shows the nonlinearity behavior gained from all the specimen types of various HA loading of 0 wt.%,0.25 wt.%, 0.5 wt.%, 1wt.%, and 2 wt.%. Pure PLA exhibited an average compression strength of 137 MPA. The addition of HA caused a small reduction up to 0.5 wt.%, followed by a clear recovery at 1 wt.%, where the properties reached their optimum value. At 2 wt. % a minor decline was recorded.

The variation in the results reflects the effect of nanoparticle dispersion within the polymer matrix. When the HA wt.% increases, the ability for particle agglomeration to take place will increase and become significant and cause stress concentration zones that weaken interfacial adhesion and trigger cracking under compression. Moreover, excessive nanoparticle loading tends to promote the formation of micro voids and pores within the PLA matrix due to poor filler dispersion (Figure 5- Figure 8).

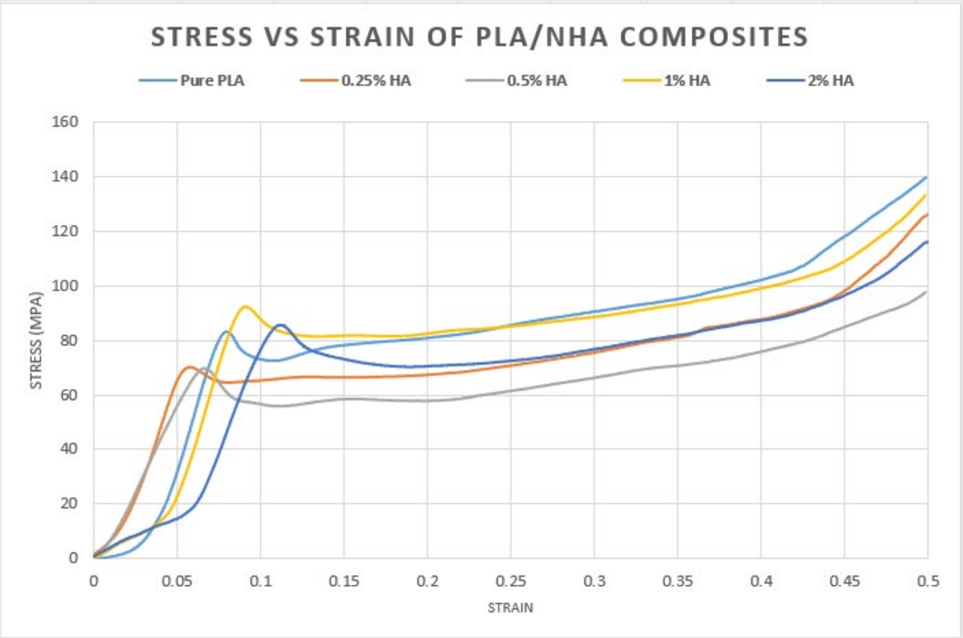

Figure 5. Stress–strain curves of PLA/nHA composite with different nano-hydroxyapatite contents at 0 wt.%, 0.25 wt.%, 0.5 wt.%, 1 wt.%, 2 wt.% HA for compression test.

3.3. Tensile Test

Tensile test was performed to evaluate the strength and deformation characteristics of the PLA/HA composites under uniaxial loading. The data presented in fig. 6 and fig. 7 show that all specimens initially undergo a linear elastic region, followed by yielding and strain softening before the final fractur. The gradual increase in ultimate tensile strength from pure PLA to 1 wt.% HA as illustrated in fig. 7 indicates the reinforcing effect of the HA nanoparticles. Beyond the optimum filler concentration (2 wt.%) a reduction in strength is observed. The tensile strength significantly increased from 53.15 MPA for pure PLA to 57.29 MPa at 1wt%, indicating an enhancement of approximately 7.8%. However, at 2 wt. %, the strength decreased slightly to 54.45 MPa, representing a reduction by 2.45% due to nanoparticle agglomeration, porosity, and void formation which limits the efficiency of stress transfer or distribution within the composite. This effect is mainly due to the manual dry mixing process which may cause non uniform distribution and weak bonding between the filler and the matrix which leads to localized stress concentrations under tensile loading.

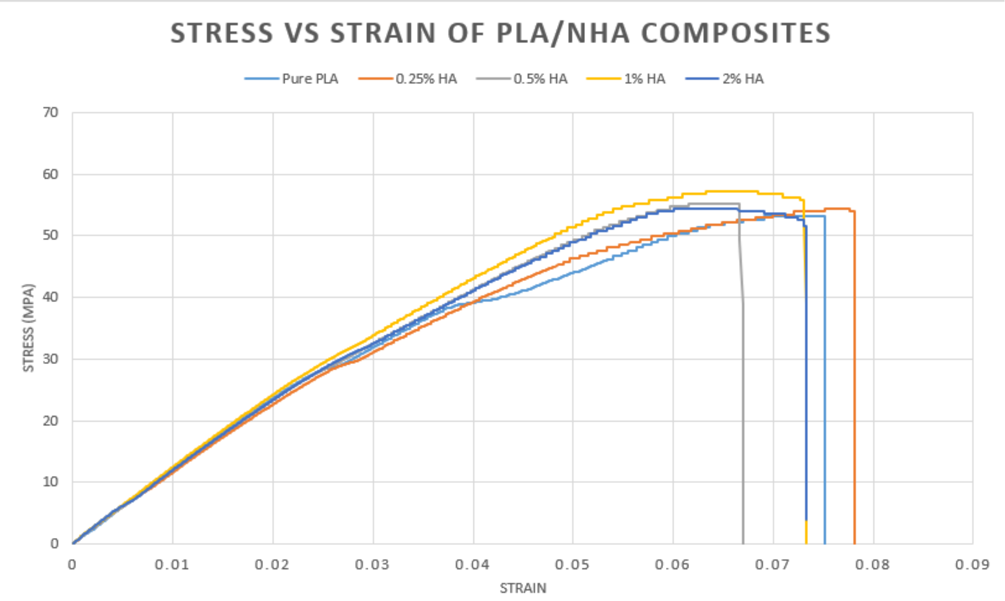

Figure 6. Stress–strain curves of PLA/nHA composites with varying nano-Hydroxyapatite contents (0 wt.%, 0.25 wt.%, 0.5 wt.%, 1 wt.%, and 2 wt.%) for tensile test.

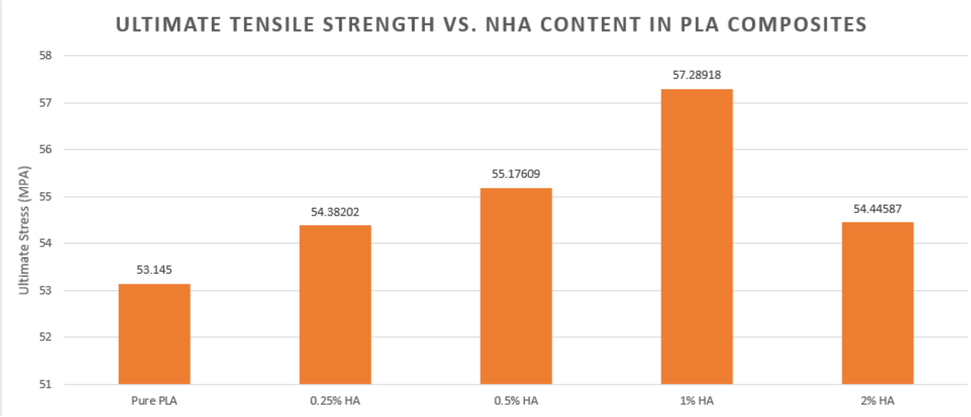

Figure 7. Ultimate Tensile Strength of PLA/nHA Composites at Different Nano-Hydroxyapatite Loadings.

3.3. Flexural Test

The flexural test gives valuable information to understand the flexural performance of the PLA/HA composite. The flexural stress-strain curve is shown in Fig. 8 exhibits that the highest flexural stress was in the pure PLA of approximately 140 MPa. The composite containing 1 wt.% of HA achieved the best improvement for the reinforced samples, reaching about 120 MPa and this is attributed to effective load transfer and mechanical interlocking between the matrix and filler. However, increasing the HA above 1 wt.% resulted in a decrease in flexural strength due to the non-uniform distribution of the HA particles leading to dominance of agglomeration. Additionally, the surface porosity behaved as stress concentrators and weak points within the composite, leading to premature failure underloads.

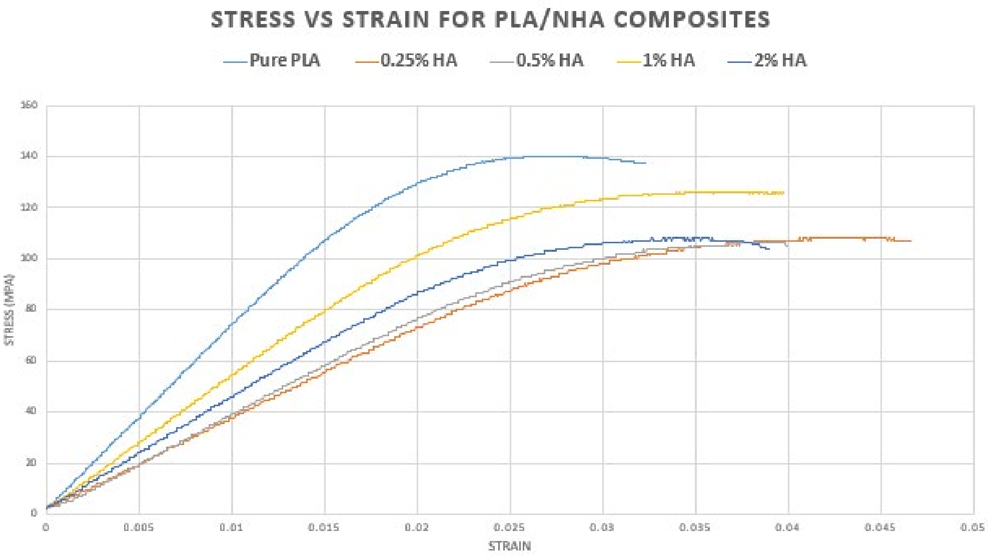

Figure 8. Stress–strain curves of PLA/nHA composites with varying nano-hydroxyapatite contents (0%, 0.25%, 0.5%, 1%, and 2%) for flexural test.

4. Conclusion

The study investigated the mechanical behavior of PLA/HA composites, focusing on the effect of nano-hydroxyapatite loading on the material compressive, tensile, and flexural performance. The results showed that incorporation of nano-HA influences the mechanical response of PLA. An optimal enhancement was achieved at 1 wt.%, indicating effective stress transfer and uniform particle interlocking within the polymer matrix. Beyond this concentration, a slight reduction in mechanical performance was observed potentially due to particle agglomeration, which caused stress, concentration, and weakness points in the material.

Future research on PLA/nHA composites could enhance the mechanical performance and particles bonding by optimizing HA nanoparticle size and surface treatment. Moreover, incorporating suitable coupling agents may also improve interfacial adhesion between polymer matrix (PLA) and the filler particles (HA). Using advanced analysis techniques such as Scanning Electron Microscopy (SEM) would provide a deeper insight into material behavior and internal failures. Also, parameter optimization, including control of cooling rate and molding pressure, could enhance crystallinity and dimension stability and influence mechanical performance. Furthermore, improving fabricating process techniques such as extrusion compounding, injection molding, or additive manufacturing to achieve better filler dispersion.

Overall, the findings clearly highlight that inclusion of 1 wt.% nano-HA provides the best balance between mechanical performance and biocompatibility, which makes it suitable for biomedical application.

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Biographies

Omar A. Al Rumhi is a Mechanical Engineering student at Sultan Qaboos University (SQU), Muscat, Oman, currently in his fifth year of the bachelor's degree program. He completed an international internship at the Manipal Institute of Technology (MIT Manipal), India, through the IAESTE program, where he worked on additive manufacturing of nickel-based superalloys, focusing on process optimization, binder jetting, and advanced microscopy characterisation. He also completed an industrial training placement at NAMA Water Services, Oman, where he gained experience in mechanical maintenance and system operations. His academic projects include the Mechanical Smart Door Lock Design, which integrates mechanical automation and security optimization, the design and development of a Waste Volume Reduction Device in collaboration with Aphcarios Engineering Solutions company, and the fabrication and testing of PLA/HA Composite Biomedical Implants as part of his final year project. He has experience using SolidWorks, AutoCAD, and ANSYS, and holds a strong foundation in 3D printing technologies.

Reem S. Al Mamari is currently pursuing her Master's degree in Mechanical Engineering at Sultan Qaboos University (SQU), specializing in material science and composite materials. Her research focuses on the development and optimization of polymer-based composites for biomedical applications, emphasizing the integration of 3D printing technologies to fabricate and characterize advanced functional materials. She holds a bachelor's degree in mechanical engineering from the University of Technology and Applied Sciences (UTAS), Oman.

Nassr A. Al Kiyumi is Mechanical Engineering undergraduate at Sultan Qaboos University, college of Engineering, Muscat, Oman with solid background about mechanical design and thermodynamics and fluid mechanics. He has proficiency with SolidWorks, Fusion 360, MathCAD, MATLAB as well as strong problem-solving and team-work abilities/ flexibility. Nassr also underwent on-the-job training at Oman Flour Mills and the Ministry of Defense (Royal Navy of Oman), performing fault-finding, maintenance and overhauling on mechanical equipment such as marine engines, pumps, valves and heat exchanger. He also worked part-time with Onsor Technologies during that time designing, testing and marketing their successful innovative devices. His academic projects include the full design of a multifunctional smart mechanical lock and a compact waste-reduction device for Apcharios Engineering Solutions. He has actively participated in professional development activities, including the Oman Materials, Corrosion and Integrity Summit, the 3D Design Course at the Military Technological College, and the Oman Competition for AI-Enhanced Drones. With over 150 operational hours of industrial experience, Nassr aims to combine creativity and engineering precision to design efficient, sustainable, and practical mechanical systems that contribute to Oman's growing engineering and industrial innovation landscape.

Issa M. Al-Sibani is a final-year Mechanical Engineering student at Sultan Qaboos University, Oman, expecting to graduate in 2026. He has gained practical engineering experience through training programs in the maintenance departments at both Oman Airport and OQ Company's refinery in Muscat. Mr. Al-Sibani has held important roles in the SQU Engineering Society, including Head of the Implementation Team, where he led a 200-member team for a large student event, and Vice-President of the Financial Team, where he secured sponsorships from over 20 companies and managed a budget over 50K OMR. He also helped organize the opening ceremony for the Mechatronics Society with special guests. His skills include problem-solving, fusion 360, data analysis, effective communication, and negotiation.

Elyas H. Al-Abbadi is a dedicated Mechanical engineering student at Sultan Qaboos University, Oman, currently in his fifth year. He is passionate about applying engineering principles, improving technical proficiency, and contributing to innovative projects. Elyas gained valuable hands-on experience during a two-month summer training program at Composite Building Innovation First (CBIF) in Tunisia, where he specializes in fiberglass production. While there, he successfully designed and implemented approved enhancements to factory machinery using SolidWorks. His core competencies include analytical thinking and problem-solving. His core competencies include analytical 3D modeling, project management, and proficiency. Elyas is committed to teamwork and professional development, evidenced by his participation in the GPAC Youth Forum in Oman. He is fluent in both Arabic and English.

Mohammed S. Al Owiemri is a researcher in the Department of Mechanical and Industrial Engineering at *Sultan Qaboos University (SQU)*, Muscat, Oman. His research focuses on the design, modeling, and fabrication of advanced polymer–ceramic composites for biomedical applications. He has contributed to studies on *high-density polyethylene (HDPE) reinforced with micro- and nano-hydroxyapatite (HA) particles*, emphasizing their thermal behavior, compatibility, and performance in additive manufacturing. His work also involves *finite element modeling* and *3D printing* of HDPE/HA composites for biomedical implants. His research interests include additive manufacturing, composite materials, computational mechanics, and materials characterisation. Mohammed's contributions reflect his commitment to integrating computational and experimental approaches to develop innovative, high-performance materials for biomedical and engineering applications.

Moosa S. M. Al-Kharusi* is an Assistant Professor in the Department of Mechanical and Industrial Engineering at *Sultan Qaboos University (SQU)*, Muscat, Oman. He received his BSc, MSc, and PhD degrees in Mechanical Engineering from Sultan Qaboos University, specializing in the nano-mechanics of polymer composites. Before joining SQU, he served as Head of the Department of Mechanical Engineering and Vehicle Technology at the *Global College of Engineering and Technology (GCET)*, Oman, in partnership with the University of the West of England, UK. His teaching and research interests include applied mechanics, computational modeling, finite element analysis, additive manufacturing, and nanocomposite materials. Dr. Al-Kharusi has participated in numerous collaborative research projects, supervised several final-year and postgraduate studies, and published in high-impact journals and international conferences. He is a member of the *Applied Mechanics and Advanced Materials Research Group (AM2RG)* at SQU and has previously been affiliated with the *American Society of Mechanical Engineers (ASME)* and the *Society of Automotive Engineers (SAE)*.