

**DESIGN, DEVELOPMENT, AND BIOMECHANICAL  
EVALUATION OF COMPOSITE MATERIALS FOR  
BIOMEDICAL APPLICATIONS**

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EVALUATION OF COMPOSITE MATERIALS FOR  
BIOMEDICAL APPLICATIONS**

by

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CENTRE FOR BIOMEDICAL ENGINEERING**

*Submitted*

**In fulfilment of the requirement of the degree of Doctor of Philosophy  
to the**



**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

**JULY 2024**



**Dedicated thankfully to**

**Almighty,**

**My  
Parents,  
Family,  
Teachers  
and  
Inspirers**



## **Declaration**

The research work embodied in this thesis entitled “DESIGN, DEVELOPMENT, AND BIOMECHANICAL EVALUATION OF COMPOSITE MATERIALS FOR BIOMEDICAL APPLICATIONS” is submitted to the Centre for Biomedical Engineering, Indian Institute of Technology Delhi. The investigations, results, findings, and conclusions reported in the thesis are original work.

This original work has not been submitted in part or in full for the award of any other degree or diploma to this or any other university or institution.

I hereby confirm the originality of the thesis research work.

**Balakumaran V**



## **Certificate**

This is to certify that the thesis titled “DESIGN, DEVELOPMENT, AND BIOMECHANICAL EVALUATION OF COMPOSITE MATERIALS FOR BIOMEDICAL APPLICATIONS” submitted by Mr. Balakumaran V to the Indian Institute of Technology Delhi for the award of the degree of Doctor of Philosophy in Centre for Biomedical Engineering, is a record of bonafide research work carried out by him. He has worked under my guidance and supervision.

The results contained in this thesis have not been submitted in part or full, to any other University or Institute for the award of any degree or diploma.

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Balakumaran V



## **Abstract**

The present thesis is focused on developing a new dispersion coating method for producing towpreg for consolidating void free composites of carbon fiber reinforced polyketones for load-bearing biomedical applications. The first part of the thesis focused on the design, development, fabrication, and installation of a new dispersion coating machine capable of producing flexible towpregs of carbon fiber with polyketones [PAEK, PEEK, and PEKK]. A biomedical application suitable method of producing a dispersion of polyketone microparticles in an aqueous medium was developed. Polyamic acid (PAA), a precursor molecule of polyimide based on Pyromellitic dianhydride-oxydianiline (PMDA-ODA), was used as the dispersing agent. Using the dispersions, towpregs were produced to consolidate composites. The mechanism and nature of the coating formed on the towpreg was studied. Three different composites for each polyketone matrix [PAEK, PEEK, and PEKK] were studied to identify the composite with maximum strength free of voids. Detailed analysis of the composites was carried out to explain the origin of strength in CF/polyketone composite. CF/PAEK composite was identified as having the maximum strength. In the later part of the thesis, studies were carried out to establish CF/PAEK composite for load-bearing biomedical applications. A ten-hole bone plate was manufactured by thermal forming using the CF/PAEK composite consolidated using dispersion coated towpreg. The performance of the composite bone plate was compared with the SS bone plate as a benchmark. The bending performance of the composite bone plate and SS bone plate was evaluated by bending test according to ASTM F382. The composite bone plate had 87% higher strength and 16% lower bending structural stiffness than the SS bone plate, establishing the suitability of CF/PAEK composite for load-bearing biomedical applications from a biomechanical performance point of view.



## सारांश

वर्तमान थीसिस लोड-असर बायोमेडिकल अनुप्रयोगों के लिए कार्बन फाइबर प्रबलित पॉलीकेटोन के शून्य मुक्त कंपोजिट को मजबूत करने के लिए टॉप्रेग के उत्पादन के लिए एक नई फैलाव कोटिंग विधि विकसित करने पर केंद्रित है। थीसिस का पहला भाग पॉलीकेटोन [PAEK, PEEK, और PEKK] के साथ कार्बन फाइबर के लचीले टॉप्रेग का उत्पादन करने में सक्षम एक नई फैलाव कोटिंग मशीन के डिजाइन, विकास, निर्माण और स्थापना पर केंद्रित था। एक जलीय माध्यम में पॉलीकेटोन माइक्रोपार्टिकल्स के फैलाव के उत्पादन के लिए एक बायोमेडिकल अनुप्रयोग उपयुक्त विधि विकसित की गई थी। पॉलीमिक एसिड (पीएए), पाइरोमेलिटिक डायनहाइड्राइड-ऑक्सीडायनिलिन (पीएमडीए-ओडीए) पर आधारित पॉलीमाइड का एक अग्रदूत अणु, फैलाने वाले एजेंट के रूप में इस्तेमाल किया गया था। फैलाव का उपयोग करते हुए, कंपोजिट को मजबूत करने के लिए टॉप्रेग का उत्पादन किया गया था। टॉप्रेग पर गठित कोटिंग के तंत्र और प्रकृति का अध्ययन किया गया था। प्रत्येक पॉलीकेटोन मैट्रिक्स [PAEK, PEEK, और PEKK] के लिए तीन अलग-अलग कंपोजिट का अध्ययन किया गया ताकि रिक्तियों से मुक्त अधिकतम शक्ति वाले समग्र की पहचान की जा सके। पॉलीकेटोन समग्र में ताकत की उत्पत्ति की व्याख्या करने के लिए कंपोजिट का विस्तृत विश्लेषण किया गया था। CF/PAEK कम्पोजिट की अधिकतम शक्ति के रूप में पहचान की गई थी। थीसिस के बाद के भाग में, लोड-असर बायोमेडिकल अनुप्रयोगों के लिए सीएफ / पीएईके समग्र स्थापित करने के लिए अध्ययन किए गए थे। एक दस-छेद हड्डी प्लेट का निर्माण थर्मल बनाने के द्वारा किया गया था सीएफ/पीएईके समग्र फैलाव लेपित टॉप्रेग का उपयोग करके समेकित किया गया। समग्र हड्डी प्लेट के प्रदर्शन की तुलना बेंचमार्क के रूप में एसएस हड्डी प्लेट के साथ की गई थी। समग्र हड्डी प्लेट और एसएस हड्डी प्लेट के झुकने प्रदर्शन एएसटीएम एफ

382 के अनुसार झुकने परीक्षण द्वारा मूल्यांकन किया गया था। समग्र हड्डी प्लेट में एसएस हड्डी प्लेट की तुलना में 87% अधिक शक्ति और 16% कम झुकने वाली संरचनात्मक कठोरता थी, जो बायोमेकेनिकल प्रदर्शन के दृष्टिकोण से लोड-असर बायोमेडिकल अनुप्रयोगों के लिए CF/PAEK समग्र की उपयुक्तता स्थापित करती है।

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## Abbreviations

CF	: Carbon fiber
GF	: Glass fiber
HP-TPs	: High-performance thermoplastics
HP-TPCs	: High-performance thermoplastic composites
PAEK	: Poly-aryl-ether-ketone
PEEK	: Poly-ether-ether-ketone
PEKK	: Poly-ether-ketone-ketone
PLC	: Programmable logic controller
HMI	: Human machine interface
LVDT	: Linear variable differential transformer
PID	: Proportional-integral-derivative
CAD	: Computer-aided design
3D	: Three-dimensional
2D	: Two-dimensional
AC	: Alternating current
UPS	: Uninterrupted power supply
CPU	: Central processing unit
SS	: Stainless steel
UD	: Unidirectional
SEM	: Scanning electron microscopy
FESEM	: Field-effect scanning electron microscopy
FTIR	: Fourier transform infrared spectroscopy
XRD	: X-ray diffraction

$\mu$ CT : Micro-computed tomography

PE : Poly(ethylene)

PA : Polyamide

PLA : Poly(lactic acid)

PS : Polysulfone

PPS : Poly(phenylene sulfide)

PES : Poly (ether sulfone)

PET : Poly (ethylene terephthalate)

BG : Bioglass

UHMWPE : Ultra-high molecular weight polyethylene

PMMA : Polymethyl meth- acrylate

PI : Polyimide

PEI : Poly (etherimide)

LCP : Liquid crystalline polymer

TPI : Thermoplastic polyimide

CoCr : Cobalt Chromium

DPS : Diphenylsulfone

FE : Finite element

DCP : Dynamic compression plate

NMR : Nuclear magnetic resonance

3D : Three-dimensional

PVDF : Poly (vinylidene fluoride)

DI : Deionized

PMDA-ODA : Pyromellitic dianhydride-oxydianiline

PAA	: Polyamic acid
PAAS	: Ammonium salt solution of polyamic acid
CNT	: Carbon nanotube
DMAc	: Dimethylacetamide
PAN	: Polyacrylonitrile
PTFE	: Polytetrafluoroethylene
MFI	: Melt flow index
DLS	: Dynamic Light Scattering
FTIR	: Fourier Transform Infrared spectroscopy
ATR	: Attenuated Transmittance Reflectance
EDX	: Energy-dispersive X-ray spectroscopy
TGA	: Thermogravimetric analysis
DSC	: Differential scanning calorimetry
CLC	: Combine loading compression
VOI	: Volume of interest
MTT	: Methyl Tetrazolium



## Nomenclatures

$T_g$	: Glass transition temperature
$T_m$	: Melting point
$U_c$	: Uniformity referred to particle distribution
$D_{50}$	: The particle size corresponding to the cumulative frequency of 50%
$X_v$	: Crystalline volume fraction
$H_f$	: Theoretical heat of fusion of pure crystalline phase (130 J/g)
$H_m$	: Heat of fusion: area under the melting peak
$H_c$	: Heat of additional crystallization: area under the crystallization peak
$\rho_c$	: Density of crystalline phase (1.4 g/cm <sup>3</sup> )
$\rho_a$	: Density of amorphous phase (1.26 g/cm <sup>3</sup> )
$X_{mr}$	: Mass fraction of reinforcement fiber
$T_d$	: Theoretical density of the composite
$w_f$	: Weight fraction fiber
$w_m$	: Weight fraction matrix
$\rho_f$	: Density of CF 1.8 g/cm <sup>3</sup>
$\rho_m$	: Density of PEEK 1.3 g/cm <sup>3</sup>
$\sigma_{fs}$	: Flexural strength
$P_{max}$	: Maximum force in load extension curve
$L$	: Support span
$b$	: Width of sample
$d$	: Thickness of sample
$E_f$	: Flexural modulus

$m$	: Slope of load extension curve
$\sigma_t$	: Tensile strength
$\sigma_c$	: Compression strength
$\sigma_{LSS}$	: Inter-laminar shear strength
$\sigma_b$	: Bending strength of bone plate
$EI$	: Bending structural stiffness of bone plate
$h$	: Loading span distance
$a$	: Central span distance
$k$	: bending stiffness