Fabrication and Evaluation of PEEK-NHA Composites Processed via Extrusion and 3D **Printing**

Moinuddin S K¹; Syed Zameer²; Mohammed Mohsin Ali H³

- ¹ Assistant Professor, Department of Mechanical Engineeing, Ghousia College of Engineering, Ramanagaram, Karnataka 562159, India, Affiliated to VTU Belagavi.
- ² Associate Professor, Department of Mechanical Engineering, Ghousia College of Engineering, Ramanagaram, Karnataka562159, India
- ³ Associate Professor, Department of Mechanical Engineeing, Ghousia College of Engineering, Ramanagaram, Karnataka562159, India

Abstract:

Polyetheretherketone (PEEK) is a highthermoplastic increasingly performance investigated for biomedical applications owing to its outstanding mechanical strength, chemical stability, and biocompatibility. Nevertheless, its inherent bioinert nature restricts osseointegration, necessitating reinforcement with bioactive phases. In the present study, nano-hydroxyapatite (nHA) was incorporated into PEEK at 8, 16, and 24 wt.% and processed into composites via extrusion and 3D printing. The mechanical, tribological, and thermal responses of the resulting materials were systematically examined. Wear testing demonstrated that neat PEEK exhibited the lowest wear volume (0.05 mm³ at 2.5 km), while higher nHA contents led to increased wear, attributable to particle pull-out and induced brittleness. In contrast, hardness improved notably with reinforcement, achieving a maximum of 45 VHN at 16 wt.% nHA, signifying an optimum balance between strength and bioactivity. Differential Scanning (DSC) revealed Calorimetry negligible changes in thermal transitions, with Tg remaining stable at 152-153 °C and Tm within 342-345 °C, confirming excellent thermal stability. Collectively, these findings highlight that PEEK-nHA composites, particularly at 16 wt.% loading, provide a superior combination of wear resistance, mechanical reliability, and thermal stability,

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making them highly promising candidates for orthopedic and dental implant applications.

Key words: Polyetheretherketone (PEEK), Nano-hydroxyapatite (nHA), Tribological, Mechanical properties, DSC.

1. Introduction:

A medical term called an implant is something that individuals have created to either replace or aid in the biological macromolecular regeneration of the anatomy of the human body [1]. Because additive manufacturing (AM) techniques, also known as threedimensional (3D) printing, can create complex geometries with less material waste than traditional subtractive manufacturing processes, they are becoming increasingly significant [2]. Complex medical implantable device shapes are frequently challenging to design and manufacturing. Complex implantable medical device design and manufacturing issues can be resolved by 3D printing technology, which can create medical implantable geometries with any complex shape without needing to take manufacturing issues into account [3]. With the chemical formula (-C6H4-O-C6H4-O-C6H4–CO–), Polyetheretherketone (PEEK) is a polyaromatic semi-crystalline thermoplastic polymer. In the 1980s, PEEK was made available to the industry. Invibio Ltd. (Thorton-Cleveleys, U.K) presented it as a material for biological application in 1998 [4]. Because of the weak interfacial bonding the

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surface modification layer, which typically tiny in size, was prone to peeling off from the matrix in the complex loading environment. Even if bioactivity is increased, the addition of bioceramics has some effect on composites' overall mechanical characteristics. The study Created PEEK/HA composite 3D-printed filaments with varying mass percentages by melt extrusion and physical mixing techniques, and assessed the mechanical properties of the fabricated 3Dprinted samples. When the nHA content was increased to 30% weight, the PEEK/HA composite's tensile modulus improved by 68.6% in comparison to the mechanical properties of PEEK, while its tensile strength declined by 48.2%. Examined how material formulation affected the mechanical properties of 3D-printed PEEK / HA composites. PEEK filaments with 10 weight percent nHA showed slight reduction in tensile approximately (14%)and modulus elasticity approximately (5%) when compared to pure PEEK [5]. However, adding short or continuous fibers to a thermoplastic matrix to modify composite materials has garnered a lot of interest and greatly enhances their mechanical qualities [6]. A three Dimensional printed PEEK object produces a thick, stiff, and chemically stable specimen in a few hrs, and the option of AM has significantly expanded the use of PEEK for customizable parts across many sectors [7]. Additionally, a broad range of unique mechanical, surface, and physical properties can be easily designed into PEEK composites. In particular, PEEK is currently being used to fabricate spinal implants with success [8]. PEEK is an alternative to metallic alloys such as Ti alloys and SS. Its mechanical properties, including tensile strength and modulus of elasticity, are familiar to those of human Consequently, Many researchers have worked on increase PEEK's bioactivity, which will support the long-term stability biocompatibility of implants and host tissues at the interface, in order to utilize the use of PEEK in orthopedic applications. following three elements are among the primary techniques employed: 1) PEEK

surface activation; 2) bioactive material mixing; and 3) porous outer layer morphology preparation. PEEK surface activation is often accomplished by applying materials like hydroxyapatite (HA) on the outer layer of PEEK implants [09]. The interest in developing manufacturing processes, like surface changes, to improve the biological and mechanical responses of PEEK samples has been highlighted by their good performance. Many surface modification methodologies been studied enhance have biocompatibility of PEEK. Although they can also impact hydrophobicity and surface integrity, techniques like plasma treatment and Sulfonation can change surface characteristics [10]. PEEK has a comparatively low modulus of elasticity (Ti: 102 to 110 GPa; PEEK: 3 to 4 GPa), which is nearer to human bone (14 GPa) than metals like Ti [11]. High strength, high modulus, high melting point, resistance to corrosion. and superior processing performance are all attributes of PEEK, a specifically developed plastic. Additionally, in a different range of pressures, temperatures, speeds, and relative roughness contact situations, it offers exceptional wear resistance [12]. To increase the biocompatibility of PEEK material, for example, a porous hydroxyapatite (HA) scaffold is first printed using FDM, and then semisolid PEEK is squeezed into the Hydroxyapatite scaffold to create a PEEK-HA composite [13]. PEEK polymers typically have a modulus of elasticity that is close to that of bone than metals, which reduces the stress shielding effect of metals like SS that are used for fixation or arthroplasty fracture $\lceil 14 \rceil$. Furthermore, they avoid the detrimental effects of releasing metallic ions into the tissues of the body, which could result in immunological reactions and osteolysis [15]. PEEK reinforced with hydroxyapatite (HA) was fabricated with varying HA content from 0% to 30% in steps of 10%. SEM results confirmed a nearly homogeneos dispersion of reinforcement in the PEEK matrix. For samples with 30% HA, the modulus of elasticity showed an increase of about 68.6% compared to PEEK, while the tensile strength

reduce by 48.2%. After optimizing the printing parameters, it was observed that improvement in mechanical properties was achieved at a 0° printing orientation [16]. Due to its bioinert nature, PEEK fails to promote osteogenesis and apatite layer formation. It also lacks antibacterial and anti-inflammatory properties. Because of these limitations, the direct use of pure PEEK in clinical trials is not permitted. However, these properties can be impacted by adding reinforcement materials such as bioceramics [17].

PEEK reinforced with Ti6Al4V, fabricated using powder bed fusion for heavy loading applications. exhibits good mechanical properties such as Tensile strength, fatigue resistance, and stiffness. It also shows strong interfacial bonding between the reinforcement and the matrix. Notably, the composite demonstrates higher compressive and flexural strength compared to pure PEEK [18]. FEA/FEM analysis for skull repair indicates that PEEK reinforced with Ti alloys exhibits resistance to dynamic loading conditions, closely matching the mechanical response of human skull bone [19].

2. Experimental Section Nano composite filament fabrication

High-performance polymer materials such as Polyetheretherketone (PEEK), have been intensively used in various industrial and biological fields. The reinforcement of PEEK with bioactive (nano hydroxyapatite, nHA) ceramic has been developed to enhance the mechanical-bioactive properties[18].

2.1 Pre- Treatment of nHA

Treating of the nHA powder with 15 percent strength salinity was done to increase interfacial bonding between reinforcement and matrix. This treatment is meant to enhance the mechanical properties of the composites through the creation of bond of adhesion between the nHA particles and PEEK matrix [16]. This saline treatment assists in eliminating any type of impurities on the surface of nHA particles and generates a more homogeneous surface to be bonded by the PEEK matrix. The mixture was filtered after

24 h using filter paper; the nHA powder was then rinsed in distilled water to eliminate any residue of saline [06].

2.2 Mixing of PEEK and nHA

The dried nHA powder was subsequently mixed with PEEK powder in a V-type blender over 1 hr until homogeneity was reached between the reinforcement and the matrix. The mixing guarantees homogeneity in PEEK composite that is essential for the uniformity of the mechanical properties [13].

2.3 Extrusion Process

The hopper then had the mixed powder fed to a twin extruder. The extruder rate was held at 20 rpm and four temperature sensors were applied to monitor the temperature during the process [18].

PEEK melting point temperature is about 400°C, and extrusion process was designed in such a way that the temperature exceeded 400°C to properly melt and mix the PEEK and nHA. Then, this extruded material was permitted through a nozzle and the filament was cooled with a cooling agent (water). This was broken down into filament pellets.

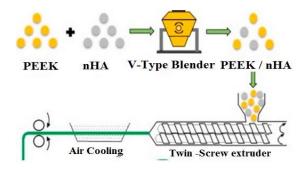


Fig 2.1. Mixing of PEEK and nHA using v type blender and filament fabrication using Twin Extruder [16].

2.5 Filament Formation:

The pellets were then passed through a single extruder, and the temperature was maintained between 370-440°C. The resulting filament was then wrapped around a pulley.

2.6 Finished Product

The finished product is a filament composed of PEEK and nHA. The filament with a uniform dia of 1.75 mm, showing that the extrusion process was successful [16]. The mechanical strength of the composite filament will be determined in future studies to find its suitability for various applications.

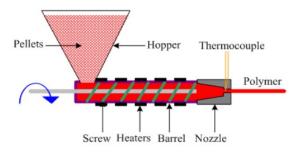


Fig 2.2. Finished fabricated filament of 1.75 mm diameter using single extruder [18]

2.7 Wear and Friction Test Specimen specification:

The ASTM D3039 specimen is a standard test method for measuring the wear resistance of PEEK and nHA composites. The specimen is designed to evaluate the friction and Wear resistance of composite material. Pure PEEK indicates superior wear resistance compare to its composites [18]. Incorporation of nano Hydroxyapatite with PEEK has significantly modified the Tribological properties of PEEK [17].

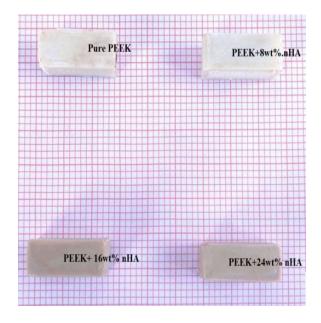




Fig 2.3. Wear Specimens of PEEK+nHA (0%,8%,16%,24%) Composites. Fig 2.4. Pin on Disc Wear machine [40]

Wear test was performed using pin on disc machine with disc diameter of 165mm x 8mm thick, EN-31 hardened to 60 HRc, ground to surface roughness 1.6 Ra. Wear is a critical factor contributing to biomaterial failure. In this study, the wear test was conducted by considering the properties of simulated body fluid. Carboxymethyl cellulose solution was adopted as a lubricant given its similarity to synovial body fluid and its wide availability [15]. Dimensions of the specimens are 18mm x 4 mm x 30 mm. The specimens were soaked in a Carboxymethyl cellulose solution with a viscosity of 0.03 poise for 48 hours, with the solution's pH at 7.3, which is similar to the pH of synovial body fluid. For sterilization samples were then immersed in formaldehyde solution for a period of 24 hrs. The samples were then cleaned and rinsed with double-distilled water [11]. The applied load on the composite specimens was determined based on the Hertzian contact pressure that occurs between the hip joint head and acetabular cup under various human physical activities, as reported [14].

2.8 Hardness Specimen

Hardness is the crucial property for biomaterials for ability to withstand

indentation and plastic deformation. In accordance with expectations, the HA, and HA-bioactive glass coating layers exhibits greater hardness values than that of the PEEK substrate due to the ductile nature of this polymer. It was noted that the HA-bioactive glass coating layers exhibited higher hardness values than the HA layer [10].

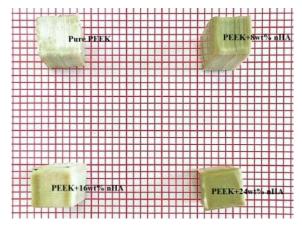




Fig 2.8. Hardness Specimens of PEEK+nHA Fig 2.9. Shimadzu Micro Hardness tester [15]. (0%,8%,16%,24%) Composites.

2.12 Differential Scanning Calorimeter [DSC].

Differential Scanning Calorimetric (DSC) is a thermo analytical method that asses the heat flow into or out of a material as it is heated, cooled, or held isothermally. It is largely used to investigate polymers, composites, ceramics, and biomaterials as it offers understanding of thermal transitions and crystallinity [11]. Glass Transition Temperature (Tg): where the amorphous regions of the polymer change

from a hard, glassy state to a soft, elastic state. For PEEK, Tg is usually observed at about 150–160 °C. Melting Temperature (Tm): where crystalline regions melt, identified as a sharp endothermic peak. For PEEK, Melting temperature is around 340–345 °C. DSC helps to evaluate how nano-hydroxyapatite (nHA) affects chain mobility, nucleation, and crystalline [10]. PEEK is semi-crystalline; addition of nHA may either increase crystallinity (nucleating effect) or decrease it (particle agglomeration disrupting order).

3. Results and Discussions

3.1 Wear and Friction behavior of PEEK and PEEK/nHA Composites.

Wear resistance is another critical property for biomedical applications, especially for loadbearing implants. Wear tests were conducted using a pin-on-disc setup. The results of wear volume as a function of sliding distance for different PEEK compositions are shown below.

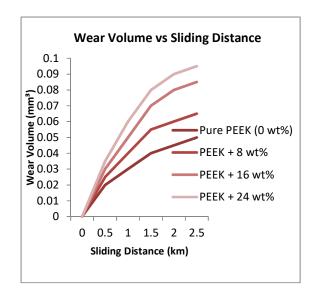


Fig. 3.1: Wear Volume vs. Sliding Distance for PEEK and PEEK/nHA composites.

Table 3.1: Wear volume vs. sliding distance

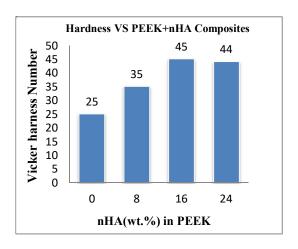
Sliding	Pure	PEEK	PEEK	PEEK
Distance	PEEK	+ 8	+ 16	+ 24
(km)	(0	wt%	wt%	wt%
	wt%)			
0.0	0.0	0.0	0.0	0.0
0.5	0.02	0.025	0.03	0.035

1.0	0.03	0.04	0.05	0.06
1.5	0.04	0.055	0.07	0.08
2.0	0.045	0.06	0.08	0.09
2.5	0.05	0.065	0.085	0.095

The graph depicts the relationship between wear volume (mm³) and sliding distance (km) for pure PEEK and PEEK composites reinforced with different nHA contents (8, 16, and 24 wt.%). X-axis: Sliding distance (km), ranging from 0 to 2.5 km. Y-axis: Wear volume (mm³), ranging from 0 to 0.1 mm³.Pure PEEK exhibits the lowest wear volume throughout the sliding distance, reaching about 0.05 mm³ at 2.5 km [08]. This indicates that neat PEEK, being a tough polymer with good self-lubricating properties, offers better wear resistance compared to its composites. Its ductile nature helps in absorbing contact stresses and reducing material removal under sliding conditions. The addition of 8% nHA increases the wear volume compared to pure PEEK, ending at around 0.065 mm3 at 2.5 km. While nHA particles enhance hardness, they can also increase surface roughness and act as abrasive third bodies during sliding, thereby increasing material loss. The interfacial bonding at lower reinforcement levels may not be strong enough prevent particle pull-out, to contributing to wear. The 16% nHA composite shows even higher wear, with wear volume reaching nearly 0.085 mm³. Although this composition showed the highest hardness in the earlier graph, excessive brittleness introduced by ceramic reinforcement reduces the material's wear resistance [10].

3.2 Hardness Behavior of PEEK and PEEK/nHA Composites.

The hardness of biomaterials is crucial for their resistance to indentation and plastic deformation. composites, For PEEK reinforcement with nHA significantly Vickers influences hardness values. The Hardness test results for different compositions are shown below.



SI No	Percentage of Reinforcement	Vickers Hardness (HV)
01	Pure PEEK (0 wt%)	25
02	PEEK + 8 wt%	35
03	PEEK + 16 wt%	45
04	PEEK + 24 wt%	44

Table 3.2: Hardness of PEEK and nHA Fig 3.2. The influence of nHA on Vickers, and it nano-composites

The bar chart represents the variation in Vickers Hardness Number (VHN) of PEEK and its composites reinforced with different percentages (wt.%) of hydroxyapatite (nHA). The x-axis shows the nHA content in PEEK (0, 8, 16, and 24 wt. %), while the y-axis shows the corresponding Vickers Hardness Number. The hardness value of neat PEEK is about 25 VHN. This relatively low value indicates the inherently softer nature of PEEK as a polymeric material. toughness While has good biocompatibility, its hardness alone insufficient for load-bearing biomedical applications like orthopedic implants [19]. When 8% nHA is added, the hardness increases significantly to around 35 VHN. This rise is due to the incorporation of hard ceramic nHA particles within the polymer matrix, which improves the resistance of the composite to localized plastic deformation. At 16% reinforcement, the hardness reaches its maximum value of 45 VHN. This optimum improvement is attributed to the uniform dispersion of nHA particles in the PEEK matrix, leading to effective load transfer, better interfacial bonding, and enhanced resistance to indentation [07]. The synergistic effect between the polymer and the ceramic reinforcement makes this composition highly suitable for biomedical applications requiring both strength and biocompatibility. At 24% nHA loading, the hardness slightly decreases to about 44 VHN. Though the hardness remains higher than pure PEEK and 8% nHA composites, the marginal reduction compared to 16% suggests that higher nHA content may cause particle agglomeration and micro structural in homogeneity [02].

3.3 Differential Scanning Calorimeter [DSC].

The Differential Scanning Calorimetric (DSC) analysis of pure PEEK and its composites reinforced with 8, 16, and 24 wt. % of nanohydroxyapatite (nHA) revealed that the incorporation of nHA did not significantly alter the thermal transition behavior of the polymer matrix.

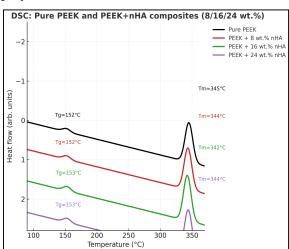


Fig 3.3. DSC graph for PEEK and its composites.

The glass transition temperature (Tg) of pure PEEK was observed at approximately 152°C, and a similar value was recorded for the 8 wt. % composite, while the 16 wt. % and 24 wt.% composites exhibited a slight increase to

153°C. This indicates that the addition of nHA particles has minimal influence on the segmental mobility of polymer chains, thereby maintaining the amorphous flexibility of PEEK [01]. The melting temperature (Tm) of pure PEEK was measured at 345°C, whereas the composites displayed values in the range of 342-344°C. A slight reduction to 342°C was observed at 16 wt.% nHA, which may be attributed to disruptions in crystalline packing due to particle-matrix interactions, while a marginal increase to 344°C at 24 wt.% suggests a possible nucleating effect of nHA in promoting more stable crystalline regions [05]. Overall, the results confirm that the addition of nHA up to 24 wt. % does not significantly affect the crystalline amorphous transitions of PEEK, and both Tg and Tm remain within a narrow and stable range [04].

4. Conclusion

- It was found that the saline treatment of nHA powder enhances interfacial bonding with the PEEK matrix, improving the mechanical properties.
- The PEEK and nHA powders were blended in a V-type blender to achieve homogeneous mixing, ensuring uniform mechanical properties in the composite.
- The PEEK/nHA composite was successfully extruded at varying temperatures, ensuring proper melting and mixing of the materials, and then pelletized for further processing.
- The extrusion process successfully produced PEEK/nHA filament with a uniform dia of 1.75 mm and smooth surface, paving the way for future evaluation of its mechanical properties.
- Uniform filament with a diameter of 1.75 ± 0.05 mm was successfully created using a 3devo Composer 450 desktop extruder. Various extrusion parameters were adjusted to achieve consistent diameter. Manual powder feeding was necessary due to the powder's low density. Optimization of parameters resulted in improved filament uniformity,

- Using the Specialized 3D printer we have fabricated composite PEEK/nHA samples for different tests, including Friction and Wear, Vickers Micro hardness, Differential Scanning Calorimeter [DSC], These samples will be used to evaluate the tribological, mechanical, chemical and thermal behavior of PEEK composites.
- In conclusion, pure PEEK exhibited the lowest wear volume of 0.05 mm³ at 2.5 km, confirming its superior wear resistance compared the composites. to incorporation of 8 wt. % nHA increased the wear volume to 0.065 mm³, while 16 wt. % and 24 wt. % nHA composites showed even higher values of 0.085 mm³ and 0.095 mm³, respectively, due to particle pull-out, agglomeration, and increased brittleness. Although these reinforcements enhanced hardness, they compromised tribological performance under sliding conditions. Hence, optimization of nHA content is necessary to balance bioactivity with acceptable wear resistance for biomedical applications.
- In conclusion, pure PEEK showed the lowest hardness of 25 VHN, confirming its softer nature as a polymeric material. The addition of 8 wt. % nHA increased the hardness to 35 VHN, while the 16 wt.% composite exhibited the maximum value of 45 VHN due to uniform particle dispersion and improved interfacial bonding. At 24 wt. % nHA, the hardness slightly decreased to VHN. likely from agglomeration and micro structural defects. Thus, 16 wt. % nHA reinforcement provides the optimal balance of strength and biocompatibility, making it the most suitable composition for biomedical applications.
- In conclusion, pure PEEK exhibited a Tg of 152°C, which increased slightly to 153°C for 16 wt.% and 24 wt.% composites, showing minimal effect of nHA on chain mobility. The Tm of pure PEEK (345°C) varied slightly between 342–344°C in the composites, with a small reduction at 16 wt.% and a marginal rise at 24 wt.%. These minor changes confirm that nHA addition

up to 24 wt.% does not significantly affect thermal transitions. Hence, PEEK–nHA composites retain excellent thermal stability for biomedical applications.

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