Fabricating Bone Scaffolds Using PEEK Incorporated With Hydroxyapatite and Carbon To Enhance Mechanical Properties

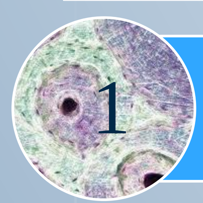
Purpose

The purpose of this experiment is to research if an artificial bone scaffold could be engineered using thermoplastic for the purpose of bone regeneration.

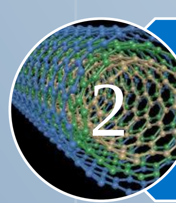
Introduction

Bone regeneration is a complex process of bone formation that results after a bone sustains damage, such as a fracture. Bone regeneration has become an increasing medical problem in the elderly and war veterans. One method that promotes the regeneration of bone is the use of scaffolds. Scaffolds are general structures that provide support that also provide an environment needed for cellular growth. Bones naturally produce a type of scaffold called the soft callus, healing tissue that holds the broken bone together and allows for further growth to take place after an injury. However, if bones have tissue damage, then then a natural scaffold will not be produced. This is where synthetic scaffolds are needed. Important characteristics of scaffolds used in bone tissue engineering are A) high porosity B) biodegradability and strong mechanical properties C) biocompatibility. High porosity allows for the facilitation of oxygen and nutrients and aid in waste removal during the healing process. The pores distribute cells evenly and promote tissue regeneration. Mechanical properties are also important because the scaffolds needs to be able to sustain the weight and movement of a person. The Young's Modulus of a longitudinal cortical bone such as the humerus is between 10-20 GPa and has a compressive strength of up to 210 MPa. One such material that is efficient in creating scaffolds is the organic thermoplastic polymer polyetheretherketone (PEEK). PEEK has exceptional tensile properties such as a Young Modulus of about 3.1 GPa, a tensile strength of 100 MPa, and elongation at break of 50%. PEEK is also resistant to high temperatures and thermal degradation.. Regarding the aspect of biocompatibility, PEEK in bioinert, meaning that it does not initiate a response or interact with biological tissues. In order for a scaffold be successful it must be able to support cellular growth and avoid inducing any type of infection or any additional immune responses. In order to combat this issue, scaffold materials are typically combined with other substances that have good biocompatibility in order to form a scaffold that has good bonding abilities. However, adding a substance that increases the PEEK biological properties decreases its mechanical properties with so reinforcements with biocompatibility must be added, like carbon fiber, which has strong crystallite covalent bonds and strong mechanical abilities such as a compressive modulus of up to 230 GPa.

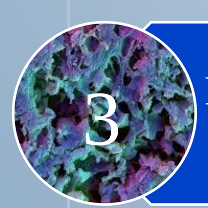
Goals



1 Fabricate scaffolds using PEEK incorporated with hydroxyapatite to increase biocompatibility



2 Increase scaffold mechanical properties through the addition of carbon fibers and carbon nanotubes



3 Fabricate scaffolds with porosity of 75% or higher for tissue formation

Results

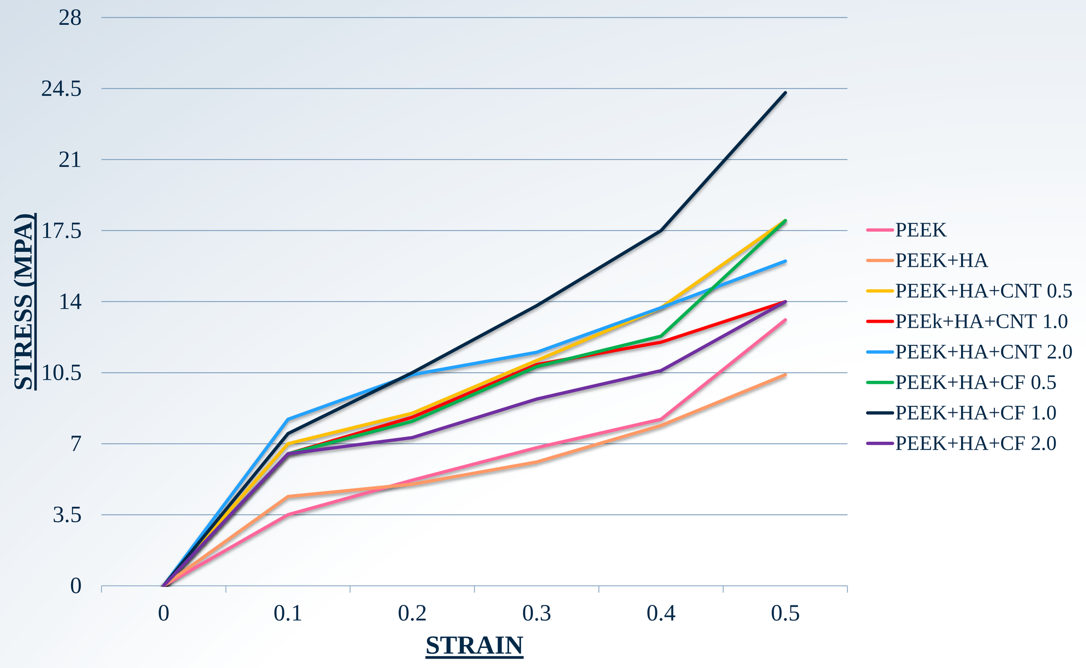


Figure 1: Shows stress-strain of the 8 biocomposites to highlight elasticity of material

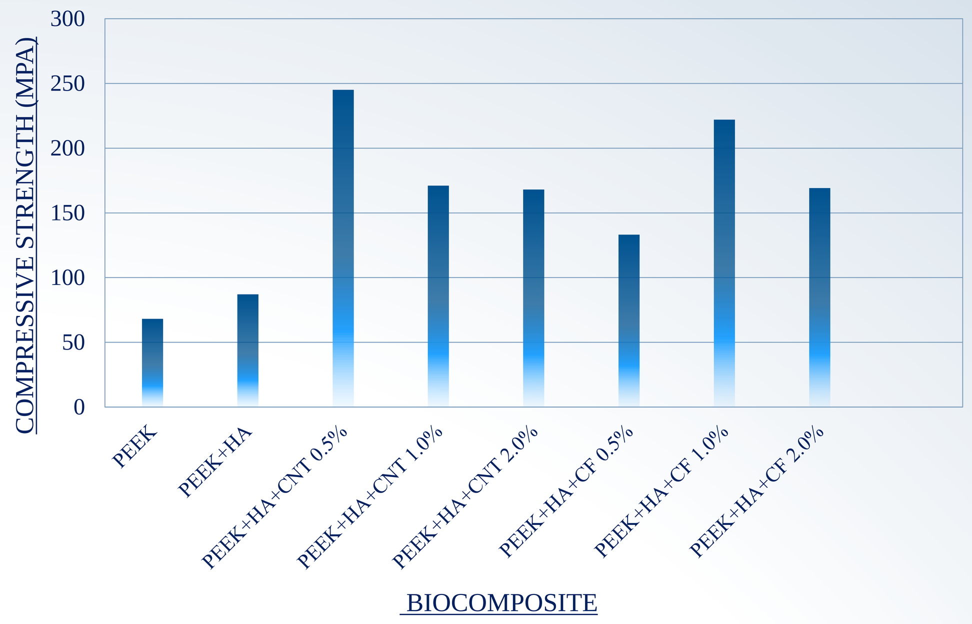


Figure 2: Shows relationship between stress/strain for the 8 biocomposites and resistance to compression of the materials on average.

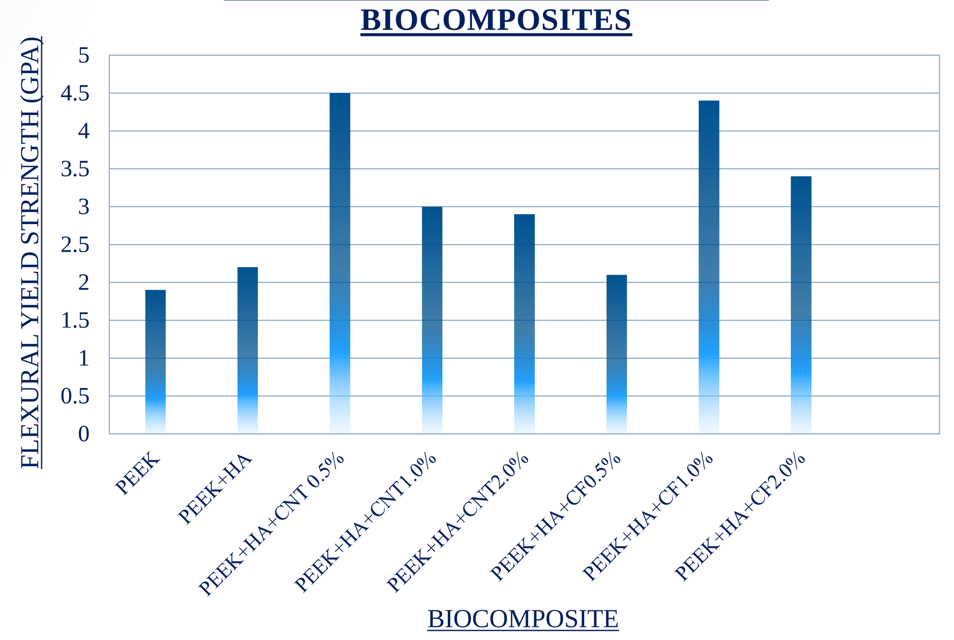


Figure 3: Shows the average stress the materials underwent before reaching yield point and undergoing plastic deformation

EQUATIONS FOR COMPUTATION:

- Stress = Ratio of applied force to a cross section area
  - $\sigma = F_N/A$, units are N/m^2
- Strain = Ratio of change in length and initial length
  - $\varepsilon = \frac{\Delta l}{l_o}$, dimensionless
- Compressive Strength = Load at point of failure ÷ initial cross-sectional surface area
  - $CS = \frac{F}{A}$, units are MPa
- $P_{CT} = \frac{TV - MV}{TV}$ x 100, average porosity of scaffold where TV is total volume, MV is scanned material volume of the scaffold

Biocomposite	Average Porosity(%)	Average Pore Size (µm)
PEEK	75.1	285
PEEK+HA	81	275
PEEK+HA+CNT 0.5%	79	248
PEEK+HA+CNT 1.0%	75.4	263
PEEK+HA+CNT 2.0%	77	287
PEEK+HA+CF 0.5%	75.7	245
PEEK+HA+CF 1.0%	75.9	266
PEEK+HA+CF 2.0%	76.1	300

Table 1: Micro-CT scan results of porosity for biocomposites

Scaffolds Methods	Young's Modulus (GPa)	Compressive Strength (MPa)	Yield Modulus (GPa)	Porosity (%)
Bone	10-20	210	13.1	75-95
PEEK+HA+CNT 0.5%	3.9	245	4.5	75.4
CaPs	2.23	84	3.2	92
Bioglass	--	150	4.9	62
Propylene Fumarate	2.34	--	1.8	--
Titanium LENS	--	215.7	15.4	95

Table 1: Comparisons to other methods and materials for scaffolding and their properties to biocomposite PEEK+HA+CNT 0.5%

Methodology

Step 1: Functionalization

- CNTs/CFs mixed with nitric acid in a 1:100 ratio. Solution was mixed on a hot plate at 250°C for 4 hours at 200 rpm
- Solution was neutralized by mixing 750 mL of pure water for 10 min on hot plate at 250°C at 200 rpm
- Applied vacuum filter
- Dried in oven at 85°C for 24 hours

Step 2: Fabrication of Biocomposites

- 0.5%, 1.0% and 2.0% solutions were made by adding CNTs/CFs to 20 mL of toluene
- Sonicated mixture in a 4×5 cycle with 5 min interval
- Added PEEK and HA particles to CNTs/CFs for 0.5%, 1.0% and 2.0%
- Drained and dried at room temp for 48 hours
- Dried in oven at 120°C for 3 hours
- Ground for 2 min
- Salt porogen was added using pulsing vortex mixer for 10 min at 3000 rpm

Step 3: Casting

- PEEK was added to 5/8 in aluminum tube and wrapped in thin layer of aluminum foil
- Molds were placed in a high temp furnace at 400°C for 4 hours and then cooled to room temp
- Tubes were placed in vacuum tube furnace at 400°C for 4 hours at pre-fixed ramp-up cooling rate of 4°C per min
- Used a lathe center, the cooled samples were cut to 0.5 in in diameter and 1 in in length

Step 4: Leaching and Properties Tests

- Samples were leached for 3 days and then dried in oven at 110°C for 2 hours
- Compression test of 1.27 mm/min were preformed on samples at 37°C
- Micro-CT scans were preformed to observe pore size, volume of pores and interconnectivity

Conclusion

There were three goals for this experiment 1) Fabricate scaffolds using PEEK incorporated with hydroxyapatite to increase biocompatibility 2) Increase scaffold mechanical properties through the addition of carbon fibers and carbon nanotubules 3) Fabricate scaffolds with porosity of 75% or higher for tissue formation. The 3 goals were reached. Functioning scaffolds were made using PEEK and HA, and afterward, all trials show that incorporating carbon fibers and carbon nanotubules increased the mechanical properties of not only the PEEK+HA, but those of polyetheretherketone alone. The biocomposite of PEEK+HA+CNT 0.5% had the highest yield strength of 4.5 GPa and compressive strength of 245.9 MPa, which is an increase of 89.1% in compressive strength and a 136.8% increase in yield strength from PEEK. The scaffolds had 76.9% porosity on average meaning uniform pore distribution.

Future Works

In order to truly understand the interconnectivity of the cells in the scaffolds, the scaffolds would have to be tested *in vivo* with sample tissue taken from patients. This would allow for the observation of tissue ingrowth and surface interactions and experimentation with biological forces *in vivo* to further apply artificial scaffolds to osteogenesis for patients with significant tissue damage.

*All figures, tables and pictures taken by experimenter