

Tribological Properties and Biocompatibility Studies of PEEK and Its Composites

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Abstract

Polyetheretherketone (PEEK) is a high-performance polymer widely explored for biomedical applications due to its excellent mechanical strength, chemical stability, and biocompatibility. However, its bioinert nature limits osteointegration, requiring reinforcement with bioactive ceramics. In this study, PEEK was reinforced with nano-hydroxyapatite (nHA) at 8, 16, and 24 wt.% and fabricated into composites using extrusion and 3D printing. The tribological, thermal, chemical, and biological properties of the composites were systematically evaluated. Wear tests revealed that pure PEEK exhibited the lowest wear volume (0.05 mm³ at 2.5 km), whereas increasing nHA content led to higher wear due to particle pull-out and brittleness. Hardness improved significantly with reinforcement, peaking at 45 VHN for 16 wt.% nHA, indicating an optimal balance between strength and bioactivity. DSC analysis confirmed minimal changes in thermal transitions, with T_g maintained at 152–153°C and T_m within 342–345°C, demonstrating excellent thermal stability. FTIR spectra verified the successful incorporation of nHA without altering PEEK's backbone chemistry, while corrosion tests showed an increase in current density at 8 wt.% (0.15 μA/cm²) followed by stabilization at higher filler levels. SBF immersion studies demonstrated progressive apatite layer formation on composites, especially at 16 and 24 wt.% nHA, confirming enhanced bioactivity. Overall, the results establish that PEEK–nHA composites, particularly at 16 wt.% reinforcement, offer an optimal combination of mechanical strength, wear resistance, thermal stability, and bioactivity, making them promising candidates for orthopedic and dental implant applications.

Keywords: Polyetheretherketone (PEEK), Nano-hydroxyapatite (nHA), Tribological properties, DSC, FTIR, SBF, Biocompatibility

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 three-dimensional (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 surface modification layer, which was 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 the 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 3D-printed 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 a slight reduction in tensile stress approximately (14%) and modulus of 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 bone.

Consequently, Many researchers have worked on increase PEEK's bioactivity, which will support the long-term stability and biocompatibility of implants and host tissues at the interface, in order to utilize the use of PEEK in orthopedic applications. The 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 have been studied to enhance the 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 fracture fixation or arthroplasty [14]. 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 homogeneous 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 were 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 good resistance to dynamic loading conditions, closely matching the mechanical response of human skull bone [19]. Porous implants fabricated by additive manufacturing promote bone growth due to their porous architecture. Heat treatment at 240 °C for 2 hours increases the crystalline of PEEK up to 37.2%, resulting in improved strength and stiffness. However, more crystallinity may lead to brittle fracture [20]. Nano-layers of black chitosan sheets coated on three-dimensional printed PEEK can enhance osteogenesis. Additionally, they release reactive oxygen species that effectively eliminate E coli

bacteria, thereby reducing the risk of post-operative infections. In vivo tests in animals have confirmed improved bone regeneration [21]. The formation of a fibrous capsule and fibrous tissue remains a major challenge, as it leads to a mismatch between the PEEK composite and the surrounding bone, ultimately resulting in implant failure. This study summarizes the immune response of PEEK implant [22]. The use of customized screw-based extrusion 3D printing results in improved mechanical behavior. A compound composite of PEEK/HA/CF fabricated using FFF techniques showed a Highest tensile strength of 115 MPa, which falls well within the range of human bone [23]. Specimens with cross-path patterns exhibited inferior compressive strength due to fabrication defects at the joints of two deposition layers. By using non-crossing patterns such as square and triangular designs, the strength can be increased by 12.5% and 18.4%, respectively [24]. Experiments on cranial defects in rabbits showed significant acceleration in bone regeneration [25]. In vivo and in vitro results reveal that PEEK/nHA composites promote osteogenesis and bone tissue regeneration [26]. 3D printing enables the manufacturing of complex and customized geometries with fewer flaws [27]. Numerical simulations confirm that the compressive strength and modulus of elasticity of PEEK composites closely match those of cranial bone. It can be concluded that PEEK reinforced with CF, GF, and Ti alloys exhibits good biocompatibility [28]. In vitro trials show that biomineralization in PEEK-CAP bioceramic cages regulates immunity and promotes bone regeneration. In vivo fusion experiments in goats further confirm that PEEK exhibits good biocompatibility, bioactivity, and bone fusion capability [29]. Sulfonation in the range of 30–45 seconds facilitates the formation of uniform micro scale pores on the surface of 3D-printed specimens and simultaneously influences both their biological and mechanical properties; Sulfonation also enhances adhesion between bone and surrounding tissue, as confirmed by in vivo tests [30]. The influence of parameters such as HA content, printing direction and pore size determines the values of elastic modulus and compressive strength. Moreover, samples printed in the Z-direction exhibit significantly lower compressive strength [31]. PEEK/nHA composites with concentrations ranging from 0–40 wt% demonstrate significantly improved cell adhesion, proliferation, and mineralization. Micro-CT analysis confirms enhanced bone growth, with an ingrowth volume of 24.3% observed for 40% nHA after 12 weeks. Overall, the composite exhibits higher bone growth compared to pure PEEK [32]. The 3D printing process of PEEK is difficult due to its high melting temperature of around 340 °C. Mechanical testing revealed a reduce in tensile strength of up to 14%, while the elastic modulus increased by 5%. In vitro tests conducted for 28 days confirmed formation of an apatite layer on the surface of the specimens [33]. 3D printing for biotechnology remains challenging due to the lack of angiogenesis, Exosomes extracted from bone marrow stem cells (BMSCs) show its ability to promote angiogenesis [34]. PAEK has the ability to support bone regeneration, and due to its extraordinary properties, it has been widely used in different fields such as orthopedics, craniofacial reconstruction, cardiac surgery, and dentistry [35]. 3D printing of PEEK is an innovation that has provided solutions for complex anatomical challenges [36]. PEEK is a polymer widely used in tribological applications, where various reinforcements such as SiC, Al₂O₃, WS₂, and ZrO₂ are incorporated to enhance its wear resistance [37]. Filament manufacturing using a twin-screw extruder is the widely used process due to its cost effective and adaptability, even with dissimilar metals [38]. PEEK as a more versatile tribological material for a wide range of applications [39]. Wear volumes of fabricated samples decreases with increase in the reinforcement [40].

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[32]. Multiple processes go into the manufacturing of PEEK and nHA composites such as nHA pre-treatment, PEEK and nHA mixing, extrusion, and filament production[33]. In this article we provide a detailed description of the PEEK/nHA composite manufacturing process.

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 generate a more

homogeneous surface to be bonded by the PEEK matrix. The nHA powder with 15% concentration of saline was placed in a beaker and stirred for 30 minutes to provide uniform mixing. The mixture of the powder was then left to settle overnight (24hrs) to enable the saline to permeate into the nHA particles. 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 [33].

PEEK and nHA powder were mixed in ratio (8, 16, and 24 wt %) then allowed to mix thoroughly 1h at 100 rpm. The mixture was later left to cool for 30 minutes allowing powders to settle as well as having a proper mixing.

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 [38]. The temperature. Sensor values were the following:

For Pure PEEK

i. Sensor 1: 365°C ii. Sensor 2: 375°C iii. Sensor 3: 385°C iv. Sensor 4: 410°C

For PEEK/8%HA

i. Sensor 1: 380°C ii. Sensor 2: 390°C iii. Sensor 3: 400°C iv. Sensor 4: 430°C

For PEEK/16%HA

i. Sensor 1: 380°C ii. Sensor 2: 395°C iii. Sensor 3: 420°C iv. Sensor 4: 435°C

For PEEK/24%HA

i. Sensor 1: 390°C ii. Sensor 2: 400°C iii. Sensor 3: 420°C iv. Sensor 4: 440°C

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.

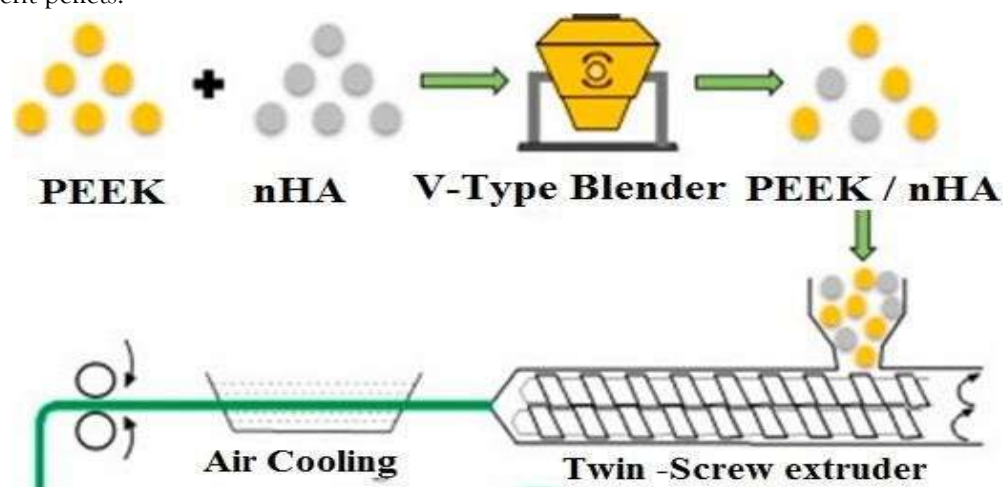


Fig 2.1. Mixing of PEEK and nHA using v type blender and filament fabrication using Twin Extruder [16]

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, 38]. The mechanical strength of the composite filament will be determined in future studies to find its suitability for various applications.

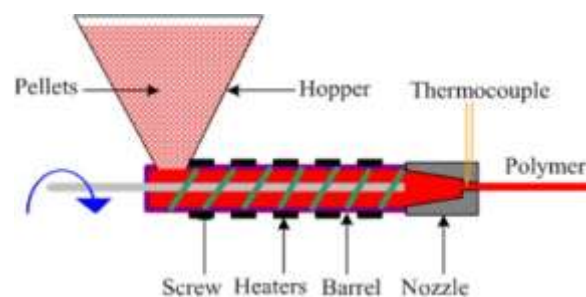


Fig 2.2. Finished fabricated filament of 1.75 mm diameter using single extruder[38]

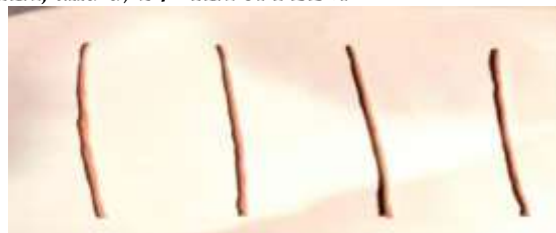
2.7 Optimization of Filament:

As seen in Figure 2.3, filaments with 1.75 ± 0.05 mm of diameter were fabricated using dry powder combinations and a commercial desktop extruder, the 3devo Composer 450. Several extrusion parameters, including puller wheel speed, nozzle diameter, temperature of various zones, cooling rate, powder feeding rate, and extrusion speed, were adjusted to create filaments with uniform diameters. Each parameter can have an impact on the finished diameter of the filaments, either positively or negatively. For example, increasing the powder feed rate, extrusion velocity and higher cooling rate increase the final diameter, whereas raising the puller wheel speed reduces it. A hopper was used to feed the powder into the extruder. However, the powder did not enter the extruder on its own because of its low density. As a result, the powder was manually supplied at a steady and continuous rate. Improper or uneven powder feeding resulted in filaments with non-uniform diameters. Figures 2.3 and 2.4 below display the filaments that were formed during and after optimization, respectively [33].

Fig 2.3 PEEK nanocomposites filaments with an uneven diameter that were produced during Optimization using a 3devo desktop extruder.



Fig 2.4 Filaments made of PEEK nanocomposites were optimized using a 3devodesktop extruder. a) PEEK/0% b) 8%HA, c) 16%HA, and d) 24%HA of PEEK



Furthermore, since the viscosity of the melt depends on both temperature and filler content, precise temperature control was essential. The feed powder allows through 4 distinct heating zones of the extruder, designated H1–H4. Each heating zone's temperature was carefully adjusted. The H-4 heating region was near the nozzle where the molten material exited the extruder, while the H-1 heating region was near the powder feeding hopper. To ensure that the powder had the proper viscosity when it emerged from the nozzle, the

temperature of the H-1 heating region was initially kept slightly more than the powder's melting point and then progressively raised. When the H1 region temperature was excessively high ($\approx 370^\circ\text{C}$), tiny air bubbles were visible in the extruded filaments. The extruder's nozzle was angled downward, allowing material to flow smoothly under the influence of gravity [33, 38].



Fig 2.5 Extrusion procedure for filament production: (a) desktop extruder, (b) molten polymer exiting the nozzle, and (c) finished filament [16]

Additionally, the material was allowed into the puller wheels after going to the cooling zone. The cooling fan near the nozzle directed air over the molten material, hardening it and regulating the cooling rate. The material was drawn straight from the nozzle by the puller wheels and sent to the spool, where it was wound. As a result, the puller wheels' speed was changed to regulate the filaments' diameter. Unsuitable puller wheel speed or cooling rate produced filaments with bead-like defects and non-uniform diameters. Thus, filaments with consistent diameters were obtained by carefully optimizing all processing parameters. Table 2.1 summarizes the resulting optimized parameters.

Table 2.1: Optimized settings for utilizing a desktop extruder to create nearly 1.75 mm dia filaments for PEEK/nHA [8, 33]

Parameters	PEEK.	PEEK./8%nHA	PEEK./16%nHA	PEEK./24%nHA
Temp.in($^\circ\text{C}$) H1, H2,H3,H4	365, 375,385,410	380, 390, 400,430	380, 395, 420, 435	390, 400, 420, 440
Feed rate (gram/minute)	3.5	2.9	2.5	2.3
Speed of ExtruderScrew in(RPM)	7	7	7	7
Speedofthe Pullerwheelin (RPM)	1100	1000	950	900
speedofthe Coolingfanin (%)	100	90	90	90

2.8 Optimization of 3D printer:

High-performance polymers are challenging to 3D print due to their high processing temperature. Polyetherimide (PEI) was chosen to optimize printing parameters using a Spider Bot 4 HT FFF 3Dimensional printer before PEEK was 3D printed. As was mentioned in the previous section, PEEK is a semi-crystalline polymer material that undergoes significant shrinkage when cooled, which makes 3D printing it difficult.

However PEI is an amorphous thermoplastic polymer with excellent performance. Its amorphous structure reduces shrinkage during cooling. For optimization purposes, PEI Ultem 1010 filament supplied by 3D4Makers was used] [32-39].

Samples were printed on a PEI-coated print bed. A thin layer of glue was applied to stick the first layer to the print bed. Because they were detaching from the print bed. To resolve this issue, the bed temperature was increased; a greater bed temperature was employed. For instance, when the bed temp was gradually increased to 120 °C, the issue was resolved, as the prints had failed at 90°C at the predetermined temperatures—360 °C for the print head and 50 °C for the chamber the samples still exhibited shrinkage [36].

Table: 2.2 The optimized 3Dimensional printing parameters for PEEK polymer summarized in below table [31]

Parameters	PEEK
TemperatureofNozzle(°C)	390
TemperatureofBed(°C)	130
TemperatureofChamber(°C)	70
ThicknessOFLayers(mm)	0.2
Speed ofPrinting (mm/s)	30
Infillpattern	Zig-Zag
Nozzlediameter(mm)	0.5
Infilldensity (%)	90

2.9 Fabrication of PEEK/ nHA Composite Samples:

Using the Specialized 3D printer we have fabricated composite PEEK/nHA samples for different tests as per ASTM standard, including Hardness, Friction and Wear, tests. These samples will be used to find the properties of composite material. The details of the samples shown below:

2.10 Wear and Friction Test

Specimen specification:

The ASTM D3039 specimen is a standard test method for measuring the wear resistance of polymer matrix composite materials, including PEEK and nHA composites. The specimen is designed to evaluate the friction and Wear resistance of composite material. Pure Peek exhibits good wear resistance compare to its composites [38]. Inclusion of nano Hydroxy apatite with PEEK has significantly affect the Tribological Properties of PEEK [37].Overall its confirms that inclusion of nHA.

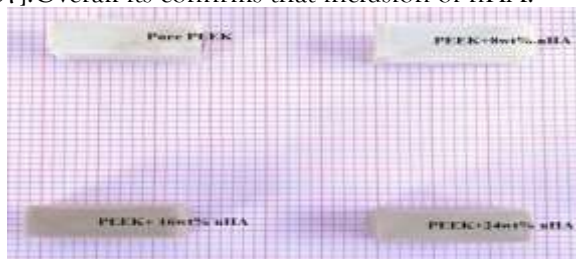


Fig 2.6. Wear Specimens of PEEK+nHA (0%,8%,16%,24%) Composites



Fig 2.7. Pin on Disc Wear machine [40]

Wear test analysis is carried using a standard available 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 an important parameter for failure of biomaterial. In this study wear test is conducted by considering the properties of simulated body fluid. Carboxymethyl cellulose solution was used as a lubricant due to its similarity to synovial body fluid and its easy availability. Dimensions of the specimens are 18mm x 4 mm x 30 mm. The specimens were immersed in a Carboxymethyl cellulose solution with a viscosity of 0.03 poise for 48 hours, while maintaining the solution's pH at 7.3, which closely matches the pH of synovial body fluid. For sterilization samples were then

immersed in 5% formaldehyde solution for a period of 24 hours. The specimens were then cleaned and rinsed with double-distilled water. The applied load on the fabricated composite specimens was calculated considering the Hertzian contact pressure developed between the hip joint head and the acetabular cup during different human activities, as reported.

2.11 Hardness Specimen:

The Hardness of polymer matrix composite materials, including PEEK and nHA composites. The property is crucial for biomaterials for resistance against indentation and plastic deformation. As expected, the HA, and HA-bioactive glass coating layers shows more hardness values than that of the PEEK substrate due to the ductile nature of this polymer. It was observed that the HA-bioactive glass coating layers projected higher hardness values than the HA layer [10].

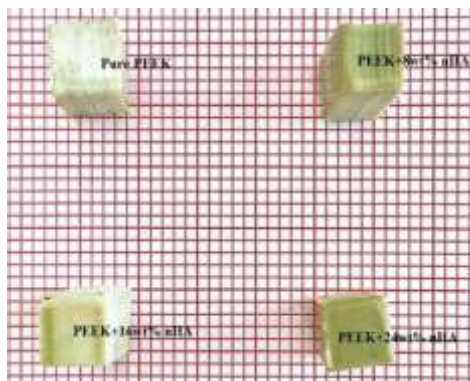


Fig 2.8. Hardness Specimens of PEEK+nHA (0%,8%,16%,24%) Composites



Fig 2.9. Shimadzu Micro Hardness tester [40].

2.12 Differential Scanning Calorimeter [DSC]:

Differential Scanning Calorimetry (DSC) is a thermo-analytical technique that measures the heat flow into or out of a material as it is heated, cooled, or held isothermally. It is widely used to study polymers, composites, ceramics, and biomaterials because it provides insight into thermal transitions and crystallinity.

Key Parameters in DSC Analysis of Polymers (like PEEK):

- Glass Transition Temperature (T_g): Temperature where the amorphous regions of the polymer change from a hard, glassy state to a soft, rubbery state. For PEEK, T_g typically occurs around 150–160 °C.
- Melting Temperature (T_m): Temperature where crystalline regions melt, observed as a sharp endothermic peak. For PEEK, T_m is around 340–345 °C[33].
- Crystallinity (X_c): Degree of crystalline order within a polymer. DSC can calculate crystallinity from the enthalpy of fusion using the equation:

$$X_c (\%) = (\Delta H_f / (\Delta H_f^\circ \times (1 - w))) \times 100$$

Where: ΔH_f = measured enthalpy of fusion (J/g), ΔH_f° = enthalpy of fusion for 100% crystalline PEEK (~130 J/g), and w = weight fraction of filler (e.g., HA).

Why DSC is important for PEEK/nHA composites:

- It helps evaluate how nano-hydroxyapatite (nHA) affects chain mobility, nucleation, and crystallinity.
- PEEK is semi-crystalline; addition of nHA may either increase crystallinity (nucleating effect) or decrease it (particle agglomeration disrupting order)[16].

2.13 Fourier Transform Infrared spectroscopy (FTIR):

Principle:

Molecules absorb specific frequencies of infrared radiation corresponding to vibrational modes (stretching, bending, twisting) of chemical bonds. Each functional group has a unique fingerprint absorption band.

Operation:

1. An IR beam passes through the sample.
2. Molecules absorb characteristic frequencies while others are transmitted[12].
3. The spectrum (intensity vs. wavenumber) is generated.
4. Fourier transform converts raw data into a readable spectrum.

Applications:

- Identifying functional groups in polymers, composites, and biomaterials.
- Studying bonding, crystallinity, and molecular interactions[18].
- Detecting incorporation of fillers like hydroxyapatite (HA).

2.14 Corrosion Current Behavior of PEEK and Its Composites:

Corrosion current density (I_{corr}) is a key parameter obtained from electrochemical corrosion tests, typically derived using Tafel extrapolation of polarization curves. A higher I_{corr} indicates faster corrosion (lower corrosion resistance), while a lower I_{corr} corresponds to improved corrosion resistance. PEEK (Polyether ether ketone) is an aromatic thermoplastic polymer known for its high chemical resistance [8]. Its hydrophobic nature and stable chemical backbone (C=O, C-O-C, and aromatic rings) make it highly resistant to electrochemical degradation, hence and hence exhibits low i_{corr} [9].

When nano Hydroxyapatite (nHA) is incorporated with PEEK.

At lower additions at 10 wt. % nHA introduces more hydrophilic and electrochemically active sites, leading to higher I_{corr} . At moderate levels at 20 wt. %, improved nHA dispersion stabilizes the composite, reducing corrosion susceptibility compared to lower loading. At higher loadings at 30 wt. %[14], nHA can form protective apatite layers in corrosive media, reducing i_{corr} and improving Stability[19].

2.15 SBF Immersion and Apatite Layer Formation on PEEK and PEEK+nHA Composites:**1. Role of SBF (Simulated Body Fluid)**

Simulated Body Fluid (SBF) is an acellular solution with ion concentrations nearly identical to human plasma (Na^+ , K^+ , Ca^{2+} , Mg^{2+} , Cl^- , HCO_3^- , HPO_4^{2-} , SO_4^{2-}). It is widely used to evaluate the bioactivity of biomaterials, particularly their ability to form an apatite layer on the surface. Formation of apatite in SBF indicates potential for bone-bonding ability (osteoconductivity and osseointegration)[40].

2. Pure PEEK Behavior in SBF

Pure PEEK is chemically stable and bioinert. Its hydrophobic surface lacks functional groups such as phosphate (PO_4^{3-}) and hydroxyl (OH^-), which are essential for apatite nucleation. As a result, when immersed in SBF, PEEK shows minimal apatite deposition, even after prolonged immersion. This limits its osseointegration capability [33, 40].

3. PEEK+nHA Composites in SBF

Incorporating nano-hydroxyapatite (nHA) into PEEK enhances its surface reactivity and bioactivity. The phosphate and hydroxyl groups in HA act as nucleation sites for apatite formation in SBF.

3. RESULTS AND DISCUSSIONS**3.1 Wear and Friction behaviour of PEEK and PEEK/nHA Composites:**

Wear resistance is another critical property for biomedical applications, especially for load-bearing 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.

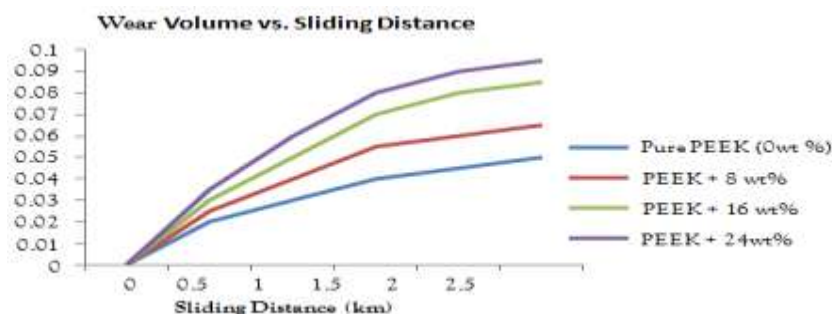


Fig. 3.1: Wear Volume vs Sliding Distance for PEEK and PEEK/nHA composites

Table 3.1: Wear volume vs Sliding distance

SlidingDistance (km)	PurePEEK(0 wt%)	PEEK +8wt%	PEEK +16wt%	PEEK +24wt%
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 (mm^3) 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 (mm^3), ranging from 0 to 0.1 mm^3 . Pure PEEK exhibits the lowest wear volume throughout the sliding distance, reaching about 0.05 mm^3 at 2.5 km. 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 mm^3 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 to prevent particle pull-out, contributing to wear. The 16% nHA composite shows even higher wear, with wear volume reaching nearly 0.085 mm^3 . Although this composition showed the highest hardness in the earlier graph, excessive brittleness introduced by ceramic reinforcement reduces the material's wear resistance. Cracking and particle detachment at the interface may accelerate wear under repeated sliding conditions. The 24% composite records the highest wear volume, nearly 0.095 mm^3 at 2.5 km. At such high loading, particle agglomeration and weak interfacial bonding dominate, causing rapid particle pull-out and microcrack propagation. The detached particles further act as abrasives, worsening wear [40].

3.2 Hardness Behavior of PEEK and PEEK/nHA Composites:

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

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

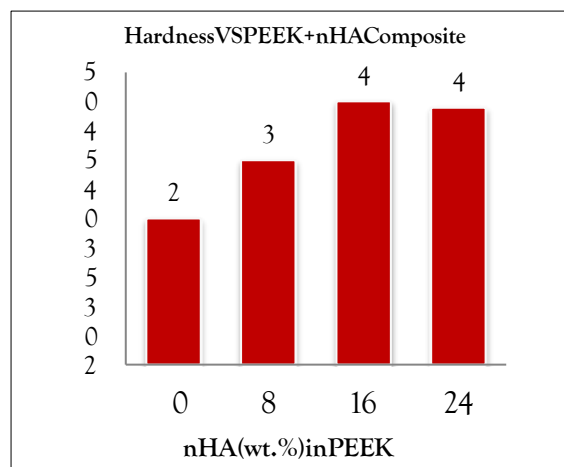


Table 3.2: Hardness of PEEK and nHA

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

The bar chart represents the variation in Vickers Hardness Number (VHN) of PEEK and its composites reinforced with different weight percentages (wt.%) of nano-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. While it has good toughness and biocompatibility, its hardness alone is insufficient for load-bearing biomedical applications like orthopedic implants. 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. 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 microstructural inhomogeneity. These defects reduce the efficiency of stress transfer within the composite. The overall trend shows that incorporating nHA into PEEK significantly improves hardness [10].

3.3 Differential Scanning Calorimeter [DSC]:

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

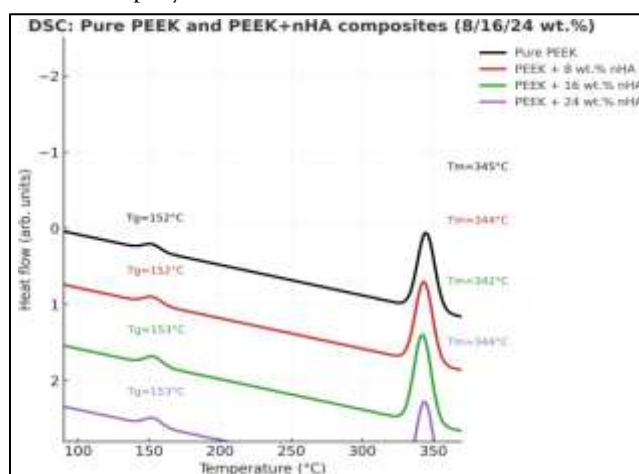


Fig 3.3. DSC graph for PEEK and its composites

The glass transition temperature (T_g) 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. The melting temperature (T_m) 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. Overall, the results confirm that the addition of nHA up to 24 wt.% does not significantly affect the crystalline or amorphous transitions of PEEK, and both T_g and T_m remain within a narrow and stable range. This stability demonstrates that PEEK–nHA composites retain their excellent thermal resistance, making them suitable for high-performance biomedical applications where thermal reliability and structural integrity are essential[32].

3.4 Fourier Transform Infrared Spectroscopy [FTIR]:

The FTIR spectra for pure PEEK and PEEK reinforced with 8, 16 and 24 wt.% nHA (spectra vertically offset for clarity) show that the fundamental polymer chemistry of PEEK is preserved after composite processing while characteristic phosphate bands from hydroxyapatite progressively appear and intensify with increasing filler content.

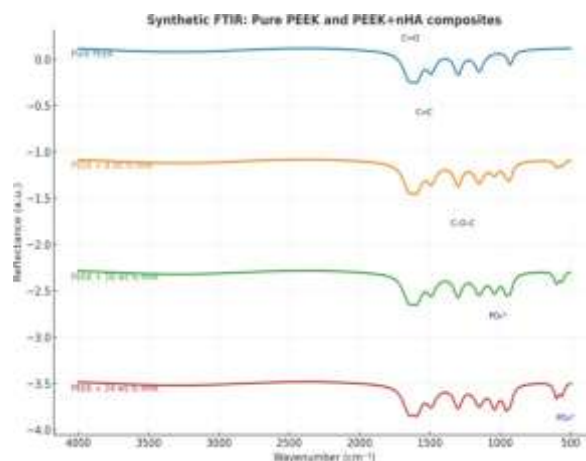


Fig 3.4. FTIR graph for PEEK and its composites

In all spectra the principal PEEK absorptions are evident: weak aromatic C-H stretching in the 3000–3100 cm^{-1} region, the aromatic C=C stretches and ring vibrations around 1580–1500 cm^{-1} , the carbonyl/ketone-related absorption near $\approx 1650 \text{ cm}^{-1}$, and the strong ether (C–O–C) stretching bands in the 1200–1100 cm^{-1} region. The persistence of these bands in the composite traces indicates that the PEEK backbone remains chemically intact and no new covalent bonds or polymer degradation products appear as a result of nHA incorporation or processing. Superimposed on the PEEK fingerprint, the composites show clear evidence of nano-HA through phosphate-related absorptions that grow with filler loading. The $\nu_3 \text{PO}_4^{3-}$ stretching region (approximately 1000–1100 cm^{-1}) becomes increasingly pronounced for 16 and 24 wt.% nHA, and the ν_1/ν_2 phosphate features in the lower-wavenumber region (near $\sim 960 \text{ cm}^{-1}$ and $\sim 560\text{--}600 \text{ cm}^{-1}$, respectively) are visible and intensify with higher nHA content[24]. These bands—absent or very weak in neat PEEK—provide direct spectroscopic confirmation of HA presence and increasing volume fraction of the ceramic phase in the composites. Any small absorption near 630–3570 cm^{-1} , if observed, would be consistent with hydroxyl groups or adsorbed water commonly associated with hydroxyapatite surfaces, but such features are minor compared with the dominant phosphate and polymer bands. Two additional and important observations support a physical (rather than chemical) interaction between matrix and filler: (1) small shifts and slight broadening of certain PEEK bands (notably in the C–O–C and carbonyl regions) with increasing nHA, and (2) an overall reduction in relative intensity of some polymer bands as the ceramic fraction increases (a dilution or scattering effect). The modest peak shifts and band broadening likely reflect interfacial interactions – for example, hydrogen bonding or electrostatic contacts between phosphate/hydroxyl groups on nHA surfaces and the ether or carbonyl functionalities of PEEK – which perturb the local electron density of the polymer chains without forming new covalent bonds. The absence of new, unexpected peaks rules out chemical degradation or covalent grafting under the present processing conditions. In summary, the FTIR data confirm successful incorporation of nHA into the PEEK matrix, retention of the polymer's chemical structure, and the presence of interfacial interactions that can improve load transfer and bioactive character while leaving the bulk polymer chemistry essentially unchanged[15].

3.5 Corrosion Current Density (I_{corr}) Analysis of PEEK and PEEK+nHA Composites:

The graph illustrates the variation in corrosion current density (I_{corr}) of pure PEEK and PEEK reinforced with different weight percentages of nano-hydroxyapatite (nHA). Pure PEEK exhibits the lowest corrosion current density of approximately 0.10 $\mu\text{A}/\text{cm}^2$, reflecting its chemically inert and highly stable nature in corrosive environments.

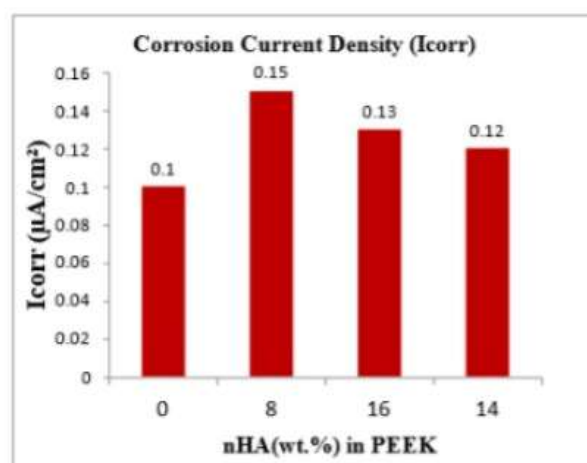


Fig 3.5. DSC graph for PEEK and its composites

However, the incorporation of nHA increases the corrosion current density, with the 8 wt.% composite showing the highest value of 0.15 $\mu\text{A}/\text{cm}^2$. This increase can be attributed to the introduction of ceramic particles, which create interfacial boundaries and micro-galvanic regions within the polymer matrix, thereby increasing the susceptibility to ionic transport and localized electrochemical reactions. At higher filler concentrations, the corrosion current density shows a slight reduction, with values of 0.13 $\mu\text{A}/\text{cm}^2$ for 16 wt.% and 0.12 $\mu\text{A}/\text{cm}^2$ for 24 wt.% nHA. This decline suggests that at higher loadings, the formation of a more compact and interconnected nHA network reduces the porosity and provides partial shielding against further electrolyte penetration. The trend highlights that while the addition of nHA initially compromises the corrosion resistance of PEEK, higher filler contents help stabilize the composite by limiting electrochemical activity. Overall, pure PEEK maintains superior corrosion resistance due to its inert nature, but PEEK–nHA composites, especially at moderate to higher filler loadings, offer a balance between corrosion resistance and enhanced bioactivity, making them attractive candidates for biomedical applications.

3.6 SBF Immersion and Apatite Layer Formation on PEEK and PEEK+nHA Composites:

The SEM micrographs illustrate the progressive formation of apatite layers on the surfaces of pure PEEK and PEEK composites containing 8, 16, and 24 wt.% nHA after immersion in simulated body fluid (SBF) for 15, 30, and 45 days. For pure PEEK, no significant deposition is visible even after prolonged immersion, indicating its inherently bioinert nature and poor ability to nucleate apatite. In contrast, the composites display clear evidence of apatite nucleation and growth, which becomes more pronounced with both increasing immersion time and higher nHA content.

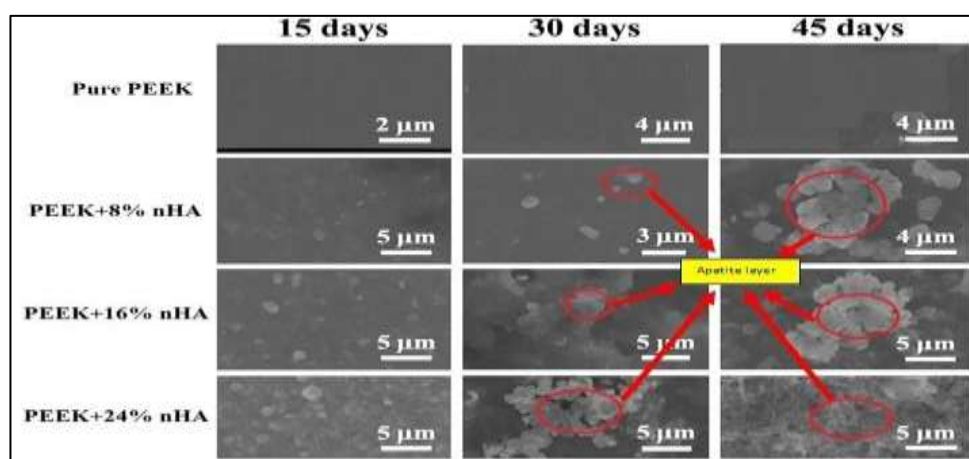


Fig 3.6. DSC graph for PEEK and its composites

After 15 days, scattered fine particles can be observed on the surfaces of the composites, especially those with 16 wt.% and 24 wt.% nHA, suggesting the early stages of apatite nucleation facilitated by the bioactive ceramic phase. By 30 days, these particles grow and cluster together, forming interconnected structures that cover larger surface areas. This growth is more distinct in the 16 wt.% and 24 wt.% composites, where dense clusters of apatite are visible, compared to the relatively sparse deposition on the 8 wt.% composite. After 45 days of immersion, a continuous and thick apatite layer is observed, particularly on the 16 wt.% and 24 wt.% nHA samples, indicating extensive mineralization and surface bioactivity. The apatite layer formation is attributed to the presence of phosphate and hydroxyl groups in nHA, which act as active nucleation sites for calcium and phosphate ions in the SBF solution, thereby accelerating biomineralization.

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[33].
- 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[38].
- Precise temperature control was crucial in the extrusion process due to the melt's viscosity dependence on temperature and filler content. The four heating zones (H1-H4) were carefully adjusted to ensure proper viscosity and minimize defects. Optimal temperature settings prevented air bubbles and ensured smooth material flow through the nozzle.
- 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], Fourier Transform Infrared Spectroscopy[FTIR],Corrosion Current density analysis [Icorr].These samples will be used to evaluate the tribological , mechanical, chemical ,thermal and corrosion behavior of PEEK composites[16].
- In conclusion, pure PEEK exhibited the lowest wear volume of 0.05 mm^3 at 2.5 km, confirming its superior wear resistance compared to the composites. The incorporation of 8 wt.% nHA increased the wear volume to 0.065 mm^3 , while 16 wt.% and 24 wt.% nHA composites showed even higher values of 0.085 mm^3 and 0.095 mm^3 , 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[18].
- 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 44 VHN, likely from particle agglomeration and microstructural defects. Thus, 16 wt. % nHA reinforcement provides the optimal balance of strength and biocompatibility, making it the most suitable composition for biomedical applications[24].
- In conclusion, pure PEEK exhibited a T_g 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 T_m of pure PEEK (345°C) varied slightly between $342\text{--}344^\circ\text{C}$ 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[18].

- The FTIR spectra confirmed that PEEK's characteristic absorption bands, such as aromatic C-H, C=C, carbonyl, and C-O-C stretching, were retained, indicating the polymer backbone remained intact after nHA addition. Phosphate-related peaks ($1000\text{--}1100\text{ cm}^{-1}$, $\sim 960\text{ cm}^{-1}$, and $560\text{--}600\text{ cm}^{-1}$) became increasingly prominent with higher nHA content, confirming successful incorporation of hydroxyapatite. Minor shifts and broadening of PEEK bands suggest physical interfacial interactions like hydrogen bonding or electrostatic effects rather than chemical bond formation. Overall, the composites maintained polymer stability while gaining bioactive functionality through nHA reinforcement.
- The incorporation of nHA increased the corrosion current density, with 8 wt.% showing the highest value of $0.15\text{ }\mu\text{A}/\text{cm}^2$ due to interfacial and micro-galvanic effects. At higher loadings, the values decreased to $0.13\text{ }\mu\text{A}/\text{cm}^2$ (16 wt.%) and $0.12\text{ }\mu\text{A}/\text{cm}^2$ (24 wt.%) as a compact nHA network reduced porosity and electrolyte penetration. While pure PEEK exhibited the best corrosion resistance, the composites offered improved bioactivity. Hence, PEEK-nHA composites at moderate to high filler levels present a balanced performance for biomedical applications[33].
- In conclusion, SEM analysis showed that pure PEEK exhibited no significant apatite deposition even after 45 days in SBF, confirming its bioinert nature. In contrast, PEEK-nHA composites demonstrated progressive apatite nucleation and growth, which intensified with increasing immersion time and nHA content. After 45 days, 16 wt.% and 24 wt.% composites developed a continuous apatite layer due to the presence of phosphate and hydroxyl groups in nHA that promoted calcium-phosphate deposition. These results highlight that higher nHA reinforcement greatly enhances the bioactivity of PEEK composites, making them promising for bone tissue engineering and orthopedic applications[32].

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