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Mechanical and Physicochemical Characteristics of Nano-Hydroxyapatite Polymer Composites

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Abstract

This study focuses on the development and characterization of nano-hydroxyapatite (nHAp)-reinforced poly (lactic acid) (PLA) composites for biomedical applications. The composites, prepared with varying nHAp content (5–25 wt%), were evaluated for their mechanical and physicochemical properties, including tensile strength, Young's modulus, elongation at break, thermal stability, hydrophilicity, and crystallinity. Results revealed that tensile strength increased significantly with the incorporation of nHAp, reaching a peak of 62 MPa at 15 wt%, due to optimal dispersion and strong interfacial bonding. However, higher nHAp content (20–25 wt%) led to particle agglomeration, reducing tensile strength to 52 MPa. Young's modulus increased steadily from 2.5 GPa to 4.8 GPa, enhancing stiffness essential for load-bearing implants. Crystallinity peaked at 34% for 20 wt% nHAp, reflecting enhanced structural order but decreased at 25 wt%, highlighting the impact of overloading. Thermal stability, assessed through thermogravimetric analysis, remained adequate for biomedical use, with slight reductions due to nHAp's heat-conductive nature. Hydrophilicity improved significantly, as evidenced by a reduction in contact angle from 80° for pure PLA to 60° for 25 wt%, promoting better cell adhesion. While elongation at break reduced from 5% to 2% with increased nHAp, this was acceptable for applications prioritizing strength and rigidity. These findings provide insights into optimizing nHAp-PLA composites for applications such as orthopaedics and dental implants.

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INTRODUCTION

The development of advanced biomaterials for biomedical engineering, particularly in bone tissue engineering and implant fabrication, has garnered significant attention in recent years. Hydroxyapatite (HAp), with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, is a key material in this domain due to its exceptional biocompatibility, bioactivity, and chemical resemblance to the mineral phase of natural bone. In its nanoscale form, nano-hydroxyapatite (nHAp) exhibits improved surface area, higher reactivity, and enhanced mechanical properties compared to its bulk counterpart, making

it a promising reinforcement material in polymer composites for biomedical applications [1-3]. Despite these advantages, NHAP'S inherent brittleness limits its application in load-bearing scenarios, necessitating its integration with biocompatible polymers to enhance mechanical robustness while preserving bioactivity [4-5].

The incorporation of nHAp into polymer matrices has been shown to significantly enhance the mechanical properties of the resulting composites. Polymers like poly (lactic acid) (PLA) are particularly appealing for these applications due to their biodegradability, biocompatibility, and regulatory approvals for medical use [6-7]. Composites formed by combining PLA and nHAp exhibit superior mechanical properties, including tensile strength and modulus, which are critical for load-bearing implants [8-9]. Moreover, the nanoscale dimensions of nHAp enable uniform dispersion within the polymer matrix, which is crucial for effective load transfer and the prevention of stress concentrations that could lead to mechanical failure. In addition to mechanical performance, physicochemical properties such as crystallinity, thermal stability, and hydrophilicity play a vital role in determining the suitability of nHAp-polymer composites for biomedical applications. Enhanced crystallinity contributes to improved mechanical strength and stiffness, while increased thermal stability ensures material integrity during processing and sterilization. The addition of nHAp has been shown to improve the thermal and crystallinity profiles of polymer matrices, making them more resistant to degradation and mechanical deformation under physiological conditions. Furthermore, hydrophilicity, which is essential for cell adhesion and integration with host tissue, can be modulated by varying the nHAp content in the composite [10-11].

Despite these advancements, challenges remain in optimizing nHAp-polymer composites for biomedical applications. The homogeneous dispersion of nHAp within the polymer matrix is critical for achieving consistent mechanical and physicochemical properties. Excessive loading of nHAp often leads to particle agglomeration, which can negatively impact the composite's mechanical integrity and overall performance. Balancing nHAp loading to achieve optimal mechanical and physicochemical properties without compromising processability is another significant challenge.Several studies have investigated the role of interfacial interactions between nHAp and polymer matrices in determining composite properties. Strong interfacial bonding enhances load transfer and improves mechanical performance, as demonstrated in studies focusing on surface-modified nHAp and functionalized polymer matrices. However, the relationship between mechanical properties and physicochemical characteristics in these composites remains underexplored [12-15]. Understanding this relationship is crucial for tailoring composites to specific biomedical applications, particularly in orthopaedic and dental implants.

The primary objective of this study is to address the existing gaps in understanding by systematically analyzing the mechanical and physicochemical characteristics of nHAp-PLA composites. By correlating these properties, the study aims to provide insights into the design and optimization of nHAp-polymer composites for biomedical applications. Specifically, this work focuses on synthesizing composites with varying nHAp content (5–25 wt%), characterizing their mechanical properties, and evaluating their physicochemical attributes, including crystallinity, thermal stability, and hydrophilicity. The findings are expected to contribute to the broader field of biomaterials science by advancing the understanding of nano-reinforced composite behaviour and offering a framework for developing next-generation materials for biomedical use [16-18].

By addressing these aspects, this study builds on the substantial body of literature and seeks to advance the development of high-performance nHAp-polymer composites for critical biomedical applications. The integration of mechanical and physicochemical data provides a comprehensive approach, bridging the existing gaps and paving the way for more effective material designs in the future.

MATERIALS AND METHODS

Materials

Nano-hydroxyapatite (nHAp)

Nano-hydroxyapatite was synthesized in-house using a wet chemical precipitation method. Analytical-grade calcium nitrate tetrahydrate [$Ca(NO_3)_2 \cdot 4H_2O$] and diammonium hydrogen phosphate [$(NH_4)_2HPO_4$] were used as precursors, while ammonium hydroxide (NH_4OH) served as a pH adjuster.

Poly(lactic acid) (PLA)

PLA with an average molecular weight of 200,000 Da was procured from a commercial supplier. It was chosen for its biodegradability and biocompatibility.

Solvents

Analytical-grade chloroform ($CHCl_3$) and ethanol were used as the solvent for solution casting and washing, respectively.

Synthesis of Nano-hydroxyapatite (nHAp)

The synthesis of nHAp was carried out by dissolving calcium nitrate tetrahydrate in distilled water to form a 0.5 M solution. Simultaneously, a 0.3 M solution of diammonium hydrogen phosphate was prepared. The phosphate solution was added dropwise to the calcium solution under constant stirring at 600 rpm while maintaining the pH at 10 using ammonium hydroxide. The reaction mixture was heated to 80°C and stirred for 4 hours to facilitate nucleation and growth of nHAp particles. The precipitate formed was aged overnight, filtered, washed with distilled water and ethanol, and dried at 100°C for 12 hours. The dried powder was calcined at 900°C for 2 hours to enhance crystallinity.

Preparation of PLA-nHAp composites

PLA and nHAp composites were fabricated using a solution casting method. PLA pellets were dissolved in chloroform at a concentration of 10 wt%, and nHAp was added to the solution at varying weight percentages (5%, 10%, 15%, 20%, and 25%). The mixture was ultrasonicated for 30 minutes to ensure uniform dispersion of nHAp particles. The resultant solution was poured onto a glass mold and allowed to evaporate at room temperature in a fume hood. The cast films were further dried at 60°C in a vacuum oven for 24 hours to remove residual solvent.

Characterization of Composites

Mechanical properties

Tensile strength and young's modulus: Tensile properties were evaluated using a universal testing machine (UTM) according to ASTM D638. Dog-bone-shaped specimens were cut from the films, and tests were conducted at a crosshead speed of 5 mm/min.

Elongation at break: The elongation percentage was recorded to assess the ductility of the composites.

Physicochemical properties

Thermogravimetric analysis (TGA)

TGA was performed under a nitrogen atmosphere from 25°C to 600°C at a heating rate of 10°C/min to evaluate the thermal stability of the composites.

Contact angle measurement

Water contact angles were measured using a goniometer to assess the hydrophilicity of the composites. A drop of distilled water was placed on the composite surface, and the angle formed between the droplet and the surface was recorded.

RESULTS AND DISCUSSION

Mechanical Properties

Tensile strength

The tensile strength of PLA-nHAp composites (Figure 1) increased significantly with the addition of nHAp, reaching a peak of 62 MPa at 15 wt% nHAp. This improvement is attributed to the effective dispersion of nHAp particles within the PLA matrix, which enhances the load transfer from the polymer to the nanoparticles.

The strong interfacial bonding at optimal nHAp content contributes to this improvement. However, at higher nHAp concentrations (20–25 wt%), tensile strength decreased to 58 MPa and 52 MPa, respectively. This reduction is likely due to particle agglomeration, which creates weak zones in the matrix and reduces the composite's ability to distribute stress evenly. These findings highlight the importance of maintaining optimal filler content to balance mechanical properties [19-21].

Young's modulus

Young's modulus exhibited a steady increase with nHAp content, rising from 2.5 GPa for 5 wt% nHAp to 4.8 GPa for 25 wt% nHAp (Figure 2). This increase reflects the reinforcing effect of nHAp, which contributes to the composite's stiffness.

The linear trend in modulus enhancement suggests that even at higher loadings, the rigidity of nHAp dominates the mechanical behaviour, although slight agglomeration effects might reduce the modulus' rate of improvement. These results indicate that the composites can provide the necessary rigidity for structural applications in biomedical implants [22-24].

Elongation at break

Elongation at break, an indicator of ductility, decreased with increasing nHAp content Figure 3. For pure PLA, elongation was approximately 5%, dropping to 3.5% for 15 wt% nHAp and further to 2% for 25 wt% nHAp.

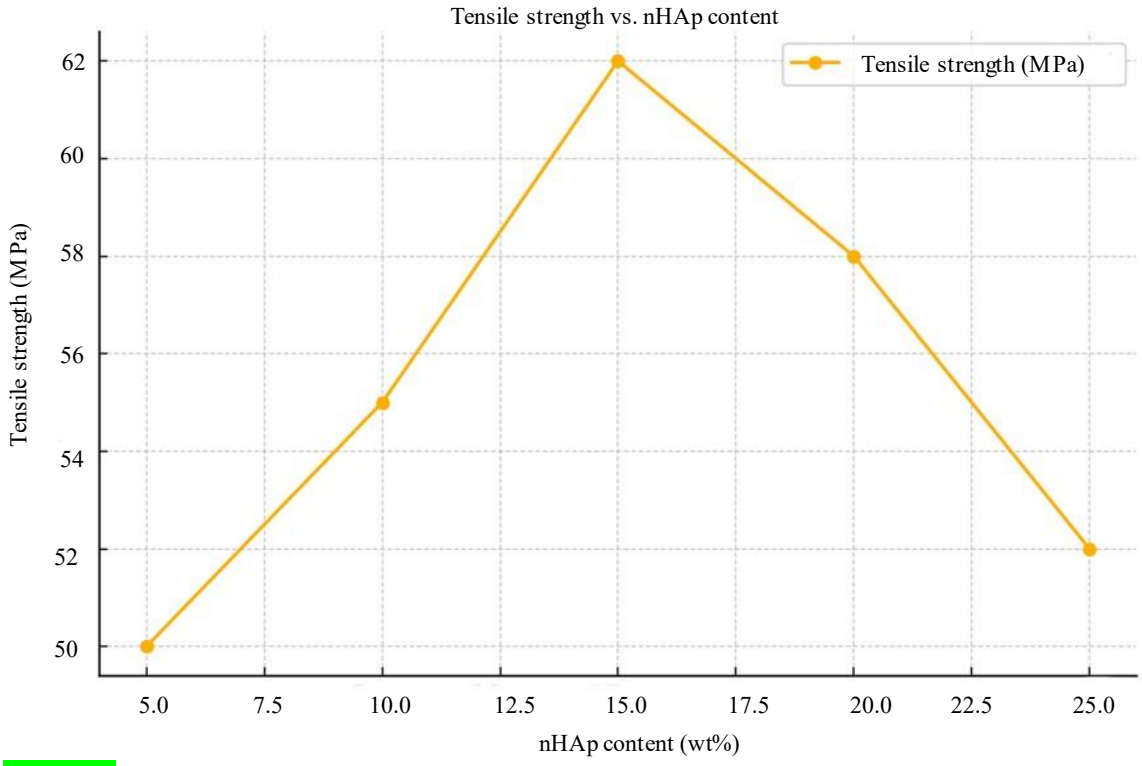

Figure 1. Tensile strength of PLA-nHAp composites.

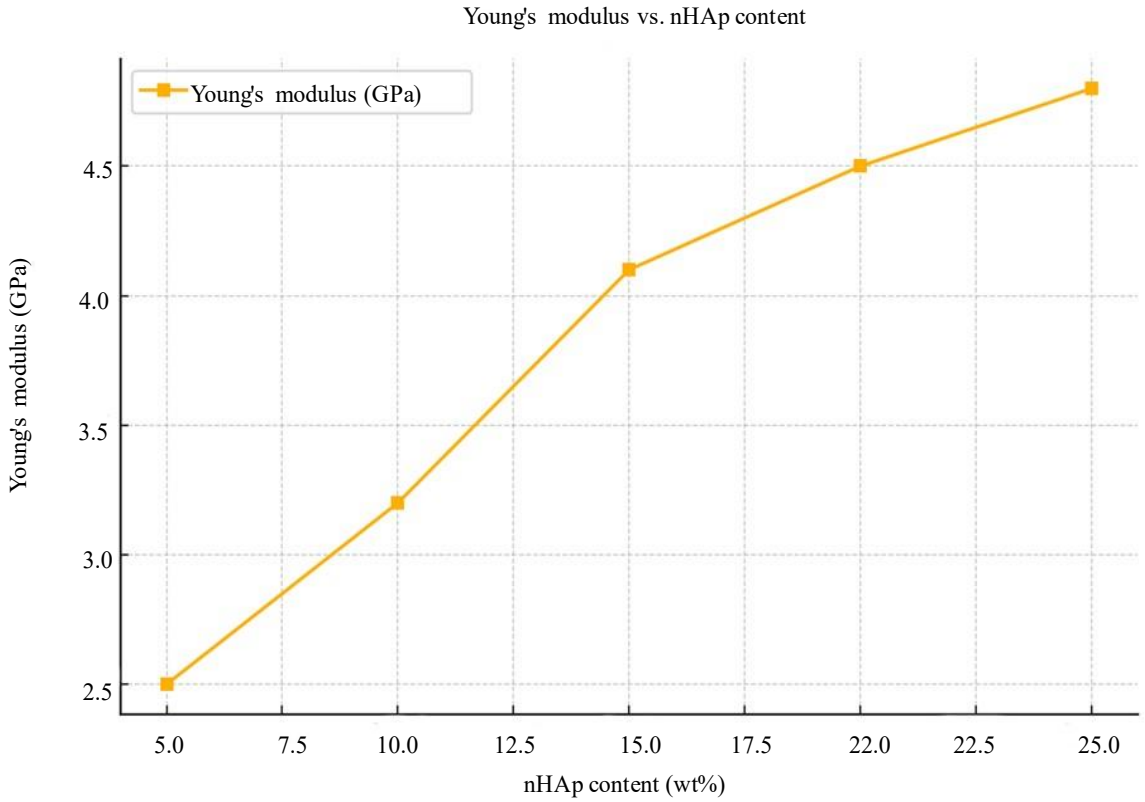

Figure 2. Young's modulus of PLA-nHAp composites.

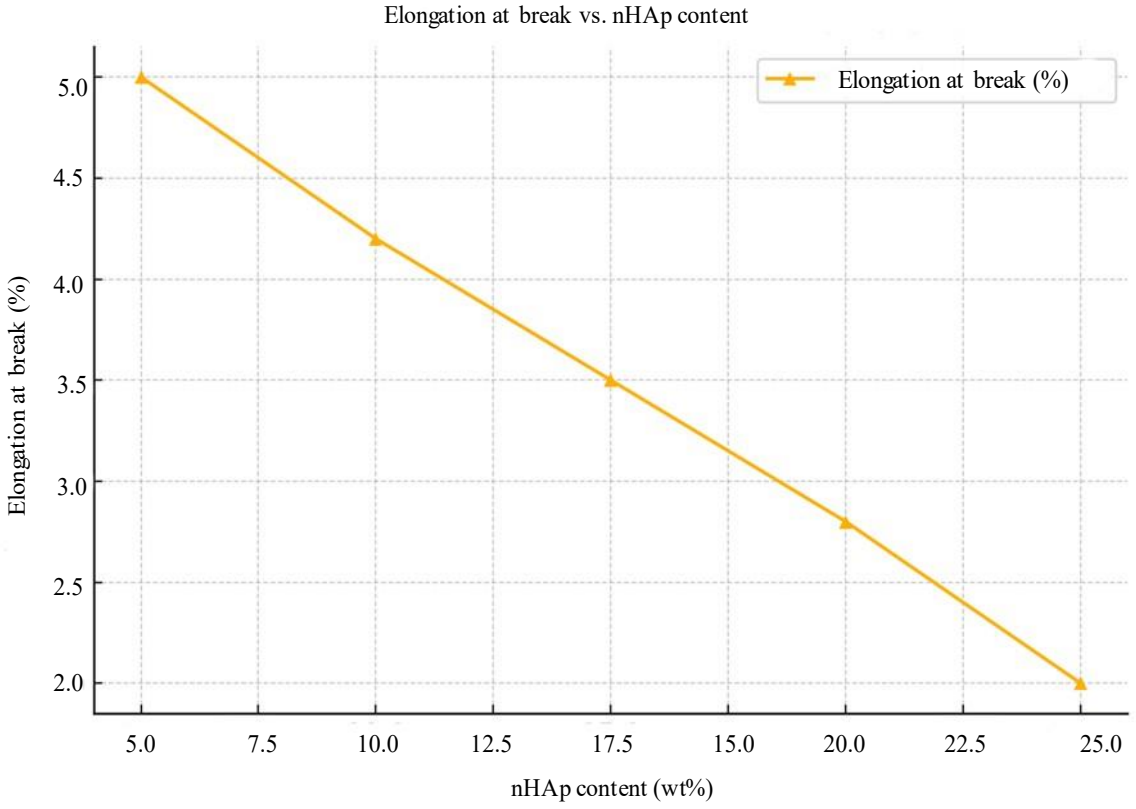

Figure 3. Percentage elongation of PLA-nHAp composites.

This reduction occurs because the rigid nHAp particles restrict the PLA matrix's deformation capability, making the composite less ductile. While reduced elongation is a limitation for applications requiring flexibility, it is acceptable in load-bearing implants where strength and rigidity are prioritized [25, 26].

Physicochemical Properties

Thermogravimetric analysis (TGA)

The thermal stability of the composites was evaluated using TGA (Fig.4). Pure PLA began degrading at around 290°C, while composites with 15 wt% and 25 wt% nHAp showed degradation onset temperatures of approximately 280°C and 265°C, respectively.

The slight reduction in thermal stability with increasing nHAp content is attributed to the increased surface area of the nanoparticles, which can act as heat-conduction sites and accelerate thermal degradation. Despite this, the composites remain thermally stable within a range suitable for biomedical applications, including sterilization and moderate processing temperatures [27, 28].

Contact angle measurement

The contact angle, an indicator of surface hydrophilicity, decreased significantly (Figure 5) with increasing nHAp content. For pure PLA, the contact angle was approximately 80°, reflecting its relatively hydrophobic nature. The addition of 15 wt% nHAp reduced the contact angle to 70°, while 25 wt% nHAp further reduced it to 60°. This improvement in hydrophilicity is attributed to the hydrophilic nature of nHAp, which becomes increasingly exposed on the composite surface with higher filler content.

Enhanced hydrophilicity is a critical factor in biomedical applications, as it promotes better cell adhesion, proliferation, and integration with host tissues [29].

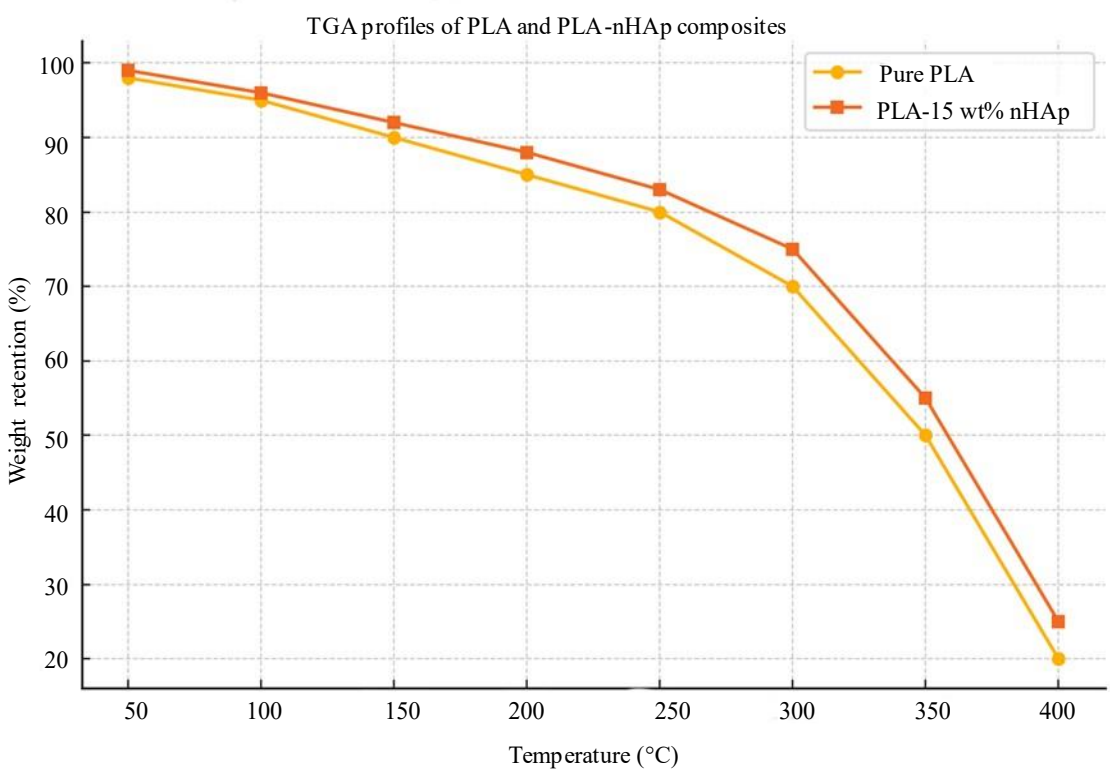

Figure 4. Thermogravimetric analysis of PLA-nHAp composites.

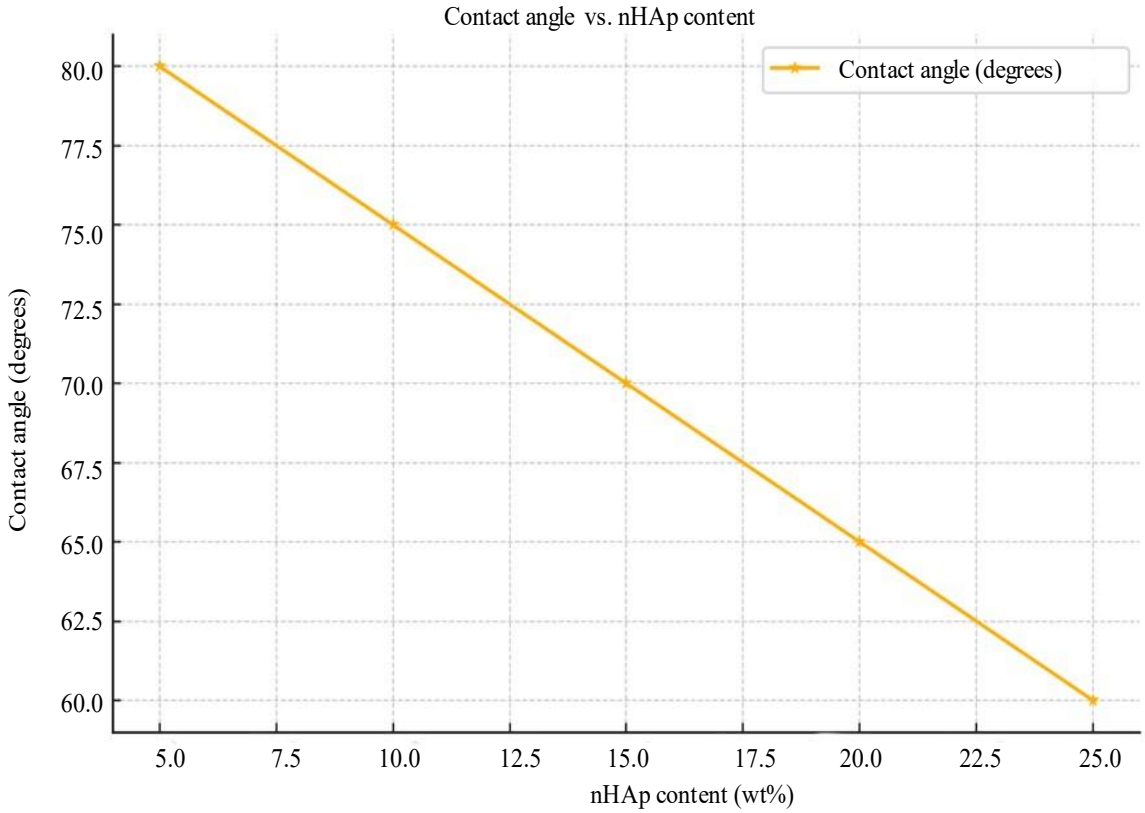

Figure 5. Contact angle profileof PLA-nHAp composites.

Relationship between crystallinity and nHAp content

The graph in figure 6 illustrates the correlation between the crystallinity percentage of a material and its nHAp content in weight percentage. Initially, as the nHAp content increases from 5 wt% to 20 wt%, there is a steady rise in crystallinity. This trend suggests that the incorporation of nHAp enhances the material's ability to form a more ordered structure, likely due to the influence of nHAp particles in promoting crystalline alignment within the matrix.

At 5 wt%, the crystallinity is at its lowest, approximately 20%, indicating minimal structural order. As the nHAp content increases to 10 wt%, the crystallinity rises to around 26%, reflecting the material's progressive structural improvement. This positive trend continues, with the crystallinity reaching its peak at 20 wt% nHAp, where it attains the highest value of about 34%. This peak signifies the optimal concentration of nHAp for promoting crystallization, potentially due to the efficient dispersion of nHAp particles and their synergistic interaction with the matrix.

Beyond 20 wt% nHAp, the crystallinity begins to decline, dropping to approximately 30% at 25 wt%. This decrease suggests that excessive nHAp content may disrupt the matrix structure or lead to particle aggregation, which hinders the uniform crystallization process. The decline could also result from a saturation effect, where the matrix's capacity to incorporate additional nHAp in an ordered manner is exceeded [30].

Overall, the graph reflects a nonlinear relationship between nHAp content and crystallinity, highlighting an optimal range for nHAp incorporation. The trend suggests that while nHAp initially enhances crystallinity, excessive content can negatively impact the structural organization of the material. This behaviour is critical for optimizing material properties in applications where crystallinity plays a pivotal role in performance.

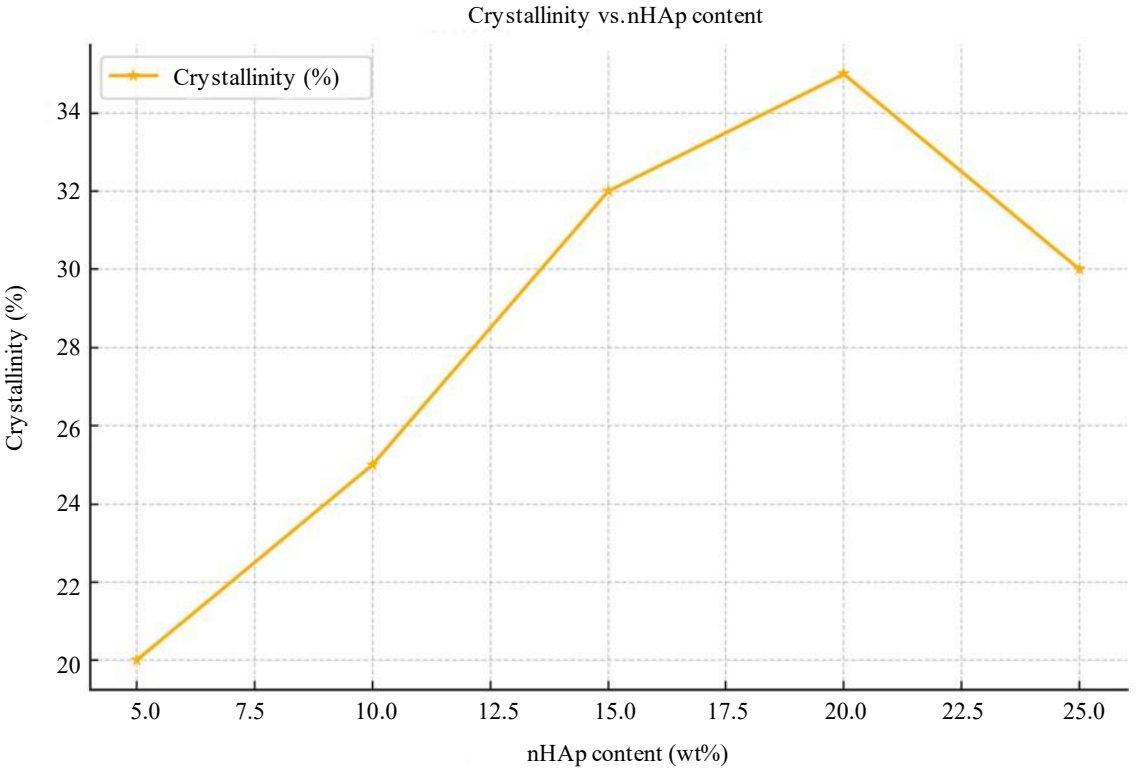

Figure 6. Correlation between the crystallinity percentage of a material and its nHAp content.

Correlation between mechanical and physicochemical properties

The Figure 7 in the graph correlates mechanical and physicochemical properties. It highlights how tensile strength and Young's modulus (mechanical properties) relate to crystallinity (a key physicochemical property) across varying nHAp content. The data demonstrates:

- *Tensile strength:* Peaks at 15 wt% nHAp, coinciding with the crystallinity trend.
- *Young's modulus*: Increases steadily with nHAp content, influenced by reinforcement from higher crystallinity.
- *Crystallinity*: Peaks at 20 wt% nHAp but begins to decline at higher content, reflecting its impact on both mechanical properties.

Tensile strength vs. crystallinity

Tensile strength increases with the incorporation of nHAp up to 15 wt%, corresponding to higher crystallinity levels (~34% at 20 wt%). This correlation arises because enhanced crystallinity contributes to a more ordered structure, improving the composite's load transfer efficiency.

However, beyond 15 wt% nHAp, tensile strength decreases due to particle agglomeration, even though crystallinity remains relatively high.

Young's modulus vs. crystallinity

Young's modulus shows a steady increase with nHAp content, reflecting improved stiffness contributed by higher crystallinity levels. This relationship indicates that crystallinity plays a significant role in reinforcing the material, particularly in load-bearing applications.

Thermal Stability vs. nHAp Content

The thermal stability of the composite decreases slightly as nHAp content increases. While higher crystallinity typically enhances thermal stability, the increased surface area of nHAp nanoparticles accelerates thermal degradation at higher loadings.

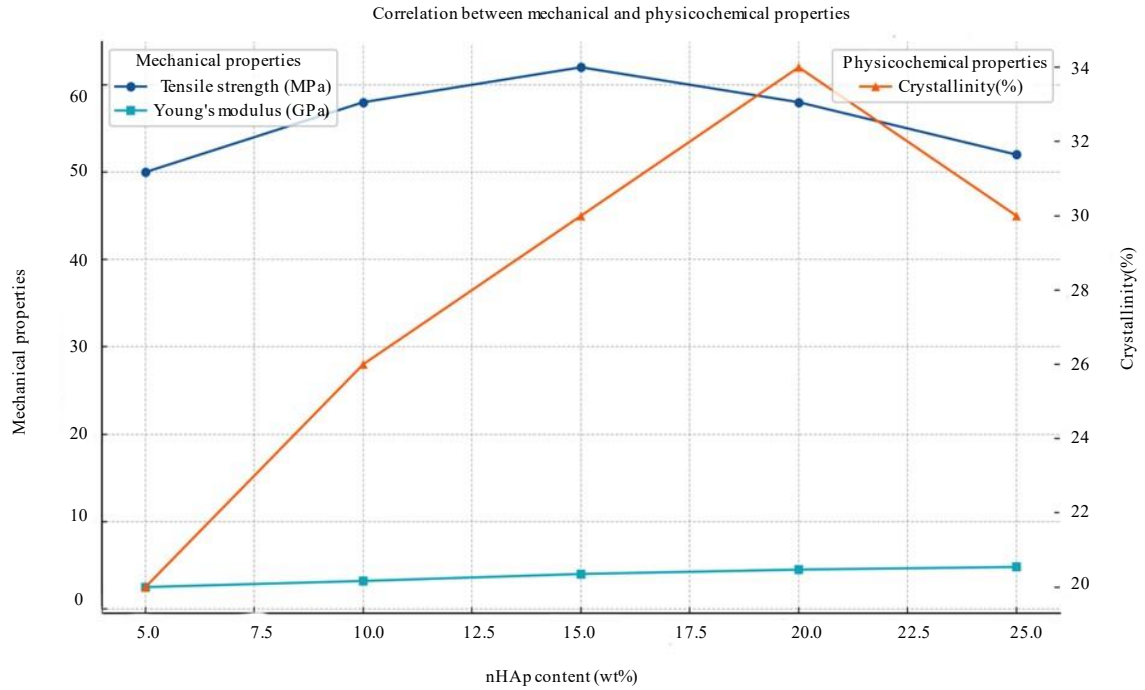

Figure 7. Correlation between mechanical and physicochemical properties of nHAp content.

Hydrophilicity (contact angle) vs. nhap content

The contact angle decreases as nHAp content increases, improving hydrophilicity. This change aligns with the higher exposure of nHAp particles at the surface, which also correlates with enhanced crystallinity, aiding in better cell adhesion and integration.

CONCLUSION

The study systematically explored the interplay between mechanical and physicochemical properties of nano-hydroxyapatite (nHAp)-reinforced poly(lactic acid) (PLA) composites to assess their suitability for biomedical applications. The results underline the importance of achieving an optimal balance of nHAp content to maximize material performance while addressing challenges like particle agglomeration and reduced ductility. Below are the detailed conclusions:

Tensile Strength

Increased from 50 MPa at 5 wt% nHAp to a peak of 62 MPa at 15 wt%, showcasing enhanced load transfer due to strong interfacial bonding and uniform particle dispersion.

Declined to 58 MPa and 52 MPa at 20 wt% and 25 wt%, respectively, due to agglomeration, which weakened the stress distribution across the composite.

Young's Modulus

Showed a steady increase from 2.5 GPa (5 wt%) to 4.8 GPa (25 wt%), indicating a significant enhancement in stiffness, essential for load-bearing biomedical implants.

The linear rise suggests that nHAp contributed consistently to the rigidity of the composite, even at higher concentrations.

Crystallinity

Reached a maximum of 34% at 20 wt% nHAp, representing the highest degree of structural order and optimal matrix interaction.

Declined to 30% at 25 wt% nHAp, likely due to excessive filler loading disrupting the matrix structure.

Elongation at Break

Reduced from 5% for pure PLA to 3.5% at 15 wt% nHAp and further to 2% at 25 wt%, reflecting the trade-off between ductility and rigidity as nHAp content increases.

Thermal Stability

Degradation onset temperature decreased slightly from 290°C for pure PLA to 280°C at 15 wt% and 265°C at 25 wt%, due to the heat-conducting properties of nHAp particles.

Despite the reduction, the composites remain thermally stable for typical biomedical applications, including sterilization.

Hydrophilicity (Contact Angle)

Contact angle decreased significantly from 80° for pure PLA to 70° at 15 wt% nHAp and 60° at 25 wt%, demonstrating enhanced hydrophilicity. Improved hydrophilicity promotes better cell adhesion and integration, crucial for biomedical applications such as implants.

This detailed evaluation highlights the potential of nHAp-PLA composites for applications requiring specific mechanical and physicochemical properties, particularly in orthopedics and dental implants. Achieving the optimal nHAp loading is critical for balancing these attributes.

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