# IMPROVE THE MECHANICAL AND PHYSICAL PROPERTIES OF HYBRID (POLY ETHER ETHERKETON) COMPOSITES REINFORCED WITH CARBON FIBER AND GLASS FIBER

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**PhD Thesis-2023**

**T.C.**
**ATATURK UNIVERSİTY**
**GRADIATE SCHOOL OF HEALTH SCIENCES**

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**ERZURUM**
**2023**

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# ACKNOWLEDGMENTS

This Study was supported by The Coordination Unit of Scientific Research Projects, Atatürk University, Erzurum, Türkiye, with grant number TDK-2022-10055.

I would like to begin by expressing my deep-felt thankful to my supervisor, Prof. Dr. Funda BAYINDIR, for granting me the opportunity to work on this PhD thesis under her proficient guidance. I am truly gratitude for her time investment and efforts in this research, as well as for providing me the necessary informations and equipments to carry out this work. With determined devotion and professionalism, she guided this project.

I want to extend my sincere appreciation to all the respected ducators who have left an ineffaceable imprint on my mind. Among them, I would like to especially mention Prof. Dr. Nuran DİNÇKAL, for making me feel a part of the family of the department, Assoc. Prof. Dr. Alper ÖZDOĞAN, for supporting and helping me with all information I needed, I express my gratitude for the help and advice provided by Prof. Dr. Zainab YEŞIL DUYMUŞ, along with all the doctors who taught me at the Prosthodontic Department, Faculty of Dentistry, Ataturk University.

Furthermore, I am vastly thankful to my family. My deceased father, whom I wished would be with me when I reached this stage, as he was a loving supporter of my academic career, and I owe my mother a debt of gratitude for backing me along the way. Special thanks to my husband for pushing me forward to finish my work. My journey was special and worthy with the existence of my two daughters who withstranded all the difficulties we faced togather in this long trip.

Last, I want to acknowledge the divine guidance of God, leading me on an unexpectedly unique journey. I consider myself blessed to have had such diverse sources of encouragement throughout my academic pursuits and aspirations.

Reem Abdulrahim Nsaif ALOBAIDY

# ÖZET

## Karbon Fiber Ve Cam Fiber İle Güçlendirilmiş Hibrit (Poli Eter Eterketon) Kompozitlerinin Mekanik Ve Fiziksel Özelliklerinin İncelenmesi

**Amaç:** Polyetheretherketone (PEEK) tıp alanlarında diş hekimliği dahil yaygın olarak kullanılmaktadır, son yıllarda birçok uygulama için geniş çapta araştırılmıştır; diş implantları, maksillofasiyal implantlar,osteosentez plakası ve kemik replasman malzemesi PEEK mükemmel biyouyumluluğu, radyolüsent, kimyasal direnci ve düşük yoğunluğu nedeniyle birçok dental uygulama için alternatif bir materyal olarak kabul edilir. Poli eter eter keton malzemesi yüksek mukavemete sahiptir; tokluk özellikleri, yüksek termo-oksidatif stabilite, mükemmel alev geciktirici,biyouyumluluk ve kimyasal ve aşınma direnci.Bu özelliklerinden dolayı önemli bir mühendislik termoplastik malzemesi olarak kabul edilmektedir.Gelişmiş üreticilerin daha yüksek performanslı malzemelere yönelik sürekli talebi, PEEK bazlı Nano kompozitlerinin ortaya çıkmasına yol açmıştır. Karbon nanotüpler ve (Nano)fiberler, silika, tungsten, hidroksiapatit ve grafen gibi nanodoldurucular, matrisin mekanik, termal ve elektriksel özelliklerini iyileştirmek için iyileştirici malzemeler olarak kullanılır. Kompozit malzemelerin üretiminde doğal veya sentetik liflerin kullanılması inşaat, mekanik ve biyomedikal gibi birçok alanda önemli başarılar ortaya çıkarmıştır.CFR-PEEK, çok yönlülüğü, teknolojinin yeni versiyonuyla uyumluluğu, mükemmel mekanik özellikleri ve biyouyumluluğu nedeniyle tıbbi implant uygulamalarında tercih edilmektedir. Bu malzeme farklı fiziksel, mekanik ve yüzey özelliklerine sahip

birçok formda üretilebilmektedir. Aynı doğrultuda, doğal dişlerin ısırma kuvvetleri dahilinde değişen cam elyaf takviyeli PEEK (GFR-PEEK) ile basınç yükleme testleri yapılmış ve yeterli yorulma limitleri ortaya konmuştur. Dental implant materyali, özellikle abutmentler ve üst yapılar için kullanılanlar için daha büyük elastisite modülüne PEEK EM gereklidir. 18 Gpa'ya kadar yüksek bir değere sahip elastik bir modül elde etmek ve daha ser bir hibrit PEEK elde etmek için; karbon fiber ve cam fiber ile güçlendirime yapılabilir. Klinik öneme genel bakış; PEEK özelliklerinin çoğunun dentin ve kemiğe benzer olduğunu gösterir, bu nedenle implantlar, abutment, sabit ve hareketli diş protezleri için diş materyali olarak kullanılabileceğini açıklamaktadır.Polietereterketon (PEEK), yarı kristal yapısal bir polimerdir, mükemmel bir biyouyumluluğa sahiptir ve son zamanlarda yük taşıyan diş uygulamalarında potansiyel kullanımı için araştırılmıştır. PEEK, metal alaşımlarına bir alternatiftir ve yüksek performanslı polimerler oldukları için giderek artan bir şekilde başarılı bir alternatif malzeme olarak kabul edilmektedir. Peek'in diğer özellikleri şunlardır:

1. Bu materyalin incelenmesi sırasında toksik formda sitotoksisite, mutajenite, kanserojenlik veya immünojenite kanıtı yoktur.
2. iyi özelliklere sahip bir materyal oluşturmak için diğer bileşenlerle birleştirilebilir.
3. Bu materyal düşük bir Elastisite modülüne (EM) sahiptir (kemiğin elastisite modülüne yakındır) ve bu materyal için en temel özellik olarak yansıtılır.

Daha iyi mekanik özellikleri nedeniyle, sabit ve hareketli protezlerin yapımında CAD-CAM malzemesi olarak PEEK ve akrilik gibi malzemeler de kullanılabilir. Polieter eter keton malzemesi sahip olduğu özellikler nedeniyle önemli bir termoplastik malzeme olarak kabul edilmektedir. daha büyük esneklik modülü PEEK EM gereklidir. Yüksek değerli bir elastik modül elde etmek ve daha tok bir hibrit PEEK elde etmek için; Karbon fiber ve cam fiberleri ile takviye edilebilir. Diş hekimliğinin birçok alanında yaygın olarak kullanılmasını sağlayan uygun fiziksel ve mekanik özellikleri sayesinde tıp ve diş hekimliği alanlarında umut vericidir . Malzemeler için ileri endüstrilerden gelen sürekli talep, PEEK tabanlı nanokompozitlerin geliştirilmesine yol açmıştır; Burada, malzeme matrisinin mekanik, termal ve elektriksel özelliklerini geliştirmek için takviye ajanları olarak karbon fiberler, nano cam fiberler gibi nano dolgu maddeleri kullanılmıştır. Çünkü günümüzde Peek malzemesi diş hekimliğinin birçok alanında ve birçok amaç için yaygın olarak kullanılmaktadır. Bu çalışmada dental (polieter eter keton) PEEK'in bazı mekanik ve fiziksel özelliklerinin iyileştirilmesi amaçlandı. PEEK malzemesine iki farklı türde Fiber takviye edilecektir; karbon fiberleri ve cam fiberleri takviyesi. Basma, Eğilme, Çekme ve Sertlik testleri uygulanacaktır. Testler fiber takviyeli ve takviyesiz olarak gerçekleştirilecektir. Ölçümler karşılaştırılacak ve sonuçlar değerlendirilecektir.

**Materyal ve Metot:** Bu çalışma Atatürk Üniversitesi Diş Hekimliği Fakültesi Etik Kurulu tarafından onaylandı (İşlem numarası 2022/36) ve Atatürk Üniversitesi Bilimsel Araştırma Koordinatörlüğü BAP (2022/TDK-2022-10055) tarafından desteklenmiştir. Bu çalışmada Atatürk Üniversitesi Diş Hekimliği Fakültesi Protetik Diş Tedavisi Bölümü araştırma laboratuvarlarında ve Erzurum

Teknik Üniversitesi YüksekTeknoloji Uygulama ve Araştırma Merkezinde (YUTAM) mekanik testler yapılmıştır. Dört farklı PEEK türü kullanılmıştır; saf PEEK extrosin tipi (Vıctrex Corp INCHR, New Material, China), saf PEEH sıkıştırma tipi (WHITEPEAKS Light copra PEEK, Germany), karbon fiber PEEK ve cam fıber PEEK tipi (Vıctrex Corp INCHR, New Material, China). Bu çalışmada kullanılan örneklem sayısı, örneklem büyüklüğünü belirlemek için (F. Kibuacha, 2021) tarafından açıklanan parametrelere ve örneklem büyüklüğünün daha küçük hata marjları sağlayacak ve daha temsili olacak şekilde nasıl hesaplanacağına dayanmaktadır. Bu tezde farklı PEEK malzemelerinin her biri için basınç dayanımı testi, bükme testi, çekme testi ve Vickers sertlik testi olmak üzere.dört farklı mekanik test ölçülmüştür; Her test için toplam 160 numune üretilmiştir (n=40). Kuru depolamada ve Ringer solüsyonunda (Polifleks® Polıforma, Polifarma İlaç Sanayi Ve Tic., Türkiye) inkübasyonun ardından farklı zaman aralıklarında 37°C'de 1 gün, 1 hafta ve 3 hafta test edilmiştir; Test edilen numuneler, her mekanik teste özel ISO standart ölçümlerine göre kesildi ve bir cilalayıcı (Buehler, Coventry, İngiltere) tarafından P4000'e Kadar (Buehler, Lake Bluff, IL, ABD) bir dizi Si aşındırıcı kağıtla Manuel olarak cilalandı. Basma dayanımı testi için; PEEK numuneleri ISO 604: 2002'ye (Plastikler — Basınç özelliklerinin belirlenmesi) göre 10×10×4 mm Numune boyutuyla kesilmiştir (Bierögel ve Grellmann, 2014; ISO 604: 2002 Plastikler). Bükme testi için PEEK numuneleri ISO 178: 2010'a (Plastikler - Bukme Özelliklerinin Belirlenmesi) göre 80×10×4 mm (ISO 178:2010 Plastikler) numune boyutlarında kesilmiştir 90 x 5 x 4 mm köpek kemiği şeklindeki çekme testi numuneleri, ISO 527-2 standardına göre kesilmiştir: (2012 Plastikler - Çekme Özelliklerinin Belirlenmesi). Mikrosertlik Vickers testi için test edilen numuneler ISO 20795-1'e (2008- Diş Hekimliği-Taban polimerleri-Protez kaidesi polimerleri) göre 30x10x2, 5 mm boyutunda dikdörtgenler halinde hazırlanmıştır. Grup oluşturulduktan sonra dört mekanik test yapılmış, her alt grup için farklı PEEK malzemelerinin kuru depolama ortamındaki örnekleri kontrol grubu olarak değerlendirilmiştir. Basınç testi, 10 KN yük hücreli ve 1, 3 mm/dak sabit yer değiştirmeli statik test sistemleriyle elektromekanik üniversal test sistemi (INSTRON, 5982, Bluehill® 2 Software, İngiltere) tarafından yapılmıştır. Bükme testi, elektromekanik üniversal test sistemi (INSTRON, 5982, Bluehill® 2 Software, İngiltere) tarafından yapılan, fikstür uzunluğu=50 ve 1 mm/dak hızda yapılan üç noktalı bükülme testi ile tahmin edilmiştir. Kalıcı deformite aşamasına ulaşmak her testin yaklaşık 6 dakikasını sürmüştür. Kaydedilen diyagramda süneklik de değerlendirilmiştir. Çekme testi ölçümleri için PEEK numuneleri (INSTRON, 5982, Bluehill® 2 Software, UK) cihazı ile 1 mm/dk hızında incelendi ve numune uzaması da kaydedilen diyagramda değerlendirildiirilmiştir.

Mikrosertlik testi; Atatürk Üniversitesi Diş Hekimliği Fakültesi Protetik Diş Tedavisi Anabilim Dalı'nda incelenmiştir. Araştırma laboratuvarında bulunan Vickers Sertlik Ölçme Cihazı (Universal Motion, Inc. No. 208, R Cube, S. No. 116/5/1, Warje Highway, Near Atul Nagar, Pune - 411052, Maharashtra) kullanıldıılmıştır. PEEK numunelerinin yüzeylerine, yüz açıları 136° olan kare tabanlı piramidal şekilli Elmas uç ile 15 saniye boyunca 10 kg'lık bir yük uygulanmıştır. Numune yüzeylerinde oluşan piramit görüntüsü belirlenere köşegen uzunluklarını gösteren D1 ve D2 uzunlukları ölçüldü. Ölçülen değerler ayrıca cihaz üzerinde hesaplanmıştır (Kg/mm2). Tüm farklı PEEK örneklerine mekanik testler uygulandıktan sonra, farklı depolama ortamlarında her gruptan rastgele bir örnek seçildi. Yüzey görüntüleme işlemi FIB/SEM tomografi elektron mikroskobunda (Quanta 3D FEG, FEI, ABD) gerçekleştirildi. Farklı PEEK gruplarına ait kırık şeklinin incelenmesi amacıyla görüntüler x 20.000x büyütme ile izlendi ve görüntüler kaydedilmiştir. Çekme mukavemeti mekanik testi tamamlandıktan sonra kırılma davranışını büyütmek için dört farklı PEEK örneği optik mikroskop altında incelendilenmiştir. Bu numuneler, farklı saklama ortamlarına sahip her gruptan rastgele seçilmiştir. Görüntüleme işlemi, uygun netlik derecesinin elde edilmesi için ZEISS/Scope tarafından 5× 0,13HD çözünürlükte gerçekleştirilmiştir. (A1 Optik mikroskop. Axiocam 503 renkli EC EPIPLAN, Almanya). Dört tedavi grubu arasındaki stres değerlerindeki farkı belirlemek için post hoc testiyle tek yönlü ANOVA (Tukey'in çoklu karşılaştırmaları) yapılmıştır: saf Peek ekstrüzyonu (PPE), saf Peek sıkıştırması (PPC), karbon fiber Peek (CFP) ve cam Fiber Peek (GFP). Ek olarak, farklı saklama süreleri/koşulları sırasında aynı grup (Lar) içindeki stres değerlerini ölçmek için tekrarlanan ölçümler ANOVA ve post-hoc testleri kullanıldı: kuru (kontrol) ve Ringer solüsyonu ortamı ile 1 gün, 1 hafta ve 3 hafta. İstatistiksel anlamlılık düzeyi P değeri<0, 05 olarak alındı.

**Bulgular:** Dört çalışma grubunun tamamında (PPE, PPC, CFP ve GFP), ortalama basınç stresi Ringer çözümünde bir haftadan sonra önemli ölçüde en yüksekti. Her grup içinde basınç stresi, solüsyonun bir gün ile bir hafta arasında saklanmasından sonra önemli ölçüde arttı. Üç grupta (PPE, PPC ve GFP), çözeltide üç haftalık depolamanın ardından basınç stresi biraz azaldı. Dört grup arasında CFP en düşük (102, çözümde bir gün kaldıktan sonra) basınç stresine sahipken GFP grubu en yüksek (188, 6, çözümde bir hafta sonra) basınç stresine sahipti. Dört grup arasında stresi bükmek için net bir model mevcut değildi. GFP grubunun bükülme gerilimi, üç Ringer çözümü ölçümüyle karşılaştırıldığında kuru durumda en düşüktü. Buna karşılık, CFP grubunun bükülme gerilimi, üç ölçümle karşılaştırıldığında çözümde üç haftadan sonra en düşük seviyedeydi. CFP ve GFP gruplarında bükülme gerilimi kuru durum ile çözeltide bir gün sonra önemli ölçüde arttı.Bükülme gerilimi ortalaması, diğer üç ölçüm/saklama koşuluyla karşılaştırıldığında, çözelti içinde CFP grubu için bir gün sonra, PPC grubu için bir hafta sonra ve PPE grubu için üç hafta sonra en yüksekti. Dört grup arasında en düşük bükülme gerilimi CFP'de (çözümde üç hafta kaldıktan sonra 130, 8) bulunurken PPE grubu en yüksek bükülme gerilimine (çözümde üç hafta kaldıktan sonra 195, 2) sahipti. Dört çalışma grubunun tamamında (PPE, PPC, CFP ve GFP), ortalama çekme gerilimi kuru durumda Ringer çözümüne göre önemli ölçüde daha yüksekti. Her grup içinde, çözelti içinde saklandıktan sonra gerilme gerilimi azaldı. Genel olarak PPE ve PPC grupları, özellikle çözelti içinde depolandıktan sonra, CFP ve GFP grubuna kıyasla daha yüksek gerilme gerilimine sahipti. CFP, dört grup arasında en düşük (çözeltide bir hafta sonra 49, 71) ve en yüksek (kuru durumda 92, 86) çekme gerilimine sahipti. Ortalama sertlik değeri, dört grup arasında çözeltide bir gün sonra en yüksek seviyedeydi ve zamanla azaldı. Dört çalışma grubu arasında en yüksek sertlik değerine CFP grubu sahip olurken, hem PPE hem de PPC en düşük sertlik değerine sahipti. Çözümde bir gün kaldıktan sonra CFP grubu en yüksek sertlik değerine (54, 79) sahip olurken, PPE çözeltide üç hafta kaldıktan sonra en düşük sertlik değerine (27, 56) sahip oldu. Farklı PEEK numuneleri üzerinde oluşan kırığın optik mikroskopta (x10) gözlenen kırılma tiplerinin değerlendirilmesi, mekanik testlere tabi tutulduktan sonra kırılma çizgisi paternine göre yapılmıştır. Belirli gruplar arasında bulunan kırık çizgisi paterninde açık bir fark vardı. PPE ve CFP numuneleri mekanik test uygulaması sırasında PEEK malzemesinin düzensizliklerini ve kaybını gösterirken, PPC ve GFP numuneleri mekanik teste tabi tutulduktan sonra daha az düzensiz desen ve PEEK malzemesi kaybı gösterdi; GFP numuneleri en organize kırılma çizgisi modelini gösterdi.

**Sonuç:** Dört ölçüm parametresi (sertlik, çekme gerilimi, basınç gerilimi ve bükülme gerilimi) dört çalışma grubu (PPE, PPC, CFP ve GFP) ve dört saklama koşulu/süresi (kuru durum ve bir gün, bir hafta ve üç) arasında değişiklik göstermiştir. Ringer çözümünde haftalar) aynı grup içinde. Her malzemenin belirli parametrelerde güçlü, diğerlerinde ise zayıf yönleri vardır. Örneğin, CFP grubu yüksek seviyede sertlik ve basınç gerilimine sahipken, düşük seviyede eğilme gerilimi ve çekme gerilimine sahiptir. Bunun tersine, KKD grubunda yüksek düzeyde bükülme ve çekme gerilimi bulunurken, düşük düzeyde sertlik ve basınç gerilimi tespit edildi. Dört çalışma grubu arasında en yüksek sertlik değerine CFP grubu sahip olurken, hem PPE hem de PPC en düşük sertlik değerine sahipti. Ortalama sertlik değeri, dört grup arasında çözeltide bir gün sonra en yüksek seviyedeydi ve zamanla azaldı. Genel olarak PPE ve PPC grupları, özellikle çözelti içinde saklandıktan sonra CFP ve GFP grubuna kıyasla daha yüksek çekme gerilimine sahipti. Dört çalışma grubunun tamamında (PPE, PPC, CFP ve GFP), ortalama çekme gerilimi kuru durumda Ringer çözümüne göre önemli ölçüde daha yüksekti. Her grupta, çözelti içinde saklandıktan sonra çekme gerilimi azaldı. Dört çalışma grubunun tamamında (PPE, PPC, CFP ve GFP), ortalama basınç stresi Ringer çözümünde bir haftadan sonra önemli ölçüde en yüksekti. Her grup içinde basınç stresi, çözeltide depolamanın 1. günü ile 1. haftası arasında önemli ölçüde arttı. Üç grupta (PPE, PPC ve GFP), çözeltide üç haftalık depolamanın ardından basınç stresi biraz azaldı. Dört grup arasında stresi bükmek için net bir model mevcut değildi. GFP grubunun bükülme gerilimi, üç Ringer çözümü ölçümüyle karşılaştırıldığında kuru durumda en düşüktü. Buna karşılık, CFP grubunun bükülme gerilimi, üç ölçümle karşılaştırıldığında çözümde üç haftadan sonra en düşük seviyedeydi. CFP ve GFP gruplarında basınç stresi kuru durum ile çözeltide bir gün sonra önemli ölçüde arttı. Bükülme gerilimi ortalaması, diğer üç ölçüm/saklama koşuluyla karşılaştırıldığında, çözelti içinde CFP grubu için bir gün sonra, PPC grubu için bir hafta sonra ve PPE grubu için üç hafta sonra en yüksekti.

**Anahtar Kelimeler:** Cam fiberler, Karbon fiberler, Mekanik özellikler, PEEK, PEEK depolama.

# ABSTRACT

Polyether ether ketone material is considered as an important thermoplastic material due to its property. larger modulus of elasticity PEEK EM is required. To obtain a high value elastic modulus obtain a tougher hybrid PEEK; It can be reinforced with carbon fiber and glass fiber. In this study, it was aimed to improve some mechanical and physical properties of dental (polyether ether ketone) PEEK. Two different types of fiber will be reinforced into the PEEK material; reinforcement of carbon fibers and glass fibers. Different mechanical properties will be measured at different time intervals.

**Materials and Methods:** In the study; A total of 160 samples were produced (n)=40 used for each test. 4 groups of different PEEK materials were used; extrusion PEEK, compression PEEK, carbon fibers PEEK and glass fibers PEEK (PPE, PPC, CFP and GFP). All PEEK specimens will be tested after dry storage and then retested after incubation in Ringer's solution for 1 day, 1 week and 3 weeks at 37°C. Four different mechanical tests were performed for each PEEK sample; Compression, Bending, and tensile tests will be applied. Hardness test done by Vickers Hardness Tester. Optical microscope and FESEM will be used to investigate the fracture morphology of samples at different test time intervals. ANOVA and post-hoc tests were used for statistical analysis.

**Results**: The results of mechanical strength tests including compression, tensile, bending and hardness tests; PEEK (CFP and GFP) reinforced with carbon and glass fibers, compared to all other PEEK groups. showed higher strength values. Incubation with Ringer's solution at different time intervals affected only the one-week and three-week incubation time values for the entire PEEK sample type. Optical microscope and FESEM examinations showed;Tension and rupture frequency were significantly higher in the reinforcement fibers group (CFP and GFP) than in the other groups ($p<0.05$). No significant difference between the reinforcement fiber groups (CFP and GFP) in terms of fracture and bending patterns ($p>0.05$).

**Conclusion**: Our findings revealed that each PEEK type has its specific meachanical behavior related to each test applied.In the absence of reinforcement fiber, the mechanical test values will be observed as normal pure PEEK material properties. The use of reinforced fiber peek for use in different dental applications has shown greater stress-strain value and will increase the rigidity to a higher level.

**Keywords**: PEEK, Carbon fibers, Glass fibers, Mechanical properties, PEEK storage

# ABBREVIATIONS

| | | |
|---|---|---|
| **AFRP** | : | Armaid fiber reinforced polymers |
| **AlN** | : | Aluminum nitrade |
| **BASF** | : | Badische Aniln and Soda Fabrik |
| **BFRP** | : | Basalt fiber reinforced polymers |
| **BioHPP** | : | Bio high- performance PEEK |
| **Bre-flex** | : | Nylone based thermoplastic material |
| **CFP** | : | Carbon fiber PEEK |
| **CFR** | : | Carbon fiber reinforcement |
| **CNF** | : | Carbon nanofiber |
| **CNT** | : | Carbon nanotubes |
| **CoCr** | : | Cobalt chromium |
| **FDA** | : | American Food and Drug Adminstration |
| **FDP** | : | Fixed dental prosthesis |
| **FDP** | : | Fixed partial denture |
| **FRPs** | : | Fiber reinforced polymers |
| **GFP** | : | Glass fiber PEEK |
| **GNS** | : | Graphene Nanosheets |
| **Gpa** | : | Young modulus |
| **GPa** | : | Giga pascal |
| **HA** | : | Hydroxy apetite |
| **HF** | : | Hydrofluoric acid |
| **HPP** | : | High performance polymers |
| **ICI** | : | Imperial Chemical İndustris |

**ISO** : International Organization for Standerization

**Kgf** : Kilogram force

**MPa** : Mega pascal

**N** : Stress cycle of fatigue limit

**Nicr** : Nickel chromium

**n-$TiO_2$** : Nano-titanium dioxide

**PAEK** : Polyaryletherketone

**PC** : Compression PEEK

**PCL** : Polycaprolactone

**PE** : Extrusion PEEK

**PEEK** : Polyetheretherketone

**PEEK-COOH**: PEEK containing carboxyl groups

**PEK** : Polyetherketone

**PEKK** : Polyetherketone Ketone

**PGA** : Polyglycolic acid

**PI** : Polyimide

**PLA** : Polylactic acid

**PLGA** : Poly lactide coglycolide

**PPC** : Pure PEEK compression

**PPE** : Pure PEEK extrusion

**PPO** : Polyphenylene oxide

**PPS** : Polyphenylene sulfide

**PTFE** : Polytetrafluoroethylene

**PTW** : Potassium titanate whiskers

| | | |
|---|---|---|
| **RAD** | : | Radiation absorbed dose |
| **Sic** | : | Silicon carbide |
| **TCP** | : | Tricalcium phosphate |
| **TFMSA** | : | Trifluoromethane sulphonic acid |
| **Tg** | : | Glass transformation tempreture |
| **Tm** | : | Crystalin melting tempreture |
| **TP** | : | Tranclucency parameter |
| **VHN** | : | Vickers hardness number |
| **Zr** | : | Zircona |

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# 1. INTRODUCTION

Over the last three decades, researchers from various expertise such as physics, engineering, chemistry, biology and medical sciences, are working in collaboration in order to develop new materials for medical implementation. In this context, several materials have been introduced over the last centuries; they developed various brands using metals, ceramics and polymers where they can be used in different parts of the human body. While researchers are choosing and/or developing materials as medical prosthesis, they mostly pay attention to their mechanical and physical properties where they have to be inert in the human body and strong enough to resist to the applied forces (Mushtaq et al., 2021; Harting et al., 2017; Pourkhalil &Maleki, 2022). (PEEK) (ether ether ketone) is considered very important engineering thermoplastics material due to their combination of high strength and toughness, their high thermo-oxidative stability, superior flame retardancy, their biocompatibility and their chemical and wear resistance (Papageorgiou et al., 2019; Elawadly et al., 2017). PEEK has appeared as one of the most important materials and has employments in demanding engineering components such as automotive and chemical process industries, medical materials, etc. (Liebermann et al., 2016; Nobre et al., 2020). The persistent demand of developed manufactures for even greater performing materials, has however led to the emergence of PEEK-based nano composites where nano fillers such as carbon nanotubes and (Nano) fibers, silica, tungsten, hydroxyapatite and grapheme have been used as reinforcing agents in order to evolve the mechanical, thermal and electrical features of the matrix. (Altstaedt et al., 2003; Rong et al., 2010; Lai et al., 2007; Wang et al., 2017).The employments of natural or synthetic fibers in the production of composite materials have revealed significant achievements in many fields such as construction, mechanical, and biomedical (Rajak et al., 2019; Zhang et al., 2019; Rui Ma & Tingting., 2014). The latest research contributes the development of hybrid composites with the combination of natural and synthetic fibers; various reinforced PEEK composites have been developed, such as carbon fiber-reinforced PEEK (CFR-PEEK) and glass fiber-reinforced PEEK (GFR-PEEK) (Papageorgiou et al., 2019; Rahmitasari et al., 2017; Ansari et al., 2020). CFR-PEEK has been of concern to the medical implant community due to its versatility, suitability to the new version of technology, excellent mechanical features, and biocompatibility. This material can be manufactured in many forms with different physical, mechanical, and surface properties (Schwitalla, et al., 2015; Najeeb et al., 2016). Compressive loading tests were preceded with glass fiber-reinforced PEEK (GFR-PEEK) ranged within the bite force of the natural dentitions, and showed adequate fatigue limits (Lee et al., 2012; Ferguson et al., 2006). The use of the hybrid GF-CF fillers in the PEEK composites, that hold their process ability in relation to their filler content, while their mechanical behavior in terms compression, tensile, and bending strength were all enhanced. Moreover, in the current literature there are no other papers reporting on the preparation and improved physico-chemical properties of PEEK reinforced with a combination of short carbon fibers and glass fibers.The number of available studies describing the improvement of the different mechanical and physical properties of the reinforced PEEK material is not sufficient. Further research and further studies are needed to develop the various properties of PEEK material with both an academic and scientific approach. So in this study our hypothesis was that Pure PEEK compression groups (PPC) would show higher mechanical stress degrees than other pure PEEK extrusion groups (PPE). All mechanical stress values for the fiber reinforced PEEK group were recorded to a higher degree than for the pure PEEK groups.

The aim of this in- vitro thesis study was to examine the effect of applying two different types of fibers reinforcement materials; carbon fibers and glass fibers on compressive strength, bending strength, tensile strength, and micro hardness strength. For this purpose the obtained data were collected and analyzed statistically and different results were evaluated and compered.

# 2. GENERAL INFORMATION

## 2.1 The History of PEEK Emergence

PEEK is a type of high-performance special engineering plastics, Aromatic polyketones were first produced in the 1960s and 1970s but did not become commercially available until the early 1980s. In the early- to mid-1970s Raychem Corporation commercially introduced a polyetherketone (PEK) under the Stilan trade name (Goharian et al., 2017). In the 1970s this semi crystalline, aromatic polyether was developed by K. Dahl and due to the strict situation needed to make PEK involved in Friedel–Crafts chemistry and required using very strong solvents such as hydrogen fluoride or an excess of aluminum chloride to retain the polymer in solution (Goharian et al., 2017; Skirbutis et al., 2018; Kassem et al., 2019).PEK was costly to industry and was mostly made for limited use. As shown in Figure (2.1) the synthesis of PEK has a Tm of 364 °C and a Tg of 166 °C. In the 1980s Raychem licensed their patents to BASF and stopped production (Basgul, 2021; Akkan, 2013).


**Figure 2.1** PEK syntheses from tri-mellitic anhydride chloride

In 1977 PEEK was developed by British ICI and industrialized by British Company in the early 1980s (Ling et al., 2020; Stober et al., 1984).

Under milder conditions via aromatic nucleophilic substitution reaction of the potassium salt of hydroquinone and 4,4-difluorobenzophenone, several companies conducted early work on PEEK, which resulted in low molecular weight polymer, owning to crystallization and precipitation from solution(Goharian et al., 2017; George et al., 2011; Schwitalla , and Muller WD, 2013). Rose at ICI (now Victrex Co.) substituted a very high boiling solvent such as diphenylsulfone at temperatures close to the melting point of the polymer and high molecular weight PEEK was obtained. In 1980 PEEK was commercialized under the Victrex PEEK trade name. PEEK has a Tm of 335 °C and a Tg of 145 °C. PEEK is the most widely used member of aromatic polyketones (George et al., 2011; Manuel et al., 2018).

At the end of 1990s, PEEK became an important fulfillment thermoplastic chosen material for replacing metal implant components, in vertebral surgery as a material of the inter-body fusion cage (Biris et al., 2018; Saeed et al., 2020; Kirchner, 2014). In 1998 PEEK that is called the tip of pyramid plastics industry was commercialized for industrial manufacture purposes, it was applied in biomedical field by in-vibio Ltd (UK) (Benakatti et al., 2019). Typically PEEK composite materials have been used as a surrogate for metallic and ceramic implants, which have a higher modulus of elasticity. PEEK was first introduced to the medical field as a fracture fixation, and it remains to be used for this aim. Regardless of many years and improvements, PEEK is still one of the most popular biomaterials on the market due to its multilateral potencies

(Manoukian et al., 2019). (Negin et al., 2012) explored incorporation of calcium carbonate and talc fillers to reduce PEEK's coefficient of thermal expansion. They found that the coefficient of thermal expansion for PEEK could be reduced to values similar to that of the copper metallization, by using the talc filler at 30 wt. %. In addition, PEEK offers similar thermal stability to PI (polyimide), with superior chemical stability (against alkaline media) and substantially lower water absorption than PI.

Interest in PEEK (poly ether ether ketone) is growing due to their successful implementation wherefore PEEK composites have been evolved by adding certain additives to boost the strength and stiffness. Among the various additives; carbon fibers, glass fibers, carbon nanotubes, and Graphene Nanosheets (GNS) have been added into the PEEK matrix for satisfying the requirements in load-bearing areas.

The mechanical properties of PEEK can be tailored by carbon and glass fillers were first applied as reinforcement additives in PEEK and carbon-fiber-reinforced PEEK (CFR-PEEK) is presently studied as components in orthopedic implants (Schwitalla & Mu¨ller, 2013; Jin & Paul , 2019; Muhsin et al., 2018; Rocha et al., 2016). The strength and elastic modulus of the CFR-PEEK composite depend on the percentage, size, length, and orientation of the fibers. Young's modulus of PEEK is 3–4 GPa, which is near but not identical to that of the human bone (Ling et al., 2020; Najeeb et al., 2016; Staniland et al., 1992) Moreover, PEEK can be easily modified by incorporating carbon fibers which may increase the elastic modulus up to 18GPa. Since PEEK's modulus approximates that of dentine and cortical bone, it could also result in the reduction of stresses transferred on the abutment teeth and also the cementation interface, when compared to titanium and other materials (Alexakou et al., 2019; Khaled & Janaideh, 2019; Liao et al., 2008). Different mechanical, physical, and biological properties have been influenced by adding different reinforcement fillers to PEEK that might represent a viable biomaterial not only to replace conventional polymers but also metals, alloys , and ceramics in the field of dentistry( Wu et al., 2021; Schwitalla et al., 2015).

## 2.2 Synthesis of PEEK

Poly ether ether ketone (PEEK) is an aromatic, semi-crystalline linear thermoplastic polymer with chemical formula (–C6H4–O–C6H4–O–C6H4–CO–) n(Panayotov et al., 2016) (Qin et al., 2021). Figure (2.2)

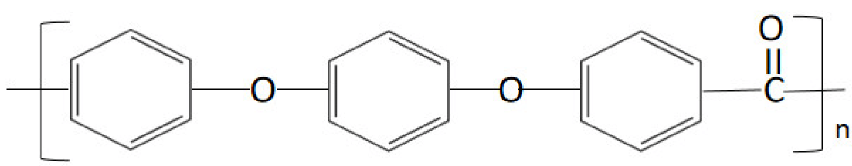


**Figure 2.2** Chemical formula of poly ether ether ketone (PEEK) Poly aromatic semi-crystalline thermoplastic polymer.

As a member of the poly aryl ether ketone (PAEK) family, PEEK was developed from bisphenol salts and aromatic di-halides via nucleophilic substitution. With the action of Williamson ether, the bisphenol salt was produced in situ from bisphenol with either added sodium or added alkali metal carbonate or hydroxide (Qin et al., 2021; Xu, et al., 2015). This chemical formula provides the material with stability at high temperatures (exceeding 300 ◦C), resistance to chemical and radical damage, greater strength (on a per mass basis) and compatibility with reinforcing agents such as glass and carbon fibers (Gonzalez et al., 2015; Krolikowski et al., 2020; Yilmaz et al., 2019).

### 2.2.1 Synthesis approach of PEEK

#### 2.2.1.1 Synthesis of Neat PEEK

The synthesis of neat PEEK was firstly accounted by (Colquhoun and Lewis, 1988) by mean of electrophilic exchanging chemical reaction, which is referred to Friedel- Crafts acylation. They melted 4-phenoxy benzoic acid in the solvent of TFMSA, in the presence of small amount of 1, 4-di (4-carboxyl) phenoxy benzene or 1, 4- diphenoxy benzene to synthesis neat PEEK successfully (Yang et al., 2020; Ma J. et al., 2019; Colquhoun & Lewis, 1988) Figure (2.3). Unfilled PEEK is available as tan pellets, extruded rods, or powder. Figure (2.4)

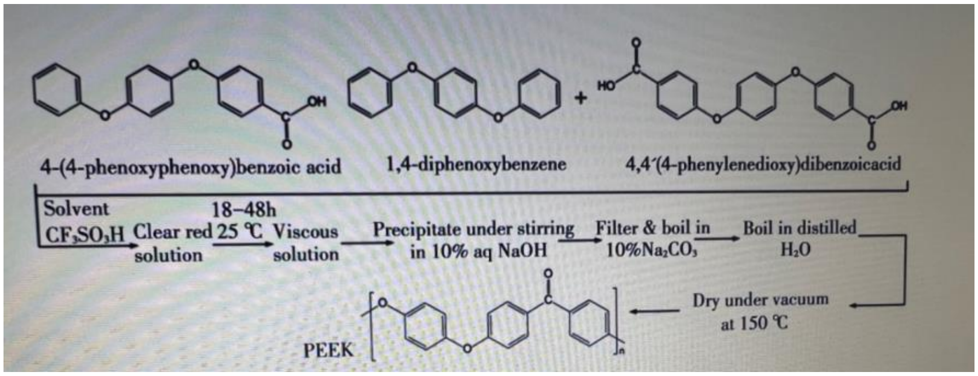


**Figure 2.3** Synthesis Pathway of PEEK in TFMSA

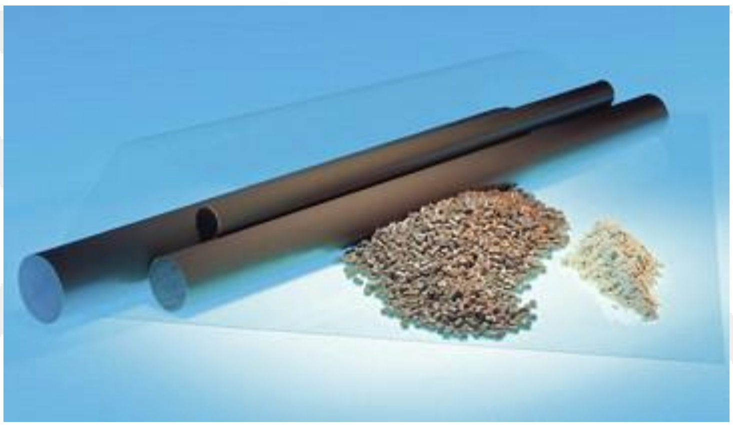

**Figure 2.4** Powder, pellets, extrude rod, and film of unfilled neat PEEK

### 2.2.1.2 Synthesis of Modified PEEK

In the neat monomers, functional bodies have been applied or other functional groups can also be chosen to produce the modified PEEK (Yang et al., 2020). New type of PEEK has been synthesized by the function of carboxyl groups as garnished PEEK (Martínez et al., 2017) Figure (2.5).

PEEK also can be obtained by containing propargyl groups through the reaction ofhydroxyl to produce PEEK; this will efficiently increase thermal stability, fracture strength, mechanical improvement for high performance engineering material (Karthikeyan et al., 2019; Eltombashki et al., 2019; Sadek, 2019; Muhsin &Salim, 2020).Another type of modified PEEK can be synthesized by applying 9- diarylflourene- containing biphenol and naphthalene-containing dihaloarene; flourene based PEEK will be produced ( Han et al., 2019; Lethaus et al., 2012; Rinaldi et al., 2018).

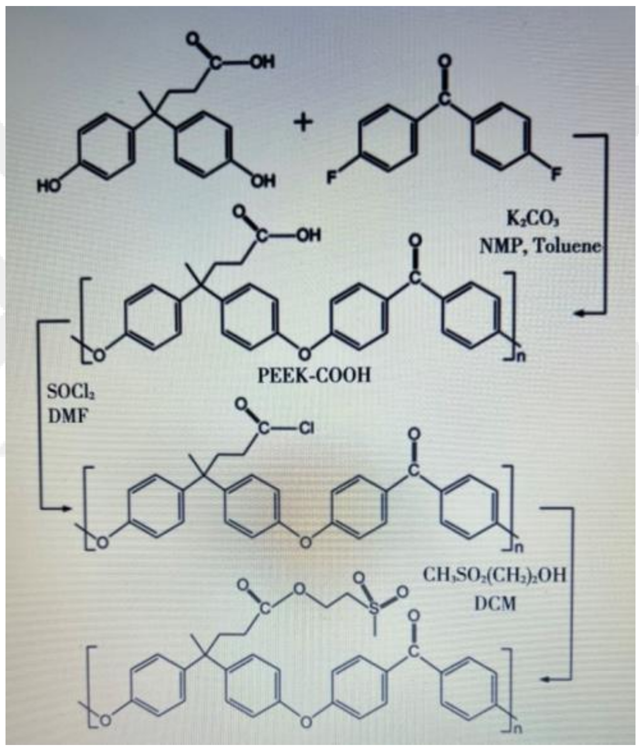


**Figure 2.5** Synthesis pathway of PEEK- SO2 through post functionalization of the corresponding PEEK –COOH

## 2.3 Classification of PEEK Composite

For biomedical application, PEEK composites can be classified into three categories; (i) conventional composites, (ii) bioactive composites and (iii) drug-release systems (Harting et al., 2017).Other classification of PEEK composite has been applied according to the size of the saturated bioactive materials; the conventional PEEK composites and the Nano-sized (<100 nm) PEEK composites. (Rui & Tang, 2014; Bakar et al., 2003; Bakar et al., 2003).

### 2.3.1 Conventional Composites

Fiber-reinforced materials are the most proper composites of PEEK. The choice of the fiber according to material, geometry, and direction relies on the application prospect. Typical materials are carbon-and glass fibers. Those fibers are used to increase the stiffness of PEEK. It is also possible to combine carbon nano fibers (CNF) with PEEK to increase the strength and generate conductivity, especially interesting for electrical devices. Another possibility to generate conductivity is to add carbon nano tubes (CNT). CNTs also improve mechanical properties and can be used with PEEK for aviation applications. Some other filler are also used as additives, as potassium titanate whiskers (PTW). This composite possesses much better properties than neat PEEK (Harting et al., 2017; Goyal et al., 2013; Awaja et al., 2012; Wang et al., 2010).

This composite has much greater advantages than neat PEEK. The strength and rigidity are increased and the temperature of crystallization is decreased. This could be a major usefulness for dental applications, because the lifetime of an implant will be increased and also it is possible to lighten up the neat PEEK to get a more natural look of an implant (Eschbach, 2000; Kurtz& Devine, 2007; Briem et al., 2005).

PEEK combined with $TiO_2$ and composites with crystalline-silica $SiO_2$ are two examples of PEEK composite; $SiO_2$ gives some important advantages for the composite. The thermal stability and the char yield are increased with $SiO_2$ and the positive effect of anti-inflammatory. (Briem et al., 2005; Besch et al., 2005).it is also possible to produce PEEK composites with ceramics, for example, aluminum nitride (AIN) (Harting et al., 2017).

### 2.3.2 Bioactive Composites

Those composites are used to obtain a better combination of implants and to get better interface between bones and PEEK-composites (Goyal et al., 2011). One example for such a composite is the combination of PEEK with hydroxyapatite (HA) Particles. HA is a bio ceramic with a good binding ability to bones. This development of bioactivity is a big advantage for medical applications, but the challenge is the suitable mixing with PEEK. Compared to pure PEEK the mechanical properties (tensile strength and strain to fracture) are lower and implants would break earlier than bones (Sturz et al, 2015; Goyal et al., 2011; EL-Wassefy & Ghorab. 2019) to maintain the mechanical properties of PEEK and improve the bioactivity it is possible to use HA nanoparticles (Rui & Tang, 2014; Xie et al., 2010). Another example of a bioactive PEEK-composite is the combination with tricalcium phosphate (TCP).The current problem with using β-TCP is that the bioactivity improvement is not proven yet. At the moment the proliferation of osteoblasts is not improved compared to pure PEEK, and there is no concentration-dependent decrease of proliferation (Ma et al., 2012). This requires more basic research.

### 2.3.3 Drug-Release Systems

The emergence of drug-release systems was gained in the researches by the application of soft synthetic biodegradable polymers, e.g. poly (lactic acid) (PLA), poly (glycolic acid) PGA, poly (lactide-co-glycol ide) (PLGA) or poly (ε-caprolactone) (PCL), whose main benefit can be found in the complete metabolization by the human organism ( Ma & Tang,2014). The diffusion of drugs will occur either by physical mechanism that is based on the diffusion of a drug molecule through a certain coating or matrix, by chemically controlled drug-release systems operate that working by polymer chain degradation, the rupture of chemically bound drugs or biomolecules or swelling of hydrophilic polymer carriers (Harting et al., 2017). For PEEK, these systems have not been spread so far, may be due to the tough processing conditions, like high melting temperature and solubility only in strong acids as HF and $H_2SO_4$ ,which can be hardly resisted by bioactive reagents. However, the establishment of a drug-delivery system on PEEK by the precipitation of a polymeric drug-release coating is possible (Ma & Tang, 2014; Almasi et al., 2016).

### 2.3.4 Nano-Sized PEEK Composites

Nanotechnology was applied by material scientists to solve the problem of de-bonding between HA filler and PEEK matrix in the conventional HA/PEEK composite (Rui & Tang, 2014). In 2010 Wang and his colleagues prepared HA/PEEK nano composites by a compounding and injection molding process. They found that this novel HA/PEEK nano composite demonstrated favorable mechanical properties and a high surface HA content, with no debonding occurred between the well-dispersed HA nanoparticles and the PEEK matrix (Wang et al., 2010; Wang et al., 2011). Other studies also found that there is an aggregation of nanoparticles during the fabrication of nanoparticle-reinforced thermoplastics (Shi et al., 2005).

To overcome the clustering of HA nanoparticles during manufacturing; In this in situ synthetic process, HA particles were first mixed into PEEK oligomers with short chains and a low viscosity and good wetting and contact were achieved between HA and PEEK (Ma et al., 2012; Barkarmo et al., 2019). The strong bonding between HA and PEEK has been referred mainly to physical factors such as the mechanical engagement between PEEK molecules and the HA surface (Rui & Tang, 2014). Webster and his team have carried out a great deal of research on the bioactivity and biocompatibility of nanomaterials. They found that nanostructured materials may boost osteoblast adhesion, proliferation, differentiation, and stimulate new bone growth compared to conventional materials (Webster et al., 1999; Webster et al., 1999; Gutwein & Webster 2002; Webster et al., 2000; Webster et al., 2001). Therefore, promoting PEEK composites reinforced with nano-sized bioactive materials is a promising strategy for both mechanical and biological benefits improvement. A study invented n-$TiO_2$ reinforced PEEK composites (n-$TiO_2$/PEEK) and studied the bioactivity of these nanocomposites in vitro and in vivo (Wu et al., 2012). The effect of surface morphology or roughness was also investigated. In vitro tests showed that n-$TiO_2$ promoted cell attachment and improved osteoblast spreading. In vivo tests showed that n-$TiO_2$ improved bone regeneration around the implants compared with pure PEEK, as assessed by micro-CT and histological analysis. Thus, n-TiO2 was considered to significantly improve the bioactivity of PEEK, especially for composites with rough surfaces (Sugita et al., 2011).

## 2.4 PEEK Processing

PEEK has a high temperature thermoplastic material that supplied in a range of viscosities according to the processing method, (Saja Muhsin 2016) table (2.1).It is processed by using different conventional techniques including machining injection molding, extrusion, compression molding, and film and fiber production. (Ryan Roeder, 2019).

**Table 2.1**: Traditional plastic processing methods for PEEK-OPTIMA

| PEEK-OPTIMA | VISCOSITY | PROCESSING METHOD | USES |
|---|---|---|---|
| LT1 | High | Machining Injection moulding | Medical device components |
| LT2 | Medium | Extrusion | Thin walled parts (tubing) |
| LT3 | Low | Injection moulding | Thin walled parts |

Whereas OPTIMA is a trade name for biomaterial PEEK, $LT_1$ referred to standard grade which is available in powder form (P) for compounding, or as granules (G) for injection moulding, $LT_2$ is an optimized grade for melt strength and viscosity and is recommended for tubing processes; and $LT_3$, is a high flow grade for injection molding (thin walled parts). However, granules are the only form commercially available for implantable grade PEEK- OPTIMA. Milling or grinding the granules is the only way to have the required particle size to get the powder type of medical grade PEEK polymer. As a quality control measure, PEEK-OPTIMA undergoes melt filtration to ensure purity and biocompatibility before the polymer is granulated into cylindrical pellets for marketing (Kurtz A, 2011; Saja Muhsin 2016).

### 2.4.1 Injection Molding

This technique is a catchy processing method for mass manufacturing of PEEK components. The PEEK pellets or granules ideally have been used for the injection molding method. In a machine, these are casting into a hopper and automatically introduced into a heated screw. The molten PEEK flows into a heated mold once the heated screw has melted and pressurizes the molten polymer (Saja Muhsin, 2016).Then, automatically the molded model is expelled from the mold after PEEK component solidification. Commercially, the injection molds were designed and optimized for each section involved taking into account the properties of the section's geometry and flow, and the pressure strength of the molten polymer injection system, the injection molding of PEEK requires barrel and nozzle temperatures of about 400 °C (Kurtz a, 2011; Schwitalla et al., 2016).

Since failure to fulfill the required mold temperature results in rapid cooling and insufficient time for the crystallization of this part. But it seems possible to increase the crystallinity of amorphous or low-crystallinity PEEK by means of overheating which may lead to deformity and dimensional changes and this should be taking into account during PEEK melt temperature (Lee & Porter, 1987).

No studies have admitted the influence of mold temperature on the PEEK's physical and mechanical properties in relation to the PEEK provided in industry as a final product. According to Kurtz and to Invibio's processing guide injection molding parameters such as mould temperatures, design, and geometry of the parts could affect PEEK flow and hence the uniformity of the matrix parts, table (2, 2) (Kurtz, 2011; Saja Muhsin 2016).

**Table 2.2:** Typical injection molding temperature profiles for PEEK-OPTIMA

| Material | Rear T (°C) | Middle T(°C) | Front T(°C) | Nozzle T(°C) |
|---|---|---|---|---|
| Unfilled PEEK | 355 | 365 | 370 | 375 |
| Filled PEEK | 355 | 365 | 370 | 375 |
| CFR-PEEK | 365 | 380 | 390 | 395 |

### 2.4.2 Extrusion

The making process by which long stock shapes like rods, sheets, and mono filament fibers are produced is called extrusion. This technique is similar to the injection molding system regarding the use of PEEK pellets and granules as a starting raw material but the exceptional step in this process is that the molten polymer is pressurized and forced through a heated die, and cooling to room temperature takes place gradually along an extrusion line (Thomas Hoskins 2015). The predominant system for the manufacturing of medical device components is the extrusion of PEEK stock shapes followed by a machining process. This material can be obtained in rods ranging from 6 to 150 mm in diameter and plate with a thickness of 40 mm. However, since the extrusion thermal scheme and overheating of stock shapes show slight variations in the mechanical properties of when compared with the injection molded method (Invibio, 2004; Kurtz, 2011).

### 2.4.3 Compression Molding

Stock shapes such as plates and thick sheets are provided by this molding technique. It consists of two heated platforms, and the resin powder or granules are placed in the lower platform on which a depression for the plate or sheet is prepared. Then, the two metal plates are pressed together and heated to unite the resin (Kurtz, 2012). This process is typically used in the production of extremely thick sections industrial components or for low volume manufacture. It is relatively more economical than the injection molding process; however, a time consuming long cycle. In addition, this technique needs the use of a heat press, oven, and a tool of low grade steel or metal to withstand the level of stress, shear, and involved loads. Furthermore, the use of granules and pellets results in weak points in the final components since they represent granular boundaries in the molded parts (Lu et al., 1996; Maharaj & Jamison 1997).Hence, the fine powder grade of PEEK polymer has been recommended for the compression method. Nevertheless, this process may offer higher crystallinity and tensile strength than the injection method (Wang et al., 2020; Zanjanijam et al., 2020; Perng et al., 1999).

### 2.4.4 Film and Fiber Production

The PEEK fiber and film with its mechanical performance and the purity of form supply promise for usage in medical devices, especially for implantable-grade application. PEEK film made up of threadlike parts is produced by processing extruded polymer using proper stain and haul-off equipment, and then the film can be handled and stored for the next procedure (Stawarczyk et al., 2015; Fischer et al., 2004). A comprehensive review of the processing guidelines of PEEK high performance polymer explained that PEEK film can be provided in either crystalline or amorphous form. (Processing Guide of Victrex.2019).This can be accomplished by controlling the temperature of the casting drums in the haul-off equipment, although the fully amorphous film becomes more challenging as the thickness of the PEEK film increases. Furthermore PEEK monofilament can be produced by drawing the PEEK extrude after extrusion. The produced monofilament has showed toughness, extreme orientation, and controllable diameter characterizations (Rokaya et al., 2018; Huang 2014; Sorte et al., 2017).

### 2.4.5 Powder Coating

A Recent study has showed that powder coating is considered as type of PEEK processing technique; VICTREX PEEK in fine powder form may be used to coat metal substrates. Thick coats of up to 2 mm (0.078 in) can be applied by electrostatic spraying or fluidized bed coating. Coatings as thin as 25 µ (0.001 in) may be obtained using dispersion techniques. Regardless of whether a part is coated by the electrostatic or dispersion process, the appearance of the coating can be modified via the last heat cycle.  Three surface finishes are possible; standard crystalline (air cooled), amorphous (quenched in cold water) or gloss (quenched in cold water and subsequently annealed to achieve a gloss finish (Alqurashi et al.,  2021; Wong et al., 2009; Xunzhi Yu et al., 2020; Rikitoku et al., 2019).

## 2.5 Properties of PEEK

### 2.5.1 Physical, chemical, and Mechanical Properties

PEKK offers excellent physical and mechanical properties, such as compressive strength and melting temperature in comparison to other polymeric materials (Alqurashi et al., 2021). In comparison to PEEK (pure and glass-reinforced), PEKK shows superior mechanical properties in terms of compressive tensile, and flexure strength (Guo et al., 2012).  Some products of PEKK like PekktonÒ Ivory (Cendres + Métaux, SA, Switzerland), has shown 80% higher compressive strength compared to un-reinforced PEEK, while others with the addition of titanium dioxide ($TiO_2$) in PEKK, increases the hardness and wear resistance (Alsadon et al., 2020).

The possibility of using PEEK as restorative material has been raised due to the shock absorbance with suitable strength (65 MPa) and fracture resistance properties (Han et al., 2016). The PEKK has similar compression strength with a lesser modulus of elasticity compared to dentin (Alqurashi et al., 2021). Tables 2.3 and 2.4 are showing that, the elastic modulus of PEKK is comparable to those of bone. Hence, PEKK can be used as a dental implant biomaterial for excellent mechanical properties and preferable stress distribution (Guo et al., 2012; Gowda et al., 2018).

**Table 2.3:** Comparison of mechanical properties between PEKK and some human Structure.

| Materials | Tensile strength (MPa) | Elastic modulus (GPa) | Flexural strength (MPa) |
|---|---|---|---|
| **PEEK** | 115 | 5.1 | 140-200 |
| **Cortical bone** | 104-121 | 14 | 50-150 |
| **Cancellous bone** | 10-20 | 1.37 | 10-20 |
| **Dentine** | 104 | 15 | 212.9 |
| **Enamel** | 67.5 | 40-83 | NA |
| **Titanium** | 954 | 102-110 | 65 |

**Table 2.4**: Mechanical properties of PEKK and other prosthetic materials

| Properties | PEEK | PEKK | Titanium | PMMA |
|---|---|---|---|---|
| **Tensile strength (MPa)** | 100.69 | 115 | 240–890 | 48–62 Mpa |
| **Elastic modulus (GPa)** | 3.5 | 5.1 | 103–114 | $3.8 \times 10^3$ |
| **Flexural strength (MPa)** | 163.88 | 200 | 65 | 107-117 |
| **Compressive strength (MPa)** | 118–169 | 246 | 130-170 | 76 Mpa |
| **Melting temperature (°C)** | 334–350 | 363–386 | 1650–1670 | 160 |
| **Hardness** | 26–29 VHN | 252 | 90 VHN | 89–95 |
| **Water absorption(mg/mm$^3$)** | 0.1–0.5 | 8.7 | 0.04 | 1–0.3 |
| **Density (g/cm$^3$)** | 1.3 | FEFF1.3 | 4.4–4.5 | 1.16–1.18 g/cc |

Lately, the fatigue behavior of PEKK have been evaluated by researchers; with bilayer crowns in comparison to zirconia and nickel chromium-based crowns they found that The fatigue limit of PEKK (754 N) was reported remarkably higher compared to zirconia (422 N), and nickel-chromium (586 N) (Fuhrmann et al., 2014).In like manner, the fatigue limit of PEKK composite veneered molar crowns is also comparable with the cobalt-chromium and poly methyl methacrylate (PMMA) (750 N). According to Burke's classification, the fracture code of PEKK was distributed between code one and two while Zr and NiCr exhibited code one and distribution between code 1 and 4, respectively, when subjected to loading below the group's fatigue limit. (Wang et al., 2010; Rajagopal et al., 2021; Beuer et al., 2008).

The addition of carbon fiber makes the material with better mechanical, Chemical and thermal resistant. PEEK with its low specific weight can be used to construct very lightweight prosthesis which will provide high patient satisfaction and comfort (Wiesli & Özcan, 2015).Therefore, PEEK has been registered by many researchers with the following eminent performance: (Ling et al., 2019; Zsidai & Kátai, 2016; Zhong-Wen, 2002; Im et al., 2010; Sasuga & Tagivara, 2011; Muhsin et al., 2019).

#### 2.5.1.1 Excellent Heat Tolerance

PEEK can be used at 250°C for a long period of time, with an immediate working temperature of up to 300°C, and almost doesn’t show decomposition at 400°C for a short period of time. Compared with other high-temperature resistant plastics such as polyimide (PI), polyphenylene sulfide (PPS), polyphenylene oxide (PPO), and polytetrafluoroethylene (PTFE), and its upper limit of operating temperature is higher by nearly 50°C.

#### 2.5.1.2 Dimensional Stability

PEEK has good dimensional stability, high rigidity, and low linear expansion coefficient, which is very close to metallic aluminum. Furthermore, PEEK also has a good creep resistance, which can withstand great stress during operation, so, there is no significant elongation due to the time extension.

#### 2.5.1.3 Hydrolysis Resistance

PEEK has resistance to chemical damage from water or high pressure water vapor.Under high temperature and pressure conditions, PEEK components in an aqueous environment can operate seamlessly with maintaining good mechanical properties. If soaked in water at 100°C continuously for 200d, its strength remains almost unchanged. PEEK has a very low water absorption, which can be used in pressurized hot water or steam at 300°C.

#### 2.5.1.4 Good Flame Retardant Performance

PEEK can reach up to the grade of UL94V-0, it has the properties of self-extinguishing, and releases less toxic gases with less smoke under the flame conditions.

#### 2.5.1.5 Strong Radiation Resistance

Since PEEK has a very stable chemical structure, its components can also operate well under high doses of ionizing radiation; it has strong resistance to gamma irradiation, excelling that of polystyrene, which is the most resistant to radiation in general-purpose resins (Green and Schlegel, 1998; Dohiema and El Afandyb 2019). It can be used to make a high-performance wire that maintains good insulation ability even when the γ-irradiation dose reaches 1100 Mrad.

#### 2.5.1.6 Toughness

PEEK is a plastic that has balanced toughness and rigidity. It has the most eminent fatigue resistance to alternating stress among all plastics, comparable to that of alloy materials.

#### 2.5.1.7 Friction and Wear Resistance

PEEK can maintain high wear resistance and low coefficient of friction at 250°C. It can provide excellent wear resistance under various conditions of pressure,temperature, speed and relatively rough contact surface; the abrasion loss of the grinding between the polyetheretherketone pure resin and H10 wheel material is of 2.7×10-4g; while the abrasion loss of the grinding between polyetheretherketone pure resin and S17 wheel material is of 9.7 × 10-4g.

#### 2.5.1.8 Excellent Chemical Resistance

PEEK, whose corrosion resistance is similar to nickel steel, can resist the corrosion of most chemicals even at high temperatures. PEEK can dissolve only the concentrated sulfuric acid under normal conditions.

#### 2.5.1.9 Good Processing Performance

PEEK has a good processing performance. Although it is a super high temperature resin, it can be processed by various processing methods such as injection molding, extrusion molding, compression molding, blow molding, melt spinning, rotational molding, powder coating due to its properties of high temperature fluidity and high thermal decomposition temperature (Elmoutawakkil and Bellemkhannate, 2020).

#### 2.5.1.10 Solubility

PEEK has a water solubility of 0.5 w% and it cannot be chemically affected by long-term water exposure, even at temperatures of up to 260°C. In a study of the physical and mechanical characteristics of PEEK and other modern esthetic CAD/CAM polymer materials, PEEK showed lower solubility and moisture absorption, while hardness values were comparable to those of PMMA materials (Monich et al., 2017: Zhoua et al., 2014).

### 2.5.2 Biological properties

PEEK reveals an excellent biocompatibility and it has been presented as promising alternative material for long-term orthopedic applications over titanium (Bubik, 2017; Wu et al., 2012). It has been approved by the FDA for oral-maxillofacial and spinal surgery (Adamzyk et al., 2016). PEEK also is being used widely in dentistry as a prosthetic and implant biomaterial. It offers metal-free restorations and helpful in patients with allergies (Kumar &Murgod, 2020; Ma et al., 2020). Many researchers investigated osteointegration in PEKK and PEEK in terms of chemistry and surface microstructure. It was reported that the other ketone group in PEKK increases the ability of surface chemical modification. With more ketone groups, the presence of -SO3H will be more on PEKK than PEEK (Yuan et al., 2018; Ma et al., 2014; Li et al., 2020). This leads to greater surface area, complex surface topography, and micro rough surface, which will affect the cell behavior and osteointegration (AlOtaibi et al., 2020). The surface modification by increasing the porosity and incorporation of HA had a positive impact on the osteointegration property (Najeeb et al., 2015; Gu et al., 2021). Several in vitro and animal studies have been performed confirming the biocompatibility of PEEK material to evaluate the oral mucosal integration at implant abutments made from PEEK material (Richardson, 2010).

(Williams et al., 1987) reported the first animal studies of PEEK in the literature. Neat PEEK and carbon-fiber reinforced samples were subcutaneously implanted in rabbits for 6 months and submuscularly implanted in rats for 30 weeks. Williams stated that the growth and attachment of osteoblasts and fibroblasts to PEEK was evaluated in a series of cell culture experiments. Khoury et al 2013 enhanced the bioactivity of PEEK without any modification in chemical structure and without addition of any bioactive substances by using Accelerated Neutral Atom Beam (ANAB) technique which employs intense directed beams of neutral gas atoms that showed enhanced growth of osteoblastic cells determined by cell proliferation assay and microscopy and bone tissue formation in rat model (Adell et al., 1981; Yua et al., 2020). Wanqi in 2020 found that 30%-CPEEK composites were fabricated and modified with amino groups. The surface characterization of composites was investigated, and the in vitro biological performance in terms of cell adhesion, proliferation, and osteogenic differentiation was evaluated by culturing MG-63 cells on the samples (Nizam Uddin et al., 2019).

### 2.5.3 Aesthetic Properties

Some studies of aesthetic features for PEEK material showed that the greyish brown color of PEEK, it is not suitable for monolithic aesthetic restorations on anterior teeth. Thus more aesthetic material like composite and zirconia proposed to be used as coating materials to improve aesthetics when used as direct or indirect restorative material rather than only as implant and framework material (Skirbutis et al., 2017).Veneering or glazing is challenging due to the stable chemical structure and the unreactive and inert surface characteristics of thermoplastic high-performance polymers (HPP) (Porojan et al., 2021; Lümkemann et al., 2020; Stawarczyk et al., 2013; Ates & Caglar, 2018).

### 2.5.4 Optical Properties and color stability

The optical properties such as translucency and opalescence are critical factors for aesthetics and to mimic the natural appearance of the restorations. Translucency Parameter (TP) is the difference in translucency at a uniform thickness measured by a spectrophotometer device over white and black backings (Stawarczyk et al., 2016). If the material is completely opaque, the TP value is zero. As the TP value increases, the translucency of the material also increases. Opalescence is defined as a scattering of wavelengths of visible light, and as a result, an object appears bluish and orange/brown in the reflected and transmitted color (Sen, & Us, 2018; Della Bona et al., 2014; Vichi et al., 2014; Paravina et al., 2002; Lee & Lu, 2005). The processes of PEEK discoloration still remain to be explained. Color stability significantly depends on surface roughness and surface-free energy during surface processing (Heimer & Schmidlin, 2007). The glazing of PEEK has a favorable effect on surface roughness and opalescence. Artificial ageing damages the color stability and roughness of PEEK; increased surface roughness adversely affects the strength of the materials (Porojan et al., 2021).

## 2.6 Applications of PEEK

### 2.6.1 Application areas of PEEK in the medical field

In the healthcare manufacture, PEEK provides advantages such as biocompatibility, durability and excellent resistance. It is preferable and convenient material for pivotal applications such as orthopedic implants, spinal implants, surgical equipment and dental equipment (Haleem & Javaid, 2019). Table 2.5 is a descriptive of some pivotal application areas of PEEK in medical field. PEEK implants systems are gaining popularity in recent years because it's compatible to contact with the human body and is also incorporated into the manufacturing of active implantable devices (Ferguson et al., 2006; Rahmitasari et al., 2017). In the last few years, it is assumptive widely in the human body more than other traditional plastic and metals. As PEEK is a hard, stiff and strong polymer so it has good wear properties that make PEEK is also useful for repairing soft tissues and grafts (Gallagher et al., 2018).

**Table 2.5:** Application areas of PEEK in the medical field

| Application areas | Description |
|---|---|
| Orthopaedic surgery | Due to high strength, PEEK is used for various orthopaedic applications like hip replacements, hip resurfacing and construction of femoral component |
| Medical tubing | PEEK is suitable for medical tubing applications; It has high purity, organic polymer with good resistance. It is also having flexural modulus and requisite tensile strength<br>PEEK tubing is used medical where high rigidity required |
| Spinal implants | For spinal implants, PEEK offers many advantages as compared to metal. Include modulus close to bone which is the ultimate solution for the surgeon |
| Bone screws and pins | For the manufacturing of screws and pins, PEEK has excellent capability in required strength used to hold the bone in place. Increase recovery chance of patient in less time |
| Minimally invasive fusion surgery | Used for innovation in spine surgery with minimally invasive fusion. It also reduces nerve root retraction and tissue dissection |
| Face reconstruction | Applicable for the reconstruction of the face of the individual patient with high strength, durability and stiffness. These implants are easily workable, non-porous for facial reconstruction |
| Heart valve and stents | Used for the manufacturing of heart valves and stents having high durability. Efficient for the cardio patient during surgery and performed high comfort as compared to other materials |

### 2.6.2 Application Areas of PEEK in Dentistry

PEEK as a promising material thanks to its convenient physical and mechanical properties; it has been used in the different fields of dentistry (Siewert et al., 2019).

The areas of use in dentistry are (Nizam et al., 2014; Zoidis et al., 2017; Najeeb et al., 2015):

I. implants

a) Implant

b) Implant supported bar

c) Abutment

d) Temporary abutment

e) Healing cap

II. Fixed prostheses

III. Endocrowns

IV. Removable dentures

V. Maxillofacial prostheses

VI. Orthodontic wire

Table 2.6 showing Use of PEEK in Prosthodontics: Advantages, Disadvantages.

#### 2.6.2.1 PEEK in Implants

The high-performance with iso-elastic characteristics of PEKK has potential applications in oral implantology (Schwitalla et al., 2015). PEKK has the advantage of being enough strength, lightweight, wear resistance, and elastic modulus close to that of dentin. Dental implants fabricated from thermoplastic resins have also shown acceptable results for the percentages of bone contact (Stawarczyk et al., 2014; Cook & Dawicki, 1994). The elastic modulus of PEEK is close to the elastic modulus of cancellous and cortical bone and is 3-4 GPa. In this way, peri-implant stress accumulation and resorption can be prevented due to the incompatible elastic modulus of conventional implants with cortical bone (Nizam et al., 2014; Geetha, 2020).

PEKK is metal-free and presents an alternative material to titanium implant; the advantage of PEKK abutments is adjustable and compatible with various veneering materials and can be used as a framework for an implant-supported prosthesis combining the PEKK attachment system with titanium can be a potential material to provide long-term retention in implant prosthesis (Schwitalla and Müller, 2013; Alsadon et al., 2020; Amelya et al., 2019; Passia et al., 2016).

The PEKK framework shows less stress to the implant and tissue under compressive stress compared to tensile stress. Therefore, the PEKK framework should be limited in some areas are they are resilient framework(Lee et al., 2017). Nonetheless other studies concluded that although unmodified PEEK may have certain limitations as implant material, various surface modifications can be done to increase the bioactivity and use it as implant material. A number of studies have been carried out to improve the wettability of the material by changing the surface properties. (Deshpande et al., 2019; Al Omar et al., 2021).

Nizam et al. examined the alumina-coated PEEK samples with oxygen plasma etching and laser modification, according to the data obtained, wettability is increased for implantation PEEK is coated with hydroxyapatite and encouraging results were achieved. High-strength polymer materials such as PEEK are also recommended as abutments in many implant-supported restorations (Wang et al., 2021; Salernitano & Migliaresi, 2003; Özden & Demir, 2020). Less bacterial biofilms are observed compared to titanium and zircon abutments.A research has shown that PEEK can be used as an immediate definitive abutment and framework material (Bathala et al., 2019).

According to Eduardo et al. when used as an abutment, PEEK exhibits lower fracture resistance than titanium alloys. It was concluded that reinforced PEEK abutments are a good alternative to titanium abutments in an animal study evaluating the effect of PEEK and titanium abutments on biological width and soft tissue stability (Eduardo et al., 2016). Other studies showed that fatigue strength for BioHPP is very high and can be used as super structure for implants (Ying et al., 2019). Thus PEEK can be used as superstructure for implants retained prosthesis. From an aesthetic point of view, gray reflections of titanium implants under the mucous membrane are undesirable in the absence of adequate tissue support. Due to these negative properties of titanium implants, zirconium dioxide ceramic implants or composite polymers implants have been introduced to the market as an alternative to titanium implants in recent years (Livaditis & Livaditis, 2013).

**Table 2.6**: The use of PEEK in Prosthodontics: Advantages, Disadvantages

| Use of PEEK in Prosthodontics | Advantages | Disadvantages |
|---|---|---|
| As RPD framework Peek containing fillers (20% ceramic fillers) (BioHPP) | • Lighter weight<br>• Aesthetic<br>• Less torquing forces on abutment tooth | PEEK direct retainers have lower retentive force compared to Co–Cr retainers |
| As crown and FPD PEEK reinforced with other inorganic fillers | • High fracture resistance<br>• High patient comfort and acceptability<br>• Better marginal fit of provisional crowns | Grayish brown color. Should be layered with composite to achieve aesthetic results. |
| As Obturator | • Lighter weight<br>• Biocompatibility<br>• Ease of polishing | - |
| As intra radicular post core material | • High fracture resistance<br>• Less chances of root fracture | - |
| As superstructure for implants BioHPP appears to be appropriate for implant superstructures | • High Fatigue strength (1200N) | - |
| As dental implant material | No common consensus in the literature with respect to osseointegrating properties of PEEK when compared to conventional implant materials such as zirconia and titanium | |

### 2.6.2.2 PEEK with Fixed Prosthesis

PEEK is a recommended material for fixed prostheses (Stawarczyk et al., 2015; Stawarczyk et al., 2014). Metal supported ceramics have been used for many years with disadvantages like corrosion, allergic potential and poor light permeability. PEEK material possess higher biocompatibility than metal-based ceramics that can be more easily repaired, does not wear down within the mouth and no deterioration is seen in the material properties during processing, thus increasing the possibility of its use as crown material (Sinha et al., 2017).

It was observed that the fracture resistance of PEEK is higher than zirconia and ceramics, and PEEK can be modified easily by incorporation of other materials (Beuer et al., 2008).Due to its mechanical properties PEEK can be used in fixed prostheses thanks to inorganic chemicals in it. Also, it is recommended as a framework material for a fixed dental prosthesis due to its hardness (Heimer et al., 2017; Liebermann et al., 2016).The load bearing capacity in a laboratory study on a three-unit inlay retained bridge was evaluted in this study.The restorations with PEEK showed high loadbearing capacity and the fractures were mostly seen in the connector region (Aldhuwayhi et al., 2022).Many studies reported high patient comfort and acceptability with preferable marginal fit of CAD/CAM interim crowns and FPDs made up of PEEK (Chen et al., 2018). Compared to ceramics, the main advantages of polymers are their low elastic modulus, which allows better absorption of functional stresses by deformation, and that they do not cause wear on the enamel of the opposing tooth (Rea et al., 2017; Mishra & Chowdhary, 2019). Manufacturers state that PEEK, which can be produced by pressing or CADCAM techniques, can be used in fixed and removable prostheses; it can be restored with PEEK composite resin materials. In this way, it is possible to use them together with different superstructure materials compatible with resin cements when they are used as infrastructure (Sinha et al., 2017; Stawarczyk et al., 2015).

PEEK is also showed as an alternative treatment for patients with para functional habits; Studies have shown that the maximum bite force of male patients with severe bruxism is 978-1000 N (Geetha, 2020). PEEK supplies a cushioning effect on occlusal forces. That when an elastic framework is combined with materials with low elastic modulus like poly (methyl methacrylate) (PMMA) pre-fabricated veneers or veneering composite resin, it will reduce the occlusal forces to the restoration and the opposing dentition (Sobieraj et al., 2005; Boyce & Arruda, 1990). Therefore, the use of PEEK could be useful for FDPs where proprioception is reduced by the absence of periodontal ligaments and drive out mechanical problems such as veneer fractures and clicking sound during function reported for metal-ceramic or monolithic zirconia restorations (Sobieraj et al., 2005; Halabi et al., 2011). Thus, PEEK prostheses have been considered qualified for occlusal forces resistance in the molar region while the failure mode observed was adhesive between veneering composite and framework (Regrain et al., 2009).Additional advantages of PEEK are its radiolucency that makes cement removal and diagnosis of screw loosening easier. With its low specific weight which allow the construction of lighter prostheses (Kurtz & Devine, 2007).

**2.6.2.3 PEEK as Intraradicular Post-Core Material**

PEEK has been examined as intraradicular postcore material. Chen et al. found in their study that post made from PEEK have high fracture resistance when compared to other usually used post and core materials with less chances of root fracture when using PEEK (Chen et al., 2016). Because of its suitable processing (milling and pressing), acceptable mechanical strength, and shock absorbing ability. PEKK biomaterial has been applied in the post-core systems. PEKK displays excellent biomechanical behavior compared to metal and fiberglass post-core systems. The PEKK showed superior fracture resistance compared to metal and fiberglass post-core systems due to lower elastic modulus and flexural strength (Lee et al., 2017; Güven et al., 2020).Tekin et al discussed the use of low elastic modulus polyetheretherketone (PEEK) as dental post and crown material with glass fiber post and metal supported ceramic crowns using finite element analysis that is less stress formation in the tooth and post core restoration compared to other post systems was observed(Tekin et al., 2020).

A review by Alqahtani revealed that the PEEK post and core showed promising results with good mechanical and bonding properties. It is a successful alternative material to be used in clinical practice (Alqahtani et al, 2021). Another study by Yu et al discussed the use CFR-PEEK and pure PEEK posts could reduce the risk of debonding and vertical root fracture, whether they were used as prefabricated posts or customized posts, but the biomechanical behavior of carbon fiber-reinforced CFR-PEEK restorations was the closest to dentin. Therefore, the CFR-PEEK post could be more suitable to restore massive tooth defects (Yu et al., 2022).

Pure PEEK needs filler reinforcement to be used for post-retained restoration. On the other hand, it was found that custom fabricated PEKK posts showed higher bond strength than prefabricated PEKK posts. The custom made PEKK post exhibited a maximum bond-strength (17.34 MPa) in the cervical region as revealed from the scanning electron image. The conventional fiber posts showed the highest bond strength values in the middle (11.53 MPa) and apical sections (6.86 MPa) (Lee et al. 2017; Guo et al., 2012).

#### 2.6.2.4 PEEK with Removable Prosthesis

Owing to its light weight, excellent mechanical, biologic, and aesthetic properties PEEK can be used to construct dentures and clasps by CAD/ CAM systems (Benakatti et al., 2019). Another application would be to make removable obturators (Parthasarathy, 2014; Villefort et al., 2021). Due to its strength and lightweight partial denture framework made of PEEK digital design has improved patient satisfaction and customizes individual anatomy (Papathanasiou et al., 2020). Other properties like; absence of metal taste with no thermal and electrical conductivity, scanner and x-ray friendly, non-allergenic all these made PEEK as favorable material in the construction of removable prosthesis ( Stock et al.,2016; Micovic et al., 2021). Whitty has discussed the use of PEEK frameworks as shock absorbent during mastication, with an excellent resistance to decay and abrasion. Although metals exhibit good strength but resiliency and patient comfort are also of great concern (Whitty, 2014). An in vitro research showed that PEEK clasps have lower resistance forces in comparison to cobalt chrome (Tannous et al., 2012). Hence lead to the development of modified PEEK containing 20% ceramic fillers known as BioHPP. Good stability, corrections potential, high polish ability and aesthetics of BioHPP produce high-quality prosthesis (Skirbutis et al., 2017).BioHPP with a great potential as framework material, makes good alternative to Cr-Co frameworks in patients with high aesthetic requirements (Nayar et al., 2015).

It was clearly demonstrated in the literature, to serve the practical purpose of designing better dental structure, compatibility between the material and specific RPD frameworks, the stress of PEEK framework was smaller and more evenly distributed compared to CoCr (Chen et al., 2019). It was suggested that the framework made of PEEK has better protection function for PDL with more even dispersion of masticatory force compared with conventional metallic materials, which is suitable for patients with poor periodontal conditions. Also the mean retentive force required to remove the PEEK Removable Partial Dentures was found to be significant more superior than this required for Bre-Flex Removable Partial Dentures (Hasanen et al., 2018). A case report suggested the use of primary metal copings and secondary CAD-CAM PEEK framework veneered with composite resin for the fabrication of double-crown-retained interim removable dental prosthesis (Hahnel et al., 2018). Another study reported the use of primary zirconia copings and secondary PEEK framework veneered with monolithic zirconia for the rehabilitation of an edentulous patient with intolerance to titanium (Papathanasiou et al., 2020; Siewert, 2018). The study reported high chewing comfort and patient satisfaction with low weight, very good fit and retention.

Telescopic attachments fabricated from zirconia primary crowns and PEEK secondary crowns could also be applicable suggestion for retaining implant over dentures, with less stresses transmitted to the implants due to the stress-breaking capacity of PEEK (Emera et al., 2019).

#### 2.6.2.5 PEEK as Maxillofacial Prostheses

Thermoplastic polymers such as PEEK have been developed as advanced biomaterial that provided attractive alternatives for bone grafting (Mayer et al., 2020; Lommen et al., 2022; Micheletti et al., 2022). PEEK with its advanced mechanical properties has increased to be used as 3D design and printing computational technology, which permits implants specifically manufactured for each patient (Luo et al., 2023). The use of custom implants provides greater anatomical fit and a shorter operative time, also PEEK material provided the patient with a better-adjusted, more functional, and lighter prosthesis (Palau et al., 2014). Researchers have suggested that implant systems with elastic modulus near to dentin are known to reduce stress concentration. Lately, this improved by the use of PEEK, which is widely used, in dentistry as craniofacial implant superstructure (Nahara et al., 2020; Ichim et al., 2006). Another recent strategy by Xian et al, 2020 illustrated the use of modify PEEK surface with a biocompatible coating to improve its surface properties as well as bio-responses with regard to fibroblast and osteoblast cells affinity, anti-inflammatory, and antibacterial biocompatibilities, suitable for oral and maxillofacial implants application (Xiana et al., 2020).

Moreover, Li et al. found that the temperature and compression time of the hot compression molding process can sensitively change the mechanical properties in a good manner related to 3D printed PEEK implants. It also has superior dimensional accuracy and reproducibility, displaying clinically acceptable morphological likeness in terms of fit and contour continuity (Li et al., 2023; Sharma et al., 2021). Furthermore PEEK is successfully used in oral and maxillofacial surgery for fracture fixation, maxillofacial bone repair, occlusal splints, and surgical guides for implant placement.

Steffen et al. found that PEEK plates did not appear to guarantee the displacement and mechanical safety of mandibular reconstruction compared to miniature titanium plates (Steffen et al., 2020). However, Avci et al. concluded that the CF/PEEK plate/screw system reduces the stress on the fixation system and provides more stable fixation than the resorb able system (Avci et al., 2022). Meantime, they can simplify the masticatory pressure absorption and provide micro motion to the teeth during mastication which could be a potential direction for the future research and application of PEEK scaffold materials in dental field (Luo et al., 2023; Salah, & Tayebi, 2020). Table 2.7

**Table 2.7:** Recent mechanical property advances of PEEK in oral applications

| Application | Subcategory | Materials Tested | Outcomes |
|---|---|---|---|
| **Oral and maxillofacial surgery** | Mandibular fracture fixation | CFR-PEEK plate/screw system - Resorbable system | CFR-PEEK plate/screw system reduces the stress on the fixation system and provides more stable fixation |
| | Bone scaffold | PEEK with carboxyl groups (PEEK-COOH) | controllable porous scaffolds had better mechanical strength and are beneficial for promoting cell adhesion |
| **Prosthodontic therapy** | Implant-supported, fixed restorations | 20%/30% $TiO_2$-filled PEEK - Veneered resin composite/digital veneering/prefabricated veneering | The highest fracture resistance of the restorations was achieved when 30% $TiO_2$-filled PEEK material was used and prefabricated veneers were applied |

## 2.7. Reinforced PEEK

As PEEK is an emerging type of thermoplastic engineering, alterations and modulations of PEEK have been widely studied (Abd El-Fattah et al., 2021; Wang et al., 2021; Akhtar et al., 2021; Abdulhamid et al., 2020).Adding fibers into thermoplastic matrix has been elucidated to effectively enhance the performance of composites. Ashrafi et al. reported that the glass fibers (GF) can improve the tensile strength of 3D printed polymers at the expense of flexibility and ductility

(Ashrafi et al., 2017). Chalioris et al. evaluated the tensile strength of thermoplastic printed carbon fiber (CF) reinforced .The results show that the tensile strength and Young’s modulus grew with the increase of fiber content, although internal porosity of printed composites sample enlarged (Chalioris et al., 2018). Recently, fiber reinforced polymers (FRPs) have become remarkably advantageous in rehabilitation, repair and reinforcement of structure (Bazli & Abolfazli, 2020; Jafari et al., 2018; Bazli et al., 2020). In recent years, FRP have been implemented in various shapes, for instance I-shaped, channels, tubes, etc. reinforcing bars, sheets, strips ,grids and tendons with different types of adhesive amid strengthening or reinforcing structural members such as slabs, beams and columns (Bazli et al., 2020; Bazli et al., 2019; Micelli & Nanni , 2004 ). Figure (2.6), (2.7).


**Figure 2.6** Four types of FRP bars


**Figure 2.7** Glass, Carbon, and Aramid Fibers

There are four major materials applied in order to make fibers predominating for the diffent medical and industrial purposes: carbon, glass, aramid and basalt to form CFRP, GFRP, AFRP and BFRP, respectively (Dai et al., 2022; Abbood et al., 2021). Carbon fiber and glass fiber have excellent mechanical properties. The machinability and application of the fiber-reinforced PEEK are influenced by changing the material properties (Ji et al., 2015; Okabe & Takeda, 2002). For CF/PEEK; carbon fiber has high hardness and modulus and is evenly distributed inside the material. Thus, the tensile strength, hardness, and modulus are improved. It also upgraded the biocompatibility and the bioactivity, wearability, non-toxicity, and low cost (Jeon et al., 2021; Lee et al., 2019; Hung et al., 2014).The major advantages of carbon fiber include low density, low conductivity, high fatigue strength, high elastic modulus, good creep level, resistant to chemical effects and do not absorb water as well (Abbood et al., 2021). However, low compressive strength and being anisotropic (decreased radial strength) are the weak features in carbon fiber. Furthermore, the relatively high energy requirement to produce carbon fiber leads to rising costs is considered as another weak feature (Günaslan et al., 2014).For GF/PEEK, the glass fiber is easy to concentrate and create defects inside the material due to its high hardness, modulus, and brittleness. So this improves the hardness and modulus and decreases the tensile strength.  The effects on tensile strength and nanomechanical properties are more important as the fiber mass fraction increases (Gao et al., 2022; Talbott et al., 1987). High strength, increase resistance to water and chemicals with low cost are the main characteristics of glass fiber. Relatively low costs compared with other types of FRPs make glass fiber the most generally applied in material construction industry (Abbood et al., 2021; Günaslan et al., 2014).

Aramid fiber is anisotropic in nature and normally yellow color. Aramid fiber is popular these fibers have high cost compared with glass fiber, and sufficient for tension applications, however, it has low compressive strength, low density, high tensile strength, high elastic modulus and sufficient stiffness (Tuakta, 2005). Basalt fiber (BFRP) is a kind of fiery rocks that be made up of the fast cooling and it is similar to that of glass fibers. Such fibers have high tensile strength, perfect resistance to high temperatures and good durability. Other advantages are having excellent electromagnetic characteristics and resistant to corrosion, acid, radiation, UV and vibration. In comparison with FRP composites which are made from glass, carbon and aramid BFRP application in the civil construction field is quite limited (Abbood et al., 2021; Banibayat, 2011).

## 2.8 Mechanical Properties of PEEK

PEEK is a semi-crystalline polymer which is suitable to be used as a dental prosthesis and restorative material, due to its interesting mechanical and thermal properties. These great properties permit it to resist the hostile environment of the oral cavity and ensure long-term durability, a crucial parameter for dental restorative materials or dental prostheses (Moby et al., 2022; Ali et al., 2020; Rajamani et al., 2021). Many studies evaluated several properties of dental materials including thermoplastics which influence and predict their performance (Wang et al., 2003; Wang et al., 2021). PEEK is a high performance thermoplastic polymeric material with extremely high melting point (343˚C), so the knowledge of their mechanical properties is essential to support the correct indication of these materials and to expect a long-term performance (Rahman et al., 2015; Wilson, 1990).

In the oral environment, restorations are exposed to stresses from mastication action. These forces act on teeth and/ or material resulting in different reactions that lead to disfigurement, the stress-strain ratio of a material is relevant to define its mechanical behavior. For each material, there is a stress-strain proportional relationship, establishing a stress-strain curve ((Wang et al., 2003; Maekawa et al., 2015; Miura et al., 1986). The knowledge and explanation of how these materials behave under such forces are relevant to understand the performance of the material.

### 2.8.1 Tensile Strength Test

The resistance of the material to axial forces in a straight line and in opposite directions load is called tensile strength. Values of stress-strain determine a curve, describing the material performance under tensile test. In this test we can measure the ductibility of a material which is an important feature of hard materials to show the ability of the material to deform under tensile forces until the fracture moment and indicates the workability of an alloy (Wang et al., 2003; Darvell, 2000). The tensile samples (dog-bone shape refer to ISO 527-1: 2012) and flexural samples (90 mm× 5mm × 4 mm) dimensions (Vukasovic et al., 2019; ISO 527-1: 2012 Plastics).

### 2.8.2 Compressive Strength Test

Compressive strength is the ability of material to resist the direct pressure of applied compression force which represents the capacity to withstand loads before failure (Samui et al., 2020). Compressive strength also is specified by the reason of load by cross-sectional area (Kgf/cm$^2$) and the stress-strain curve of the studied material is determined as elastic modulus can also be determine by the ratio stress-strain (Wang et al., 2003; ISO 604:2002 Plastics). The compression test was carried out according to ISO604-2002. The size of the samples was (10×10×5) mm (Zhang et al., 2019; Garcia-Gonzalez et al., 2015).

### 2.8.3 Flexural Bending Strength Test

The flexural strength of a material is its ability to bend before it breaks. It is obtained when the ultimate flexibility of one material is achieved before its proportional limit. A high flexural strength is preferred once these materials are under the action of chewing force that might cause permanent deformation (Anusavice, 1993). To evaluate flexural strength of a dental material, it is generally used bar-shaped specimens with dimension of (80 mm × 10 mm × 4 mm) were printed according to ISO 178 standard (ISO 178: 2010 Plastics). Specimens are placed on two supports and a load is applied at the center. This test is known as three-point bending test.

### 2.8.4 Hardness Test

Surface hardness is a parameter used to evaluate material surface resistance to plastic deformation by penetration, the usual method to achieve hardness value is to measure the depth or area of an indentation left by an indenter of a specific shape with a specific force applied for a specific time (Maekawa et al., 2015; Craig, 1997). The Vickers hardness test method is one of the common standard test methods for stating the hardness of a material which consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 Kgf. The load is normally applied for 10 to 15 seconds. (Yahya, 2021; Şahin et al., 2022). A Vickers indentation is less sensitive to surface conditions and more sensitive to measurement errors than other hardness tests (Wang et al., 2003).All the test specimens were prepared in rectangles of the size (30×10×2.5) mm, ISO 20795-1 (Şahin et al., 2022; ISO 20795-1, 2022).

### 2.9 SEM Analyzing

A SEM (Scanning Electron Microscope) is one of the electron microscopes that produce images of a sample by scanning the surface with a focused beam of electrons (Hurjui et al.,, 2021). It has been a needful device in research since its invention in 1962, and has significantly contributed towards biology, medicine and material sciences research (Langer & Vacanti, 1993; Akkan, 2013).SEM is a scientific device that examines objects on a very fine scale yielding the information about the topography, morphology, composition and crystallographic information. Electron Microscopy permits the scanning of images at high magnification (50x - 10.000x and above).

#### 2.9.1 SEM Dental Applications:

Electron microscopy has a wide range of applications involving dental biomaterial and dental hard or soft tissues (Syed, 2017):

1. Evaluation of micro cracks/gaps and deformities in biomaterials and dental hard tissues.
2. Details of surface topography and roughness of different dental biomaterials /dental hard tissues.
3. Details of subsurface structures of dental hard tissues/dental biomaterials.
4. Differentiate various dental hard tissue and biomaterial structures.
5. Evaluation of bonds between various biomaterials and structures.

## 2.10 Optical Microscope

Optical microscopy is a technique used to view a sample closely through the magnification of a lens with visible light. This is the traditional form of microscopy, which was first invented before the 18th century and is still in use today (Mukherjee & Dasgupta, 2021).Digital optical microscopes (DOM) are becoming more approachable and present a powerful and more economical alternative to SEM in less demanding functions with magnification resolution 100–1000× 100–1000 nm (Banaszek et al., 2016; Nawrocka et al., 2021). A study shows that digital optical microscopy can be considered a promising, reliable and repeatable method to perform marginal analysis (Daher et al., 2021). Having this time and cost-effective method can be beneficial in the assessment of newer techniques.

# 3. MATERIALS AND METHODS

This study was approved by Atatürk University Faculty of Dentistry Ethics Committee (Process number 2022/36) and it was supported by Atatürk University Scientific Research Coordination Unit (2022/TDK-2022-10055). In this study, mechanical tests were carried out in the research laboratories of Atatürk University Faculty of Dentistry, Department of Prosthetic Dentistry, and at the Erzurum Technical University High Technology Application and Research Center (YUTAM). Four different types of PEEK were used in this study. The materials (samples) to be examined are given in (Table 3.1). The number of samples used in this study is based on the parameters explained by (F. Kibuacha, 2021) to determine sample size and how to calculate the sample size with yield smaller margins of error and are more representative.

In this thesis four different mechanical tests were performed for each of the different PEEK materials; compression strength test, bending test, tensile test, and Vickers hardness test.  A total of 160 samples were produced (n=40) for each test. They were tested at dry storage and after incubation in Ringer's solution ( Polifleks® Polıforma, Polifarma İlaç Sanayi Ve Tic., Turkey). (Manual Instruction, 2013) at different time intervals; 1 day, 1 week, and 3 weeks at 37°C (Figure 3.1). The devices used for measurements in this study are given in  Table 3.2.


**Figure 3.1** Lactate Ringer's solution

**Table 3.1**. Material Used in The Study and Technical Characteristics

| Abbreviation | Brand name | Composition | Manufacturing processes * | Specific gravity |
|---|---|---|---|---|
| I/ PE | Vıctrex Corp PEEK INCHR New Material China | pure PEEK R10 sheet | extrusion | 1.29g/cm |
| II/PC | Light copra PEEK Germany | pure PEEK disk | compresion molding | 1.22g/cm |
| III/CFP | Vıctrex Corp PEEK INCHR New Material China | PEEK with carbon fibers of 4-6 mm length | extrusion | 1.41g/cm |
| IV/GFP | Vıctrex Corp PEEK INCHR New Material China | PEEK with glass fibers of 4-6 mm length | extrusion | 1.52g/cm |

*Working Temperature 350-420C

**Table 3.2** Devices Used in The Study

| Device brand | Manufacturer |
|---|---|
| İnstron 5982 | Bluehill® 2 Software, UK |
| Vickers Hardness test device | Near Atul Nagar, 411025, Maharashtra |
| FESEM analyzing | Apreo 2S Quantax EBSD 400i, USA |
| Optic Microscope | ZEISS, Axiocam 503,Germany |

### 3.1 PEEK Samples Prepration

Four different types of PEEK material used in this study; pure PEEK R10 sheet (Victrex Corp INCHR, New Material, China), pure PEEK disk (WHITEPEAKS Light copra PEEK, Germany), PEEK with carbon fibers sheet, and PEEK with glass fibers sheets (Vıctrex Corp INCHR, New Material, China) (Figure 3.2 and 3.3).


**Figure 3.2** Light copra pure PEEK disk


**Figure 3.3** Victrex Corp INCHR**,** PEEK sheet

The tested samples were cutted according to ISO standareds that were specific for each meachanical test and were manually polished with a series of SiC abrasive papers up to P4000 (Buehler, Lake Bluff, IL, USA) by a polisher (Buehler, Coventry, UK). The samples were cutted and polished at dental laboratory (By Dental -Karabaglar/ Izmir, Turkey) (Figure 3.4).

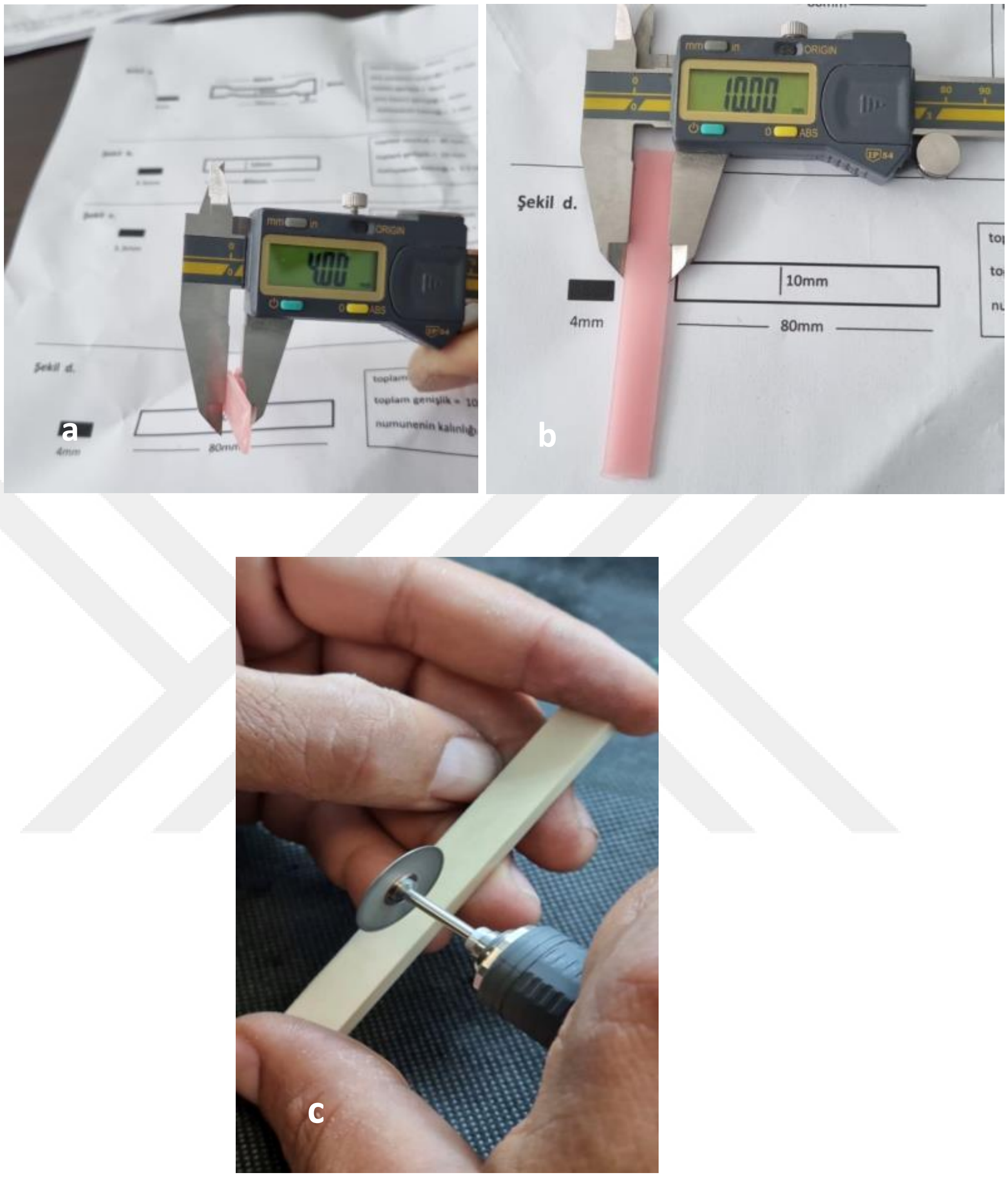


**Figure 3.4(a,b,c)** Prepairing PEEK samples

For compression strength test; the PEEK samples were cutted according to ISO 604: 2002 (Plastics—Determination of compressive properties) with specimen size of 10×10×4 mm (Bierögel, & Grellmann,2014; ISO 604: 2002 Plastics).

(Figure 3.5).For bending test the PEEK samples were cutted according to ISO 178: 2010 (Plastics— Determination of Flexural Properties) with specimen dimensions of 80×10×4 mm (ISO 178: 2010 Plastics). (Figure 3.6).

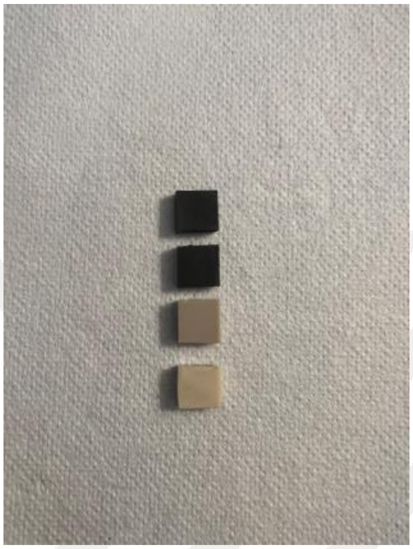

**Figure 3.5** PEEK samples for compression test

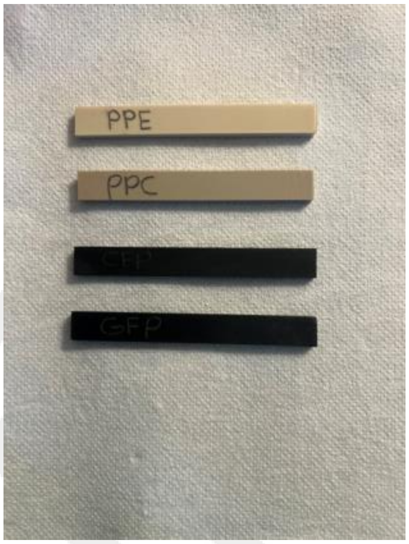


**Figure 3.6** PEEK samples for bending test

The dog-bone shape tensile testing specimens 90 × 5 × 4 mm were cutted according to ISO 527-2 standard **(** 2012 Plastics—Determination of Tensile Properties). (Figure 3.7). For microhardnesss vickers test the tested samples were prepared in rectangles of the size 30×10×2.5 mm according to ISO 20795-1 (2008- Dentistry-Base polymers-Denture base polymers). (Figure 3.8).


**Figure 3.7** PEEK samples for tensile test


**Figure 3.8** PEEK samples for hardness test

### 3.2 Tested Groups Creation

A total of 160 samples were produced for this study;these samples devided into four groups(n =40) according to the type of PEEK material used for each meachanical test. Then these four groups were devided into four subgroups (n =10) according to samples storage status. The group chart is shown at Table 3.3.

**Table 3.3** Grouping Arrangement of Study

| PEEK type | Environment storage status | group code | No. of samples |
|---|---|---|---|
| **Pure extrusion PEEK** | Dry storage | $PPEt_0$ | 10 |
| **Pure extrusion PEEK** | 1 day storage | $PPEt_1$ | 10 |
| **Pure extrusion PEEK** | 1 week storage | $PPEt_2$ | 10 |
| **Pure extrusion PEEK** | 3 weeks storage | $PPEt_3$ | 10 |
| **Pure compression PEEK** | Dry storage | $PPCt_0$ | 10 |
| **Pure compression PEEK** | 1 day storage | $PPCt_1$ | 10 |
| **Pure compression PEEK** | 1 week storage | $PPCt_2$ | 10 |
| **Pure compression PEEK** | 3 weeks storage | $PPCt_3$ | 10 |
| **Carbon fiber PEEK** | Dry storage | $CFPt_0$ | 10 |
| **Carbon fiber PEEK** | 1 day storage | $CFPt_1$ | 10 |
| **Carbon fiber PEEK** | 1 week storage | $CFPt_2$ | 10 |
| **Carbon fiber PEEK** | 3 weeks storage | $CFPt_3$ | 10 |
| **Glass fiber PEEK** | Dry storage | $GFPt_0$ | 10 |
| **Glass fiber PEEK** | 1 day storage | $GFPt_1$ | 10 |
| **Glass fiber PEEK** | 1 week storage | $GFPt_2$ | 10 |
| **Glass fiber PEEK** | 3 weeks storage | $GFPt_3$ | 10 |
| | | | Total no. =160 |

After the group forming, the four meachanical tests were taken, the samples of different PEEK materials at dry storage environment were considered as control group for each subgroup.

### 3.3 Samples Storage

The different PEEK samples; PPE, PPC, CFP, and GFP were measured at four different storage environments three samples tested for each PEEK type; 1st at dry storage (control group), then storage with ringer solution for 1 day, 1 week, and 3 weeks time intervals. PEEK samples were allowed to dry at room tempreture ($25^0$ C) before testing. Figure (3.9).


**Figure 3.9 (a,b,c,d)** PEEK group samples at different storage time

### 3.4 Compression Test Measurements

In this study the compression test was taken by electromechanical universal testing system (INSTRON, 5982, Bluehill® 2 Software, UK),with 10 KN load cell and constant displacement of 1.3mm/min static testing systems at the Erzurum Technical University High Technology Application and Research Center (YUTAM). Figure (3.10), Figure (3.11).


**Figure 3.10** INSTRON, 5982

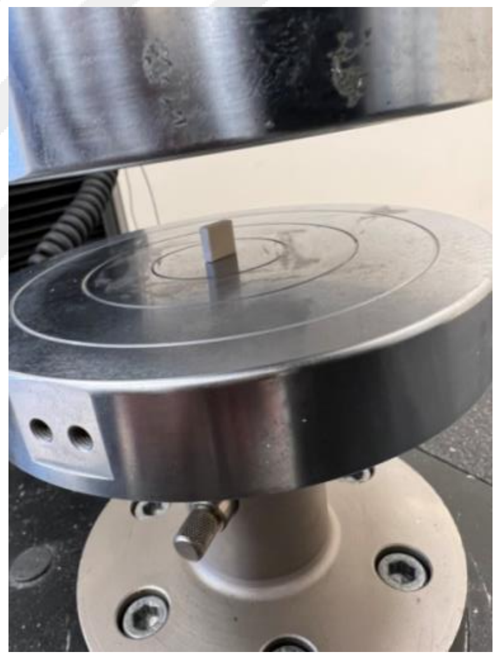

**Figure 3.11** PEEK sample on compression test

### 3.5 Bending Test Measurements

In this study the three point bending test taken by electromechanical universal testing system (INSTRON, 5982, Bluehill® 2 Software, UK), with fixture length=50 and at speed of 1mm/min. Each test took about 6 min to reach a permanent deformity phase. Ductility also was valued within the recorded diagram.  This test was measured at the Erzurum Technical University High Technology Application and Research Center (YUTAM). (Figure 3.12). The obtained values were recorded .

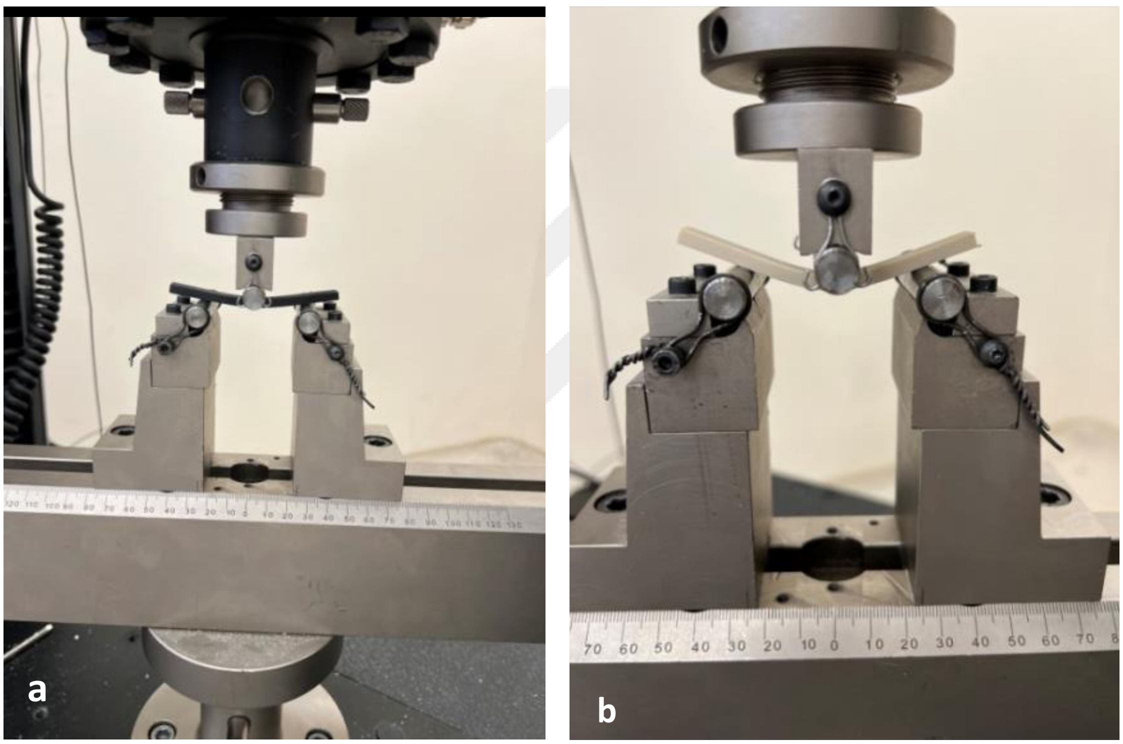


**Figure 3.12 (a,b)**  PEEK sample on bending test

### 3.6 Tensile Test Measurements

For tensile test measurements the PEEK samples were examined with  device (INSTRON, 5982, Bluehill® 2 Software, UK) at speed of 1mm/min. Sample elongation was also was valued within the recorded diagram. This test was measured at the Erzurum Technical University High Technology Application and

Research Center (YUTAM). (Figure 3.13), (Figure 3.14). The obtained values were recorded.


**Figure 3.13** PEEK sample on tensile test

**Figure 3.14** PEEK sample elongation

### 3.7 Vickers Microhardness Test Measurements

Microhardness test was performed at Atatürk University Faculty of Dentistry, Department of Prosthodontics. The Vickers Hardness Tester (Universal Motion, Inc. No. 208, R Cube, S. No. 116/5/1, Warje Highway, Near Atul Nagar, Pune - 411052, Maharashtra) located in the research laboratory was used. A 10 kg load was applied to the surfaces of the PEEK specimens for 15 seconds with a square-based pyramidal-shaped diamond indenter with face angles of 136° (Figure 3.15).

The pyramid image formed on the sample surfaces was determined, and the D1 and D2 lengths, which indicate the diagonal lengths, were measured. The measured values are again calculated on the device ($Kg/mm^2$).

**Figure 3.15** vickers hardness test device

### 3.8 FESEM Analyzing

After mechanical tests were applied to all different PEEK samples, with different storage environments a random sample was selected from each group. The surface imaging process was performed on the ThermoFisher scientific electron microscope (Apreo 2S Quantax EBSD 400i, USA) in the research laboratory of the Technical Park Engineering Department of the Faculty of Engineering of Yildiz Technical University, with surface coating process on the selected samples to avoid overheated energy and get clear pictures. (Figure 3.16) (Figure 3.17). Images viewed with x 20.000times magnification, and the images were saved.


**Figure 3.16** FESEM Analyzer

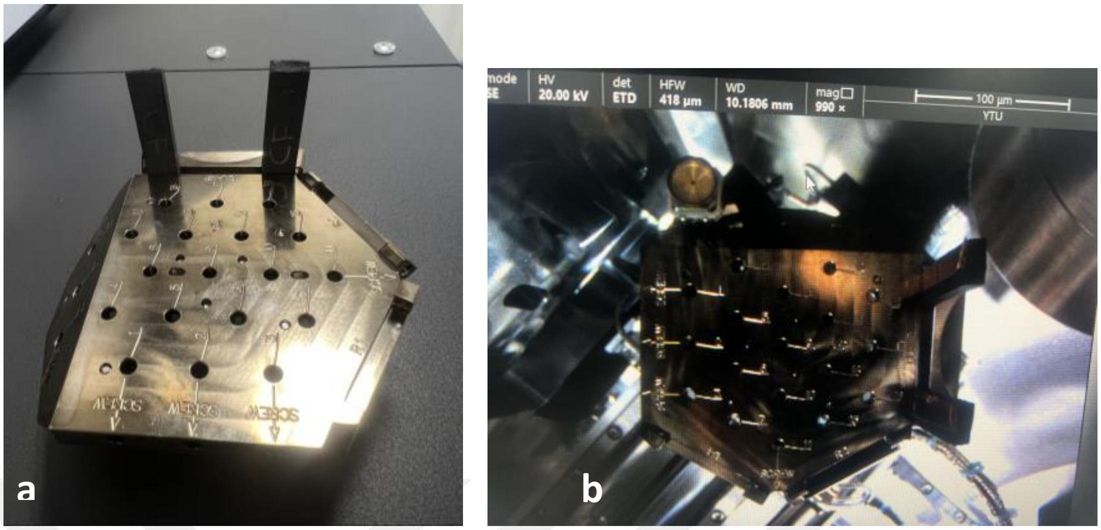


**Figure 3.17 a.** PEEK samples with fracture surface in upper direction **b.** PEEK samples inside the electron microscope

### 3.9 Optic Microscope

Four different PEEK samples were examined under the optic microscope to magnify the fracture behavior after tensile strength meachanical test accomblished. These samples were randomly selcted from each group with different storage enviroments. The imaging process was performed by ZEISS/ Scope with resolution of 5× 0.13HD to obtain suitable degree of clarity. (A1 Optic microscope. Axiocam 503 color EC EPIPLAN, Germany). (Figure 3.18) This test was accomplished at in the research laboratory of the Mechanical Engineering Department of the Faculty of Engineering and Architecture of Erzurum Technical University.

**Figure 3.18** A1 Optic microscope

### 3.10 Statistical Analysis

The analyses and figures were conducted using GraphPad Prism 8.0. Descriptive statistics (means, standards deviations,) of the two experimental sets were calculated. The Shapiro Wilk-W test was conducted to check the normality of the continuous variables. Since the variables were normally distributed, the one-way ANOVA with post hoc test (Tukey's multiple comparisons) were run to identify the difference in the hardness or stress values across the four treatment groups: pure Peek extrusion (PPE), pure Peek compression (PPC), carbon fiber Peek (CFP) and glass fiber Peek (GFP). In other words, we measured the differences in the one parameter (stress in three groups and hardness in one group) taking the groups type into consideration.

Additionally, the repeated measure ANOVA and post-hoc tests were used to measure the hardness or stress values within the same group(s) across different storage times/conditions: dry (control), and 1 day, 1 week and 3 weeks with Ringer solution environment. Statistical significance level was taken as P-value $<0.05$.

# 4. RESULTS

## 4.1. Statistical Analyzes of Pressure Test

Across all the four study groups (PPE, PPC, CFP and GFP), the mean of pressure stress was significantly highest after one week in the Ringer solution (Figure 4.1). Within each group, the pressure stress increased significantly after one day and one week of storage in the solution (Figure 4.1). Across three groups (PPE, PPC and GFP), pressure stress slightly reduced after three weeks storage in the solution. The CFP had the lowest (102, after one day in the solution) pressure stress while GFP group had the highest (188.6, after one week in the solution) pressure stress among the four groups.

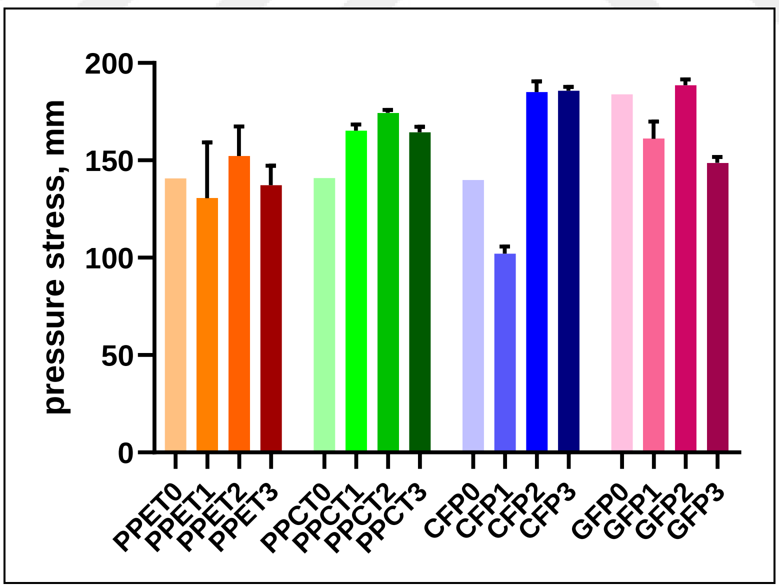


**Figure 4.1**: The difference in the pressure stress value across four different storage conditions/ times and among the four different material groups: pure Peek extrusion (PPE), pure Peek compression (PPC), carbon fiber Peek (CFP) and glass fiber Peek (GFP).

### 4.1.1The pressure stress value across different storage conditions/ times within PPE group

The mean of pressure stress value within Pure Peek Extrusion (PPE) group ranged from 130.6 mm after one day to 152.2 mm after one week in the Ringer solution.There was fluctuation in the pressure stress over the four-time measurements. More specifically, the pressure stress reduced after one day storing in the Ringer solution then increased after one week and decreased again after three weeks of storage in the solution (Figure 4.1). According to repeated measure ANOVA, there was no significant (P-value >0.05) difference in pressure stress within PPE group across different storage conditions/ times (Table 4.1).

**Table 4.1**: The difference in the pressure stress value across different storage conditions/ times within the same group (pure Peek extrusion)

| **repeated measure ANOVA p value** | | **0.499** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET0 vs. PPET1** | 140.8 | **130.6** | 10.19 | 16.51 | No | ns | 0.9185 |
| **PPET0 vs. PPET2** | 140.8 | 152.2 | -11.37 | 8.776 | No | ns | 0.6426 |
| **PPET0 vs. PPET3** | 140.8 | 137.3 | 3.541 | 5.792 | No | ns | 0.9203 |
| **PPET1 vs. PPET2** | 130.6 | **152.2** | -21.56 | 25.25 | No | ns | 0.8302 |
| **PPET1 vs. PPET3** | 130.6 | 137.3 | -6.648 | 15.24 | No | ns | 0.9665 |
| **PPET2 vs. PPET3** | 152.2 | 137.3 | 14.91 | 11.8 | No | ns | 0.6559 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons) Time-0=dry (control) condition, Time-1=1 day in Ringer solution, Time-2=1 week in Ringer solution, Time-3= 3 weeks in Ringer solution.

### 4.1.2 The pressure stress value across different storage conditions/ times within PPC group

The mean of pressure stress value within Pure Peek Compression (PPC) group ranged from 141.0 mm in the dry condition to 174.4 mm after one week in the Ringer solution (Table 4.2 and figure 4.1). There was fluctuation in the pressure stress over the four-time measurements. More specifically, the pressure stress increased after storing in the Ringer solution compared to dry condition. According to repeated measure ANOVA and post-hoc tests, there were five significant (P-value<0.05) differences in the pressure stress within PPC group across different storage conditions/ times. The pressure stress means in the dry condition and after three weeks in Ringer solution were significantly lower than after one day and one week in the solution .More specifically, the pressure stress in the control (dry) condition within PPC group was significantly lowest than all the three measurements after the storing in the Ringer solution (Table 4.2).

**Table 4.2:** The difference in the pressure stress value across different storage conditions/ times within the same group (pure Peek compression)

| **Repeated measure ANOVA p value** | | **0.0003** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPCT0 vs. PPCT1** | 141 | 165.3 | -24.36 | 1.758 | Yes | * | 0.0131 |
| **PPCT0 vs. PPCT2** | **141** | **174.4** | -33.45 | 0.869 | Yes | *** | 0.0004 |
| **PPCT0 vs. PPCT3** | 141 | 164.4 | -23.42 | 1.625 | Yes | * | 0.0121 |
| **PPCT1 vs. PPCT2** | 165.3 | 174.4 | -9.083 | 0.903 | Yes | * | 0.0243 |
| **PPCT1 vs. PPCT3** | 165.3 | 164.4 | 0.942 | 1.128 | No | ns | 0.8378 |
| **PPCT2 vs. PPCT3** | 174.4 | 164.4 | 10.03 | 0.995 | Yes | * | 0.0242 |

*The measurements of pressure stress are in mm.

### 4.1.3 The pressure stress value across different storage conditions/ times withinCFP group

The mean of pressure stress value within carbon fiber Peek (CFP) group ranged from 102 mm after one day to 185.8 mm after three weeks in the Ringer solution (Table 4.3 and Figure 4.1). There was fluctuation in the pressure stress over the four-time measurements. More specifically, the pressure stress reduced after one day storing in the Ringer solution then increased after 1-3 weeks of storage in the solution. According to repeated measure ANOVA and post-hoc tests, there were five significant (P-value<0.05) differences in pressure stress within CFP group across different storage conditions/ times. The pressure stress was significantly higher after 1-3 weeks in the solution compared to the dry condition and after one day in the solution.. However, there was no significant change in the pressure stress between week one and week three in the solution (Table 4.3).

**Table 4.3**: The difference in the pressure stress value across different storage conditions/times within the same group (carbon fiber Peek)

| **Repeated measure ANOVA P-value** | | **0.0002** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **CFP0 vs. CFP1** | 139.9 | **102** | 37.93 | 2.129 | Yes | ** | 0.0081 |
| **CFP0 vs. CFP2** | 139.9 | **185.1** | -45.19 | 3.174 | Yes | * | 0.0124 |
| **CFP0 vs. CFP3** | 139.9 | **185.8** | -45.83 | 1.181 | Yes | *** | 0.0003 |
| **CFP1 vs. CFP2** | 102 | 185.1 | -83.12 | 4.634 | Yes | ** | 0.008 |
| **CFP1 vs. CFP3** | 102 | 185.8 | -83.76 | 2.943 | Yes | ** | 0.0022 |
| **CFP2 vs. CFP3** | 185.1 | 185.8 | -0.6394 | 3.848 | No | ns | 0.9979 |

### 4.1.4The pressure stress value across different storage conditions/ times within GFP group

The mean of pressure stress value within GFP group ranged from 148.6 mm after three weeks to 188.6 mm after one week in the Ringer solution (Table 4.4 and figure 4.1). There was fluctuation in the pressure stress over the four-time measurements. More specifically, the pressure stress reduced after one day storing in the Ringer solution then increased after one week and decreased again after three weeks of storage in the solution. More specifically, the mean of pressure stress was significantly lower after three weeks in the solution than that in the dry condition and after one week in the solution (Table 4.4).

**Table 4.4:** The difference in the pressure stress value across different storage conditions/times within the same group (glass fiber Peek)

| **Repeated measure ANOVA P-value** | | **0.0091** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **GFP0 vs. GFP1** | 184 | 161 | 22.97 | 5.121 | No | ns | 0.1126 |
| **GFP0 vs. GFP2** | 184 | **188.6** | -4.556 | 1.749 | No | ns | 0.2804 |
| **GFP0 vs. GFP3** | 184 | **148.7** | 35.3 | 1.788 | Yes | ** | 0.0066 |
| **GFP1 vs. GFP2** | 161 | 188.6 | -27.53 | 4.443 | No | ns | 0.0618 |
| **GFP1 vs. GFP3** | 161 | 148.7 | 12.33 | 6.146 | No | ns | 0.4045 |
| **GFP2 vs. GFP3** | 188.6 | 148.7 | 39.86 | 3.534 | Yes | * | 0.0194 |

#### 4.1.5 The Differences in the Pressure Stress among the four groups (PPE, PPC, CFP and GFP) within each time point

##### 4.1.5.1The pressure stress value across the four groups after one day in the solution

The mean of pressure stress on day 1 in the Ringer solution ranged from 102 mm for CFP group and 165.3 mm for PPC group. In other words, the CFP had the lowest pressure stress while the PPC group had the highest-pressure stress after one day in the solution (Table 4.5). According to one-way ANOVA and Tukey's multiple comparisons test, there were two significant differences among the four groups. The mean of pressure stress in the PPC and GFP groups were significantly (P-value<0.05) higher compared to that in the CFP group (Table4.5).

**Table 4.5:** The difference in the pressure stress value across the four groups at day 1 in the solution environment

| **Ordinary ANOVA P-value** | | **0.003** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET1 vs. PPCT1** | 130.6 | **165.3** | -34.68 | 12.38 | No | ns | 0.0881 |
| **PPET1 vs. CFP1** | 130.6 | **102** | 28.64 | 12.38 | No | ns | 0.174 |
| **PPET1 vs. GFP1** | 130.6 | 161 | -30.38 | 12.38 | No | ns | 0.1433 |
| **PPCT1 vs. CFP1** | 165.3 | 102 | 63.31 | 12.38 | Yes | ** | 0.004 |
| **PPCT1 vs. GFP1** | 165.3 | 161 | 4.297 | 12.38 | No | ns | 0.9846 |
| **CFP1 vs. GFP1** | 102 | 161 | -59.02 | 12.38 | Yes | ** | 0.0062 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

#### 4.1.5.2 The pressure stress value across the four groups after one week in the solution

The mean of pressure stress after one week in the Ringer solution ranged from 152.2 mm for PPE group to 188.6 mm for GFP group. In other words, the PPE had the lowest pressure stress while the GFP group had the highest-pressure stress after one week in the solution (Table 4.6). According to one-way ANOVA and Tukey's multiple comparisons test, there were three significant differences among the four groups. The mean of pressure stress in PPE and PPC groups were significantly ($P<0.05$) lower compared to that in the CFP and GFP groups.More specifically, the mean of pressure stress in PPE group was significantly ($P<0.05$) lower (152.2 mm) compared to that in the other three groups (PPC, CFP and CFP after one week storing in the solution (Table 4.6).

**Table 4.6:** The difference in the pressure stress value across the four groups after one week in the solution environment

| **Ordinary ANOVA p value** | | **0.0026** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET2 vs. PPCT2** | 152.2 | 174.4 | -22.2 | 6.742 | Yes | * | 0.044 |
| **PPET2 vs. CFP2** | 152.2 | 185.1 | -32.93 | 6.742 | Yes | ** | 0.0053 |
| **PPET2 vs. GFP2** | **152.2** | **188.6** | -36.35 | 6.742 | Yes | ** | 0.0029 |
| **PPCT2 vs. CFP2** | 174.4 | 185.1 | -10.72 | 6.742 | No | ns | 0.4345 |
| **PPCT2 vs. GFP2** | 174.4 | 188.6 | -14.15 | 6.742 | No | ns | 0.232 |
| **CFP2 vs. GFP2** | 185.1 | 188.6 | -3.427 | 6.742 | No | ns | 0.9548 |

#### 4.1.5.3 The pressure stress value across the four groups after three weeks in the solution

The mean of pressure stress after three weeks in the Ringer solution ranged from 137.3 mm for PPE group to 185.8 mm for CFP group. In other words, the PPE had the lowest pressure stress while the CFP group had the highest-pressure stress after three weeks in the solution (Table 4.7). According to one-way

ANOVA and Tukey's multiple comparisons test, there were five significant differences among the four groups. The mean of pressure stress in PPE and GFP groups were significantly (P-value<0.05) lower compared to that in the CFP and PPC groups .More specifically, the mean of pressure stress in PPE group was significantly (P-value<0.05) lower (137.3 mm) compared to that in the other three groups (PPC, CFP and CFP after three weeks storing in the solution .Additionally, the mean of pressure stress in the CFP group was significantly higher than that in GFP group after three weeks in the solution (Table 4.7).

**Table 4.7:** The difference in the pressure stress value across the four groups after week 3 in the solution environment

| **Ordinary ANOVA P-value** | | **<0.0001** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P-Value** |
| **PPET3 vs. PPCT3** | 137.3 | 164.4 | -27.09 | 4.515 | Yes | ** | 0.0015 |
| **PPET3 vs. CFP3** | **137.3** | **185.8** | -48.48 | 4.515 | Yes | **** | <0.0001 |
| **PPET3 vs. GFP3** | 137.3 | 148.7 | -11.4 | 4.515 | No | ns | 0.1297 |
| **PPCT3 vs. CFP3** | 164.4 | 185.8 | -21.39 | 4.515 | Yes | ** | 0.0064 |
| **PPCT3 vs. GFP3** | 164.4 | 148.7 | 15.69 | 4.515 | Yes | * | 0.0342 |
| **CFP3 vs. GFP3** | 185.8 | 148.7 | 37.07 | 4.515 | Yes | *** | 0.0002 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

### 4.2. Statistical Analyzes of Bending Test

No clear pattern existed for bending stress across the four groups. The bending stress of GFP group was the lowest at dry condition compared to the three Ringer solution measurements (Figure 4.2). In contrast, the bending stress of CFP group was the lowest after three weeks in the solution compared to the three measurements. Within CFP and GFP groups, the bending stress increased significantly between dry condition and after one day in the solution. The bending stress mean was the highest after one day for CFP group, after one week for PPC group and after three weeks for PPE group in the solution compared to other three measurements/storage conditions (Figure 4.2). The CFP had the lowest (130.8, after three weeks in the solution) bending stress while PPE group had the highest (195.2, after three weeks in the solution) bending stress among the four groups.

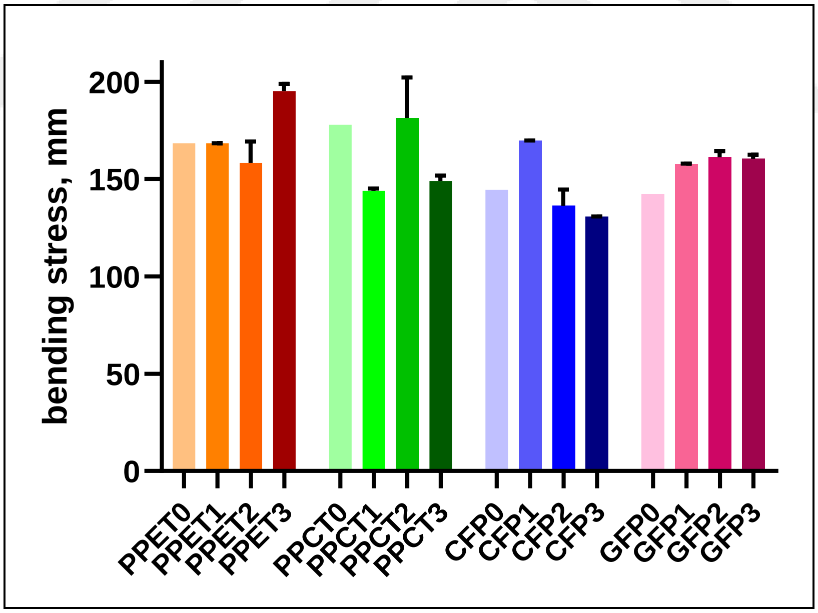


**Figure 4.2:** The difference in the bending stress value across four different storage conditions/ times and among the four different material groups: pure Peek extrusion (PPE), pure Peek compression (PPC), carbon fiber Peek (CFP) and glass fiber Peek (GFP).

### 4.2.1 The bending stress value across different storage conditions/ times within PPE group

The mean of bending stress value within pure Peek extrusion (PPE) group ranged from 158.3 mm after one week to 195.2 mm after three weeks in the Ringer solution (Table 4.8 and figure 4.2). In other words, bending stress within PPE group was at the lowest level after one week and at the highest level after three weeks in the solution. There was no change in bending stress within PPE group between storage in dry condition and after one day storing in the solution. Thereafter, there was fluctuation in the bending stress after one week and three weeks of storage in the solution. More specifically, the bending stress reduced after one week storing in the Ringer solution then increased after three weeks of storage in the solution (Figure 4.2).

According to repeated measure ANOVA and post-hoc tests, there were two significant ($P < 0.05$) differences in bending stress within PPE group across different storage conditions/ times .The bending stress was significantly higher after three weeks in the solution compared to after one week in the solution. However, there were no significant ($P > 0.05$) changes in the bending stress between dry condition from one side and one day and one week in the solution from the other side (Table 4.8).

**Table 4.8:** The difference in the <u>bending stress</u> value across different storage conditions/ times within the same group (pure Peek extrusion)

| **repeated measure ANOVA p value** | | | **0.0472** | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET0 vs. PPET1** | 168.5 | 168.4 | 0.114 | 0.059 | No | ns | 0.4239 |
| **PPET0 vs. PPET2** | 168.5 | **<u>158.3</u>** | 10.16 | 6.394 | No | ns | 0.5309 |
| **PPET0 vs. PPET3** | 168.5 | **<u>195.2</u>** | -26.73 | 2.104 | Yes | * | 0.0154 |
| **PPET1 vs. PPET2** | 168.4 | 158.3 | 10.05 | 6.453 | No | ns | 0.5422 |
| **PPET1 vs. PPET3** | 168.4 | 195.2 | -26.85 | 2.049 | Yes | * | 0.0145 |
| **PPET2 vs. PPET3** | 158.3 | 195.2 | -36.9 | 8.302 | No | ns | 0.1145 |

*The measurements of bending stress are in mm.

### 4.2.2 The bending stress value across different storage conditions/ times within PPC group

The mean of bending stress value within PPC group ranged from 143.9 mm after one day to 181.4 mm after one week in the Ringer solution (Table 4.9 and Figure 4.2). There was fluctuation in the bending stress over the four-time measurements. More specifically, the bending stress reduced after one day storing in the Ringer solution then increased after one week and decreased again after three weeks of storage in the solution. More specifically, the mean of bending stress was significantly lower after one day and three weeks in the solution than that in the dry condition (Table 4.9).

**Table 4.9**: The difference in the bending stress value across different storage conditions/ times within the same group (pure Peek compression)

| **repeated measure ANOVA p value** | | **0.0908** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPCT0 vs. PPCT1** | 178 | **143.9** | 34.07 | 0.744 | Yes | **** | <0.0001 |
| **PPCT0 vs. PPCT2** | 178 | **181.4** | -3.401 | 12.06 | No | ns | 0.99 |
| **PPCT0 vs. PPCT3** | 178 | 149 | 28.91 | 1.631 | Yes | ** | 0.0082 |
| **PPCT1 vs. PPCT2** | 143.9 | 181.4 | -37.47 | 12.50 | No | ns | 0.2253 |
| **PPCT1 vs. PPCT3** | 143.9 | 149 | -5.154 | 0.889 | No | ns | 0.0701 |
| **PPCT2 vs. PPCT3** | 181.4 | 149 | 32.31 | 13.13 | No | ns | 0.3052 |

### 4.2.3The bending stress value across different storage conditions/ times within CFP group

The mean of bending stress value within CFP group ranged from 130.8 mm after three weeks to 169.7 mm after one day in the Ringer solution (Table 4.10 and figure 4.2). There was fluctuation in the pressure stress over the four-time measurements. More specifically, the bending stress increased sharply after one day storing in the Ringer solution compared to dry condition then reduced over time in the solution. The bending stress was the highest after one day and the lowest after three weeks storing in the solution.  According to repeated measure ANOVA and post-hoc tests, there were three significant ($P < 0.05$) differences in the bending stress within CFP group across different storage conditions/ times.

The bending stress means in the dry condition and after three weeks in Ringer solution were significantly lower than that after one day in the solution. Moreover, the bending stress in the dry condition was significantly higher than that after three weeks in the solution (Table 4.10).

**Table 4.10:** The difference in the bending stress value across different storage conditions/ times within the same group (carbon fiber Peek)

| **repeated measure ANOVA P-value** | | **0.0188** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **CFP0 vs. CFP1** | 144.5 | **169.7** | -25.24 | 0.077 | Yes | **** | <0.0001 |
| **CFP0 vs. CFP2** | 144.5 | 136.3 | 8.186 | 4.767 | No | ns | 0.4881 |
| **CFP0 vs. CFP3** | 144.5 | **130.8** | 13.73 | 0.042 | Yes | **** | <0.0001 |
| **CFP1 vs. CFP2** | 169.7 | 136.3 | 33.42 | 4.834 | No | ns | 0.0502 |
| **CFP1 vs. CFP3** | 169.7 | 130.8 | 38.97 | 0.074 | Yes | **** | <0.0001 |
| **CFP2 vs. CFP3** | 136.3 | 130.8 | 5.547 | 4.761 | No | ns | 0.6972 |

### 4.2.4The bending stress value across different storage conditions/ times within GFP group

The mean of bending stress value within GFP group ranged from 142.4 mm in the dry condition to 161.3 mm after one week in the Ringer solution (Table 4.11 and Figure 4.2). In other words, the bending stress was the lowest at dry condition for GFP group. There was fluctuation in the bending stress over the four-time measurements. More specifically, the bending stress increased after storing in the

Ringer solution compared to dry condition. According to repeated measure ANOVA and post-hoc tests, there were three significant ($P < 0.05$) differences in the bending stress within GFP group across different storage conditions/ times. The pressure stress means in the dry condition was significantly lower than the three measurements after storing in the Ringer solution. More specifically, the bending stress in the control (dry) condition within GFP group was significantly lowest than all the three measurements after the storing in the Ringer solution (Table 4.11).

**Table 4.11:** The difference in the bending stress value across different storage conditions/ times within the same group (glass fiber Peek)

| **Repeated measure ANOVA P-value** | | **0.0181** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **GFP0 vs. GFP1** | 142.4 | 157.8 | -15.35 | 0.075 | Yes | **** | <0.0001 |
| **GFP0 vs. GFP2** | **142.4** | **161.3** | -18.88 | 1.847 | Yes | * | 0.0236 |
| **GFP0 vs. GFP3** | 142.4 | 160.4 | -18.02 | 1.155 | Yes | * | 0.0104 |
| **GFP1 vs. GFP2** | 157.8 | 161.3 | -3.527 | 1.901 | No | ns | 0.446 |
| **GFP1 vs. GFP3** | 157.8 | 160.4 | -2.665 | 1.120 | No | ns | 0.3203 |
| **GFP2 vs. GFP3** | 161.3 | 160.4 | 0.8623 | 2.976 | No | ns | 0.9892 |

### 4.2.5 The Differences in the Bending Stress among the four groups (PPE, PPC, CFP and GFP) within each time point

#### 4.2.5.1 The bending stress value across the four groups after one day in the solution

The mean of bending stress on day 1 in the Ringer solution ranged from 143.9 mm for PPE group and 169.7 mm for CFP group. In other words, the CFP had the highest bending stress while the PPE group had the lowest bending stress after one day in the solution (Table 4.12). According to one-way ANOVA and Tukey's multiple comparisons test, there were five significant differences among the four groups. The mean of bending stress in the PPC and GFP groups were significantly (P-value<0.05) higher compared to that in the CFP group.

**Table 4.12**: The difference in the bending stress value across the four groups after day 1 in the solution environment

| **Ordinary ANOVA P-value** | | **0.003** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET1 vs. PPCT1** | 168.4 | **143.9** | 24.47 | 0.533 | Yes | **** | <0.0001 |
| **PPET1 vs. CFP1** | 168.4 | **169.7** | -1.377 | 0.533 | No | ns | 0.1199 |
| **PPET1 vs. GFP1** | 168.4 | 157.8 | 10.59 | 0.533 | Yes | **** | <0.0001 |
| **PPCT1 vs. CFP1** | 143.9 | 169.7 | -25.84 | 0.533 | Yes | **** | <0.0001 |
| **PPCT1 vs. GFP1** | 143.9 | 157.8 | -13.87 | 0.533 | Yes | **** | <0.0001 |
| **CFP1 vs. GFP1** | 169.7 | 157.8 | 11.97 | 0.533 | Yes | **** | <0.0001 |

#### 4.2.5.2 The bending stress value across the four groups after one week in the solution

The means of bending stress after one week in the Ringer solution ranged from 136.3 mm for CFP group to 181.4 mm for PPC group. In other words, the CFP had the lowest pressure stress while the PPC group had the highest- bending stress after one week in the solution (Table 4.13). According to one-way ANOVA and Tukey's multiple comparisons test, there was only one significant difference in bending stress among the four groups in after one week in the solution. More specifically, the mean of bending stress in PPC group was significantly ($P<0.05$) higher (181.4 mm) compared to that in the CFP group after one week storing in the solution (Table 4.13).

**Table 4.13:** The difference in the bending stress value across the four groups at week 1 in the solution environment

| **Ordinary ANOVA P-value** | | **0.0161** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET2 vs. PPCT2** | 158.3 | **181.4** | -23.05 | 10.31 | No | ns | 0.1931 |
| **PPET2 vs. CFP2** | 158.3 | **136.3** | 22.0 | 10.31 | No | ns | 0.2215 |
| **PPET2 vs. GFP2** | 158.3 | 161.3 | -2.984 | 10.31 | No | ns | 0.9909 |
| **PPCT2 vs. CFP2** | 181.4 | 136.3 | 45.05 | 10.31 | Yes | * | 0.0102 |
| **PPCT2 vs. GFP2** | 181.4 | 161.3 | 20.07 | 10.31 | No | ns | 0.2828 |
| **CFP2 vs. GFP2** | 136.3 | 161.3 | -24.98 | 10.31 | No | ns | 0.1495 |

### 4.2.5.3 The bending stress value across the four groups after three weeks in the solution

The means of bending stress after three weeks in the Ringer solution ranged from 130.7 mm for PPC group to 195.2 mm for PPE group. In other words, the PPC had the lowest bending stress while the PPE group had the highest- bending stress after three weeks in the solution (Table 4.14). According to one-way ANOVA and Tukey's multiple comparisons test, there were six significant differences among the four groups. The mean of bending stress in CFP and PPC groups were significantly (P<0.05) lower compared to that in the PPE and GFP groups .More specifically, the mean of bending stress in PPE group was significantly (P<0.05) higher (195.2 mm) compared to that in the other three groups (PPC, CFP and CFP after three weeks storing in the solution. The mean of bending stress in the GFP group was significantly higher than that in PPC and CFP groups after three weeks in the solution (Table 4.14).

**Table 4.14:** The difference in the bending stress value across the four groups at week 3 in the solution environment

| **Ordinary ANOVA P-value** | | **<0.0001** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET3 vs. PPCT3** | 195.2 | 149 | 46.16 | 2.052 | Yes | **** | <0.0001 |
| **PPET3 vs. CFP3** | **195.2** | **130.8** | 64.44 | 2.052 | Yes | **** | <0.0001 |
| **PPET3 vs. GFP3** | 195.2 | 160.4 | 34.78 | 2.052 | Yes | **** | <0.0001 |
| **PPCT3 vs. CFP3** | 149 | 130.8 | 18.28 | 2.052 | Yes | **** | <0.0001 |
| **PPCT3 vs. GFP3** | 149 | 160.4 | -11.38 | 2.052 | Yes | ** | 0.0024 |
| **CFP3 vs. GFP3** | 130.8 | 160.4 | -29.67 | 2.052 | Yes | **** | <0.0001 |

### 4.3. Statistical Analyzes of Tensile Test

Across all the four study groups (PPE, PPC, CFP and GFP), the mean of tensile stress was significantly higher in the dry condition than that in the Ringer solution (Figure 4.3). Within each group, the tensile stress reduced after storing in the solution .In general PPE and PPC groups had higher tensile stress compared to that in CFP and GFP group particularly after storing in the solution. The CFP had the lowest (49.71, after one week in the solution) and the highest (92.86, in the dry condition) tensile stress among the four groups.

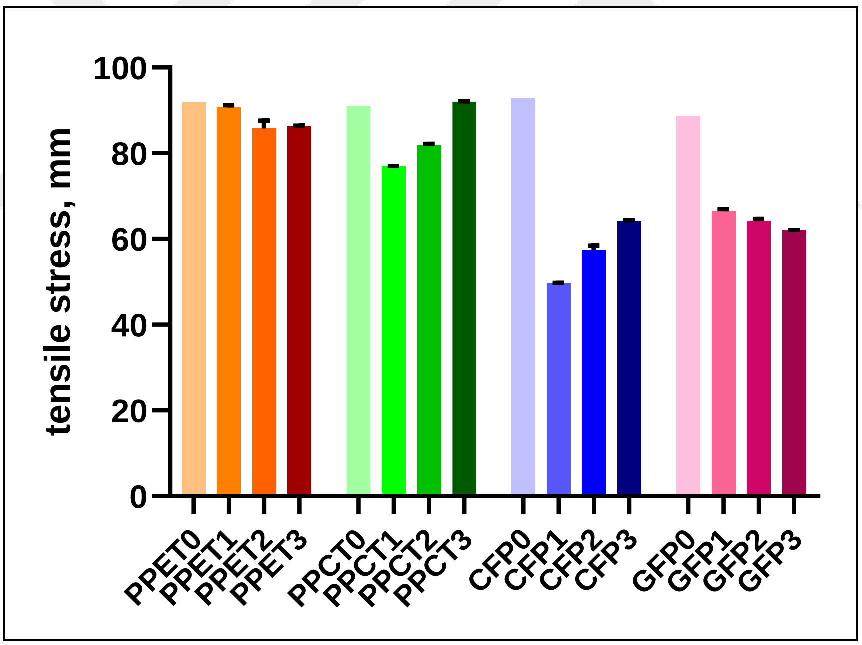


**Figure 4.3:** The difference in the tensile stress value across four different storage conditions/ times and among the four different material groups: pure Peek extrusion (PPE), pure Peek compression (PPC), carbon fiber Peek (CFP) and glass fiber Peek (GFP).

### 4.3.1The tensile stress value across four storage conditions/ times within the PPE group

The mean of tensile stress value within Pure Peek Extrusion (PPE) group ranged from 85.8 mm after one week in Ringer solution to 92.0 mm in the control (dry) condition (Table 4.15 and Figure 4.3). In other words, the tensile stress reduced gradually after storing in Ringer solution. According to repeated measure ANOVA and post-hoc tests, there were two significant (P >0.05) differences in tensile stress within PPE group across different storage conditions/ times .In other words, the tensile stress mean after three weeks in Ringer solution was significantly lower than that in control (dry) condition and after one day in the solution.

**Table 4.15:** The difference in the tensile stress value across different storage conditions/ times within the same group (pure Peek extrusion)

| **Repeated measure ANOVA p value** | | | **0.0327** | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET0 vs. PPET1** | 92 | 90.7 | 1.3 | 0.306 | No | ns | 0.1244 |
| **PPET0 vs. PPET2** | **92** | **85.8** | 6.207 | 1.037 | No | ns | 0.0661 |
| **PPET0 vs. PPET3** | 92 | 86.43 | 5.57 | 0.034 | Yes | **** | <0.0001 |
| **PPET1 vs. PPET2** | 90.7 | 85.8 | 4.907 | 1.314 | No | ns | 0.1559 |
| **PPET1 vs. PPET3** | 90.7 | 86.43 | 4.27 | 0.277 | Yes | * | 0.0107 |
| **PPET2 vs. PPET3** | 85.8 | 86.43 | -0.637 | 1.071 | No | ns | 0.9255 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons) Time-0=dry (control) condition, Time-1=1 day in Ringer solution, Time-2=1 week in Ringer solution, Time-3= 3 weeks in Ringer solution.

### 4.3.2The tensile stress value across four storage conditions/ times within the PPC group

The mean of tensile stress value within Pure Peek Compression (PPC) group ranged from 76.98 mm after one day in Ringer solution to 92.0 mm after three weeks in the solution (Table4.16 and Figure 4.3). In other words, the tensile stress initially (after one day) reduced after storing in Ringer solution but increased after longer period of storing in the solution. According to repeated measure ANOVA and post-hoc tests, there were six significant ($P < 0.05$) differences in tensile stress within PPC group across different storage conditions/ times. The tensile stress means after one day and one week in Ringer solution were significantly lower than that in control (dry) condition and after three weeks in the solution . More specifically, the tensile stress in the control (dry) condition within PPC group was significantly higher than all the three measurements after the storing in the Ringer solution (Table 4.16).

**Table 4.16:** The difference in the tensile stress value across different storage conditions/ times within the same group (pure Peek compression)

| repeated measure ANOVA p value | | | <0.0001 | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPCT0 vs. PPCT1** | 91.06 | **76.98** | 14.08 | 0.032 | Yes | **** | <0.0001 |
| **PPCT0 vs. PPCT2** | 91.06 | 81.88 | 9.185 | 0.188 | Yes | **** | <0.0001 |
| **PPCT0 vs. PPCT3** | 91.06 | **92.01** | -0.946 | 0.067 | Yes | * | 0.0126 |
| **PPCT1 vs. PPCT2** | 76.98 | 81.88 | -4.899 | 0.180 | Yes | ** | 0.0026 |
| **PPCT1 vs. PPCT3** | 76.98 | 92.01 | -15.03 | 0.046 | Yes | **** | <0.0001 |
| **PPCT2 vs. PPCT3** | 81.88 | 92.01 | -10.13 | 0.218 | Yes | **** | <0.0001 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons). The measurements of tensile stress are in mm.

### 4.3.3 The tensile stress value across four storage conditions/ times within the CFP group

The mean of tensile stress value within Carbon Fiber Peek (CFP) group ranged from 49.7 mm after one day in the solution to 92.9 mm in the dry condition (Table 4.17 and Figure 4.3). In other words, the tensile stress reduced sharply after storing in Ringer solution. According to repeated measure ANOVA and post-hoc tests, there were six significant (P <0.05) differences in tensile stress within CFP group across different storage conditions/ times. The tensile stress means significantly reduced after one day in Ringer solution compared to dry condition. More specifically, the tensile stress in the dry condition within CFP group was significantly higher than all the three measurements after the storing in the Ringer solution (Table4.17).

**Table 4.17:** The difference in the tensile stress value across different storage conditions/times within the same group (carbon fiber Peek)

| **repeated measure ANOVA p value** | | | **0.0002** | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **CFP0 vs. CFP1** | **92.86** | **49.71** | 43.15 | 0.037 | Yes | **** | <0.0001 |
| **CFP0 vs. CFP2** | 92.86 | 57.46 | 35.39 | 0.580 | Yes | **** | <0.0001 |
| **CFP0 vs. CFP3** | 92.86 | 64.25 | 28.61 | 0.087 | Yes | **** | <0.0001 |
| **CFP1 vs. CFP2** | 49.71 | 57.46 | -7.754 | 0.617 | Yes | * | 0.0157 |
| **CFP1 vs. CFP3** | 49.71 | 64.25 | -14.54 | 0.123 | Yes | **** | <0.0001 |
| **CFP2 vs. CFP3** | 57.46 | 64.25 | -6.786 | 0.494 | Yes | * | 0.0133 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

### 4.3.4 The tensile stress value across four storage conditions/ times within the GFP group

The mean of tensile stress value within Glass Fiber Peek (GFP) group ranged from 62.0 mm after three weeks in the solution to 88.7 mm the dry condition (Table 4.18 and Figure 4.3). In other words, the tensile stress reduced sharply after storing in Ringer solution .According to repeated measure ANOVA and post-hoc tests, there were six significant (P <0.05) differences in tensile stress within GFP group across different storage conditions/ times .The tensile stress means significantly reduced after one day in Ringer solution compared to dry condition .More specifically, the tensile stress in the dry condition within CFP group was significantly higher than all the three measurements after the storing in the Ringer solution (Table 4.18).

**Table 4.18**: The difference in the tensile stress value across different storage conditions/times within the same group (glass fiber Peek)

| **repeated measure ANOVA p value** | | | **<0.0001** | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **GFP0 vs. GFP1** | 88.71 | 66.67 | 22.05 | 0.1533 | Yes | **** | <0.0001 |
| **GFP0 vs. GFP2** | 88.71 | 64.26 | 24.46 | 0.2588 | Yes | **** | <0.0001 |
| **GFP0 vs. GFP3** | **88.71** | **62.05** | 26.66 | 0.05333 | Yes | **** | <0.0001 |
| **GFP1 vs. GFP2** | 66.67 | 64.26 | 2.41 | 0.3108 | Yes | * | 0.0403 |
| **GFP1 vs. GFP3** | 66.67 | 62.05 | 4.615 | 0.1 | Yes | **** | <0.0001 |
| **GFP2 vs. GFP3** | 64.26 | 62.05 | 2.205 | 0.2683 | Yes | * | 0.036 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

### 4.3.5 The Differences in the Tensile Stress among the four groups (PPE, PPC, CFP and GFP) within each time point

#### 4.3.5.1 The tensile stress value across the four groups after one day in the solution environment

The mean of tensile stress on day 1 in the Ringer solution ranged from 49.7 mm for CFP group and 90.7 mm for PPE group. In other words, the CFP had the lowest tensile stress while the PPE group had the highest tensile stress after one day in the solution (Table 4.19). According to one-way ANOVA, there were six significant differences among the four groups. According to Tukey's multiple comparisons test, the mean of tensile stress in PPE and PPC groups were significantly (P <0.05) higher compared to that in the CFP and GFP groups.

More specifically, the mean of tensile stress in PPE group was significantly (P <0.05) higher (90.7 mm) compared to that in the other three groups (PPC, CFP and GFP after one day storing in the solution.

**Table 4.19**: The difference in the tensile stress value across the four groups after day 1 in the solution environment

| **Ordinary ANOVA p value** | | **<0.0001** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET1 vs. PPCT1** | **90.7** | 76.98 | 13.73 | 0.2447 | Yes | **** | <0.0001 |
| **PPET1 vs. CFP1** | 90.7 | 49.71 | 40.99 | 0.2447 | Yes | **** | <0.0001 |
| **PPET1 vs. GFP1** | 90.7 | 66.67 | 24.04 | 0.2447 | Yes | **** | <0.0001 |
| **PPCT1 vs. CFP1** | 76.98 | **49.71** | 27.27 | 0.2447 | Yes | **** | <0.0001 |
| **PPCT1 vs. GFP1** | 76.98 | 66.67 | 10.31 | 0.2447 | Yes | **** | <0.0001 |
| **CFP1 vs. GFP1** | 49.71 | 66.67 | -16.95 | 0.2447 | Yes | **** | <0.0001 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

#### 4.3.5.2 The tensile stress value across the four groups after one week in the solution environment

The mean of tensile stress after one week in the Ringer solution ranged from 57.5 mm for CFP group and 85.8 mm for PPE group. Like after one day in the solution, the CFP had the lowest tensile stress while the PPE group had the highest tensile stress after one week in the solution (Table 4.20). According to one-way ANOVA, there were six significant differences among the four groups.

According to Tukey's multiple comparisons test, the mean of tensile stress in PPE and PPC groups were significantly (P<0.05) higher compared to that in the CFP and GFP groups .More specifically, the mean of tensile stress in PPE group was significantly (P<0.05) higher (85.8 mm) compared to that in the other three groups (PPC, CFP and GFP after one week storing in the solution.

**Table 4.20:** The difference in the tensile stress value across the four groups after one week in the solution environment

| **ordinary ANOVA p value** | | **<0.0001** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET2 vs. PPCT2** | 85.8 | 81.88 | 3.919 | 0.8704 | Yes | ** | 0.0086 |
| **PPET2 vs. CFP2** | **85.8** | **57.46** | 28.33 | 0.8704 | Yes | **** | <0.0001 |
| **PPET2 vs. GFP2** | 85.8 | 64.26 | 21.54 | 0.8704 | Yes | **** | <0.0001 |
| **PPCT2 vs. CFP2** | 81.88 | 57.46 | 24.41 | 0.8704 | Yes | **** | <0.0001 |
| **PPCT2 vs. GFP2** | 81.88 | 64.26 | 17.62 | 0.8704 | Yes | **** | <0.0001 |
| **CFP2 vs. GFP2** | 57.46 | 64.26 | -6.791 | 0.8704 | Yes | *** | 0.0002 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

#### 4.3.5.3 The tensile stress value across the four groups after three weeks in the solution environment

The mean of tensile stress after one week in the Ringer solution ranged from 62.0 mm for CFP group and 92.0 mm for PPC group. In other words, the CFP had the lowest tensile stress while the PPC group had the highest tensile stress after one week in the solution (Table 4.21).

According to one-way ANOVA, there were six significant differences among the four groups. According to Tukey's multiple comparisons test, the mean of tensile stress in PPE and PPC groups were significantly (P <0.05) higher compared to that in the CFP and GFP groups. More specifically, the mean of tensile stress in PPC group was significantly (P <0.05) higher (92.0 mm) compared to that in the other three groups (PPE, CFP and GFP after three weeks storing in the solution.

**Table4.21**: The difference in the tensile stress value across the four groups after week 3 in the solution environment

| **ordinary ANOVA P-value** | | **<0.0001** | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Significant?** | **Summary** | **Adjusted P Value** |
| **PPET3 vs. PPCT3** | 86.43 | **92.01** | -5.574 | 0.0894 | Yes | **** | <0.0001 |
| **PPET3 vs. CFP3** | 86.43 | 64.25 | 22.18 | 0.0894 | Yes | **** | <0.0001 |
| **PPET3 vs. GFP3** | 86.43 | **62.05** | 24.38 | 0.0894 | Yes | **** | <0.0001 |
| **PPCT3 vs. CFP3** | 92.01 | 64.25 | 27.76 | 0.0894 | Yes | **** | <0.0001 |
| **PPCT3 vs. GFP3** | 92.01 | 62.05 | 29.96 | 0.0894 | Yes | **** | <0.0001 |
| **CFP3 vs. GFP3** | 64.25 | 62.05 | 2.2 | 0.0894 | Yes | **** | <0.0001 |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

### 4.4. Statistical Analyzes of Hardness Test

The mean hardness value was the highest after one day in the solution across the four groups then reduced over time (Figure 4.4). The CFP group had the highest hardness value while both PPE and PPC had the lowest hardness value among the Four-study group.

The CFP group after one day in the solution had the highest hardness value (54.79) while the PPE had the lowest hardness value (27.56) after three weeks in the solution.

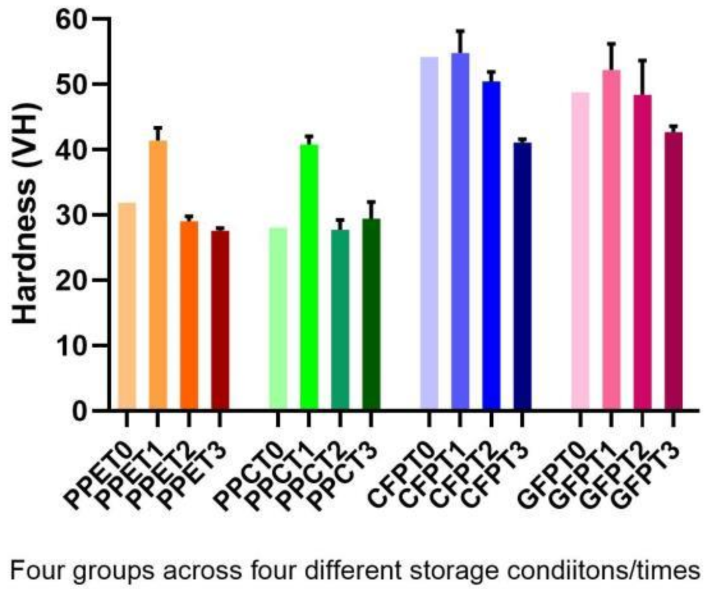


**Figure 4.4**: The difference in the hardness value across four different storage conditions/ times and among the four different material groups: pure Peek extrusion (PPE), pure Peek compression (PPC), carbon fiber Peek (CFP) and glass fiber Peek (GFP)

### 4.4.1 The hardness value across four storage conditions/ times within PPE group

The mean of hardness value within pure Peek extrusion (PPE) ranged from 27.6 after three weeks in Ringer solution and 41.4 after one day in the solution (Table4.22 and Figure 4.4). In other words, the storage condition after one day in

Ringer solution had the highest hardness value while after three weeks in the same solution had the lowest hardness value within PPE group .According to repeated measure ANOVA, there was significant difference in hardness value within PPE across different storage conditions/ times. According to post hoc test (Tukey's multiple comparisons), there were four significant differences in the hardness value across different storage conditions/ times within the pure Peek extrusion group. The hardness of PPE after one day in Ringer solution was significantly (P <0.05) higher compared to the other three storage conditions: Control (dry) group and storage in Ringer solution after one and three weeks. Additionally, the hardness at control (dry) condition was significantly higher compared to the hardness after three weeks in Ringer solution within the same group (PPE) (Table 4.22)

**Table 4.22**: The difference in the hardness value across different storage conditions/ times within the same group (pure Peek extrusion)

| **Repeated measures ANOVA P-value** | **<0.0001** | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P Value** | **Significant?** | **Summary** |
| **PPET0 vs. PPET1** | 31.9 | **41.4** | -9.50 | 0.919 | 0.0002 | Yes | *** |
| **PPET0 vs. PPET2** | 31.9 | 29 | 2.90 | 0.919 | 0.0727 | No | ns |
| **PPET0 vs. PPET3** | 31.9 | **27.56** | 4.34 | 0.919 | 0.013 | Yes | * |
| **PPET1 vs. PPET2** | 41.4 | 29 | 12.40 | 0.919 | <0.0001 | Yes | **** |
| **PPET1 vs. PPET3** | 41.4 | 27.56 | 13.84 | 0.919 | <0.0001 | Yes | **** |
| **PPET2 vs. PPET3** | 29 | 27.56 | 1.44 | 0.919 | 0.4611 | No | ns |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)
Time-0=dry (control) condition, Time-1=1 day in Ringer solution, Time-2=1 week in Ringer solution, Time-3= 3 weeks in Ringer solution.

### 4.4.2 The hardness value across four storage conditions/ times within PPC group

The mean of hardness value within pure Peek compression (PPC) ranged from 27.7 after one week in Ringer solution and 41.8 after one day in the solution (Table 4.23 and Figure 4.4). In other words, the storage condition after one day in Ringer solution had the highest hardness value while after one week in the same solution had the lowest hardness value within PPC group .According to repeated measure ANOVA, there was significant ($P<0.05$) difference in hardness value within PPC group across different storage conditions/ times. According to post-hoc test (Tukey's multiple comparisons), there were three significant differences in the hardness value across different storage conditions/ times within the PPC group. The hardness of PPC after one day in Ringer solution was significantly ($P<0.05$) higher compared to the other three storage conditions: Control (dry) group and storage in Ringer solution after one and three weeks (Table 4.23).

**Table 4.23:** The difference in the <u>hardness</u> value across different storage conditions/ times within the same group (pure Peek compression)

| Repeated measures ANOVA P-value | <0.0001 | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P Value** | **Significant?** | **Summary** |
| **PPCT0 vs. PPCT1** | 28.02 | **40.77** | -12.75 | 0.919 | <0.0001 | Yes | **** |
| **PPCT0 vs. PPCT2** | 28.02 | **27.73** | 0.29 | 0.919 | 0.9884 | No | ns |
| **PPCT0 vs. PPCT3** | 28.02 | 29.42 | -1.40 | 0.919 | 0.4802 | No | ns |
| **PPCT1 vs. PPCT2** | 40.77 | 27.73 | 13.04 | 0.919 | <0.0001 | Yes | **** |
| **PPCT1 vs. PPCT3** | 40.77 | 29.42 | 11.35 | 0.919 | <0.0001 | Yes | **** |
| **PPCT2 vs. PPCT3** | 27.73 | 29.42 | -1.69 | 0.919 | 0.3436 | No | ns |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons). The measurements of bending stress are in mm.

### 4.4.3 The hardness value across four storage conditions/ times within CFP group

The mean of hardness value within carbon fiber Peek (CFP) ranged from 41.1 after three weeks in Ringer solution and 54.8 after one day in the solution (Table 4.24 and Figure 4.4). In other words, the storage condition after one week in Ringer solution had the highest hardness value while after three weeks in the same solution had the lowest hardness value within CFP group .According to repeated measure ANOVA, there was significant (P<0.05) difference in hardness value within CFP group across different storage conditions/ times. According to post-hoc test (Tukey's multiple comparisons), there were three significant differences in the hardness value across different storage conditions/ times within the CFP group. The hardness of PPC after three weeks in Ringer solution was significantly (P<0.05) lower compared to the other three storage conditions: Control (dry) group and storage in Ringer solution after one and two weeks (Table 4.24).

**Table 4.24:** The difference in the hardness value across different storage conditions/times within the same group (carbon fiber Peek)

| **Repeated measures ANOVA P-value** | **0.0005** | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P Value** | **Significant?** | **Summary** |
| **CFPT0 vs. CFPT1** | 54.19 | **54.79** | -0.60 | 1.624 | 0.9809 | No | ns |
| **CFPT0 vs. CFPT2** | 54.19 | 50.44 | 3.75 | 1.624 | 0.1973 | No | ns |
| **CFPT0 vs. CFPT3** | 54.19 | 41.07 | 13.12 | 1.624 | 0.0008 | Yes | *** |
| **CFPT1 vs. CFPT2** | 54.79 | 50.44 | 4.36 | 1.624 | 0.1261 | No | ns |
| **CFPT1 vs. CFPT3** | 54.79 | 41.07 | 13.72 | 1.624 | 0.0006 | Yes | *** |
| **CFPT2 vs. CFPT3** | 50.44 | 41.07 | 9.36 | 1.624 | 0.0048 | Yes | ** |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

### 4.4.4 The hardness value across four storage conditions/ times within GFP group

The mean of hardness value within glass fiber Peek (GFP) ranged from 42.7 after three weeks in Ringer solution and 52.2 after one week in the solution (Table 4.25 and Figure 4.4). In other words, the storage condition after one week in

Ringer solution had the highest hardness value while after three weeks in the same solution had the lowest hardness value within GFP group. According to repeated measure ANOVA and post-hoc tests, there was no significant (P >0.05) difference in hardness value within GFP group across different storage conditions/ times (Table 4.25).

**Table4.25:** The difference in the hardness value across different storage conditions/times within the same group (glass fiber Peek)

| Repeated measures ANOVA p value | 0.1026 | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P Value** | **Significant?** | **Summary** |
| **GFPT0 vs. GFPT1** | 48.8 | 52.2 | -3.4 | 3.094 | 0.7031 | No | ns |
| **GFPT0 vs. GFPT2** | 48.8 | 48.4 | 0.4 | 3.094 | 0.9991 | No | ns |
| **GFPT0 vs. GFPT3** | 48.8 | 42.7 | 6.1 | 3.094 | 0.2944 | No | ns |
| **GFPT1 vs. GFPT2** | 52.2 | 48.4 | 3.8 | 3.094 | 0.6334 | No | ns |
| **GFPT1 vs. GFPT3** | 52.2 | 42.7 | 9.5 | 3.094 | 0.0791 | No | ns |
| **GFPT2 vs. GFPT3** | 48.4 | 42.7 | 5.7 | 3.094 | 0.3412 | No | ns |

### 4.4.5 The Differences in the hardness value among the four groups (PPE, PPC, CFP and GFP) within each time point

#### 4.4.5.1 The hardness value across the four groups after day 1 in the solution environment

The mean of hardness value on day 1 in the Ringer solution ranged from

41.4 for PPE group and 54.8 for CFP. In other words, the PPE had the lowest hardness value while the CFP group had the highest hardness value after one day in the solution (Table 4.26). According to one-way ANOVA, there were four significant differences among the four groups. According to Tukey's multiple comparisons test, the mean of hardness value in PPE and PPC groups were significantly (P<0.05) lower compared to that in the CFP and GFP groups.

**Table 4.26:** The difference in the hardness value across the four groups after day 1 in the solution environment

| **One-Way ANOVA P-value** | **0.0005** | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P-Value** | **Significant?** | **Summary** |
| **PPET1 vs. PPCT1** | **41.4** | 40.77 | 0.6267 | 2.32 | 0.9926 | No | ns |
| **PPET1 vs. CFPT1** | 41.4 | **54.79** | -13.39 | 2.32 | 0.0019 | Yes | ** |
| **PPET1 vs. GFPT1** | 41.4 | 52.2 | -10.8 | 2.32 | 0.0071 | Yes | ** |
| **PPCT1 vs. CFPT1** | 40.77 | 54.79 | -14.02 | 2.32 | 0.0014 | Yes | ** |
| **PPCT1 vs. GFPT1** | 40.77 | 52.2 | -11.43 | 2.32 | 0.0051 | Yes | ** |
| **CFPT1 vs. GFPT1** | 54.79 | 52.2 | 2.593 | 2.32 | 0.6895 | No | ns |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

#### 4.4.5.2The hardness value across the four groups after week 1 in the solution environment

100

The mean of hardness value on week 1 in the Ringer solution ranged from 27.7 for PPC group and 50.4 for CFP. In other words, the PCE had the lowest hardness value while the CFP group had the highest hardness value after one week in the solution (Table 4.27). According to one-way ANOVA, there were four significant differences among the four groups. According to Tukey's multiple comparisons test, the mean of hardness value in PPE and PPC groups were significantly (P<0.05) lower compared to that in the CFP and GFP groups at week 1 in the solution (Table 4.27).

**Table 4.27:** The difference in the hardness value across the four groups after week 1 in the solution environment

| **ordinary ANOVA p value** | **0.0001** | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P Value** | **Significant?** | **Summary** |
| **PPET2 vs. PPCT2** | 29 | **27.73** | 1.267 | 2.325 | 0.9454 | No | ns |
| **PPET2 vs. CFPT2** | 29 | **50.44** | -21.44 | 2.325 | <0.0001 | Yes | **** |
| **PPET2 vs. GFPT2** | 29 | 48.4 | -19.4 | 2.325 | 0.0001 | Yes | *** |
| **PPCT2 vs. CFPT2** | 27.73 | 50.44 | -22.7 | 2.325 | <0.0001 | Yes | **** |
| **PPCT2 vs. GFPT2** | 27.73 | 48.4 | -20.67 | 2.325 | <0.0001 | Yes | **** |
| **CFPT2 vs. GFPT2** | 50.44 | 48.4 | 2.037 | 2.325 | 0.8171 | No | ns |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

#### 4.4.5.3 The hardness value across the four groups after week 3 in the solution environment

The mean of hardness value on week 3 in the Ringer solution ranged from 27.6 for PPE group and 42.7 for GFP. In other words, the PPE had the lowest hardness value while the GFP group had the highest hardness value after one week in the solution (Table 4.28). According to one-way ANOVA, there were four significant differences among the four groups. According to Tukey's multiple comparisons test, the mean of hardness value in PPE and PPC groups were significantly (P<0.05) lower compared to that in the CFP and GFP groups at week 1 in the solution (Table 4.28).

**Table 4.28**: The difference in the hardness value across the four groups after week 3 in the solution environment

| Ordinary ANOVA p-value | 0.0001 | | | | | | |
|---|---|---|---|---|---|---|---|
| **Tukey's multiple comparisons test** | **Mean 1** | **Mean 2** | **Mean Diff.** | **SE of diff.** | **Adjusted P Value** | **Significant?** | **Summary** |
| **PPET3 vs. PPCT3** | **27.56** | 29.42 | -1.863 | 1.137 | 0.4111 | No | ns |
| **PPET3 vs. CFPT3** | 27.56 | 41.07 | -13.52 | 1.137 | <0.0001 | Yes | **** |
| **PPET3 vs. GFPT3** | 27.56 | **42.7** | -15.14 | 1.137 | <0.0001 | Yes | **** |
| **PPCT3 vs. CFPT3** | 29.42 | 41.07 | -11.65 | 1.137 | <0.0001 | Yes | **** |
| **PPCT3 vs. GFPT3** | 29.42 | 42.7 | -13.28 | 1.137 | <0.0001 | Yes | **** |
| **CFPT3 vs. GFPT3** | 41.07 | 42.7 | -1.627 | 1.137 | 0.5163 | No | ns |

*Significant (P-value<0.05) according to post hoc test (Tukey's multiple comparisons)

Across the three time-measurements (day 1, week 1 and week 3) in the solution, the mean of hardness value in PPE and PPC groups were significantly (P-value<0.05) lower compared to that in the CFP and GFP groups.

### 4.5 FESEM Analyzing Evaluation

This invitro study has exmined the deep shape of fracture line with FESEM (field emission SEM which has the ability to produce clearer, less electrostatically distorted images with spatial resolution down to 1 1/2 nanometers – three to six times better Compared with convention scanning electron microscopy (SEM). PPE sample groups with its extrusion pressurized and forced matrix showed a compact and fewer voids mold in comparision to spaced matrices of PPC samples type and which confirmed the behavior of PPE samples at different storage solution intervals with higher degrees of stress values in comparision to PPC types which needed longer time of storage at ringer solution to show higher stress strength (Figures 4.5).

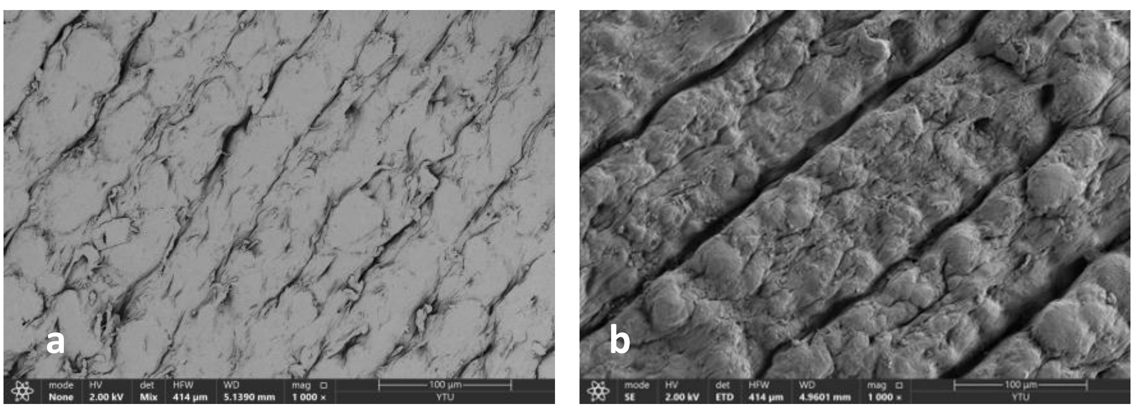


**Figure 4.5**: **a.** PPE sample after 1 week storage **b.** PPC sample after 1week storage

Reinforcement fiber PEEK groups demonstrated higher stress strength values with GFP samples rather than CFP samples although both showed almost uniform matrix shape under FESEM with more coarse and gruff shape of CFP samples in relation to

GFP samples with less roughness format after storage in ringer solution at different time intervals (Figures 4.6).

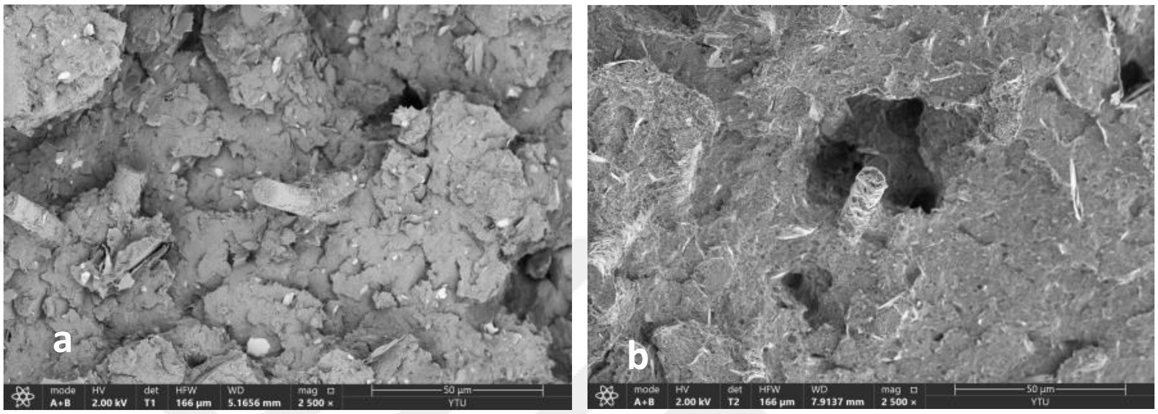


**Figure 4.6**: **a.** CFP sample after 1 week storage **b.** GFP sample after 1week storage

### 4.6 Optic Microscope Evaluation

Evaluation of the fracture types observed under the optical microscope (x10) of the fracture formed on the different PEEK samples were made according to the fracture line pattern after subjected to mechanical tests.There was a clear difference in the pattern of fracture line found between certain groups; (Figures 4.5, 6, 7, 8).the PPE and CFP samples showed the irregularities and loss of PEEK material during mechanical test application while the PPC and GFP samples showed the less irregular pattern and loss of PEEK material after subjected to the mechanical test, GFP samples showed the most organized pattern of fracture line.

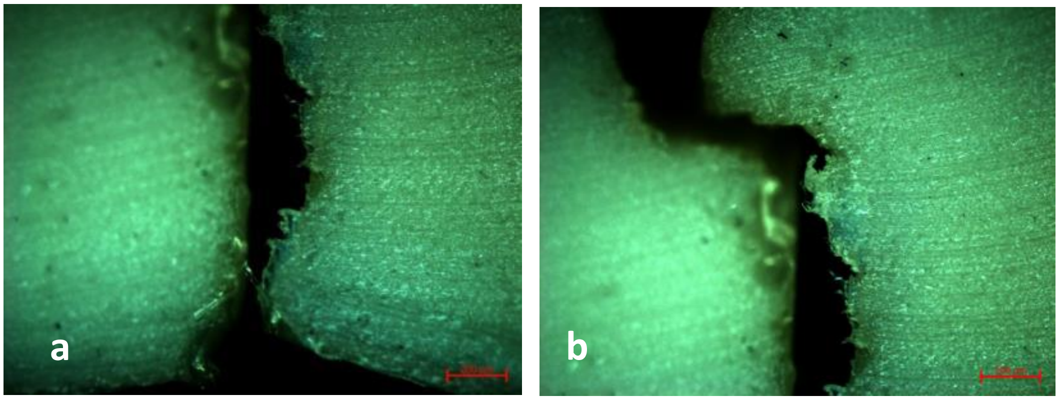


**Figure 4.7(a,b)** : Fracture line pattern of PPE samples

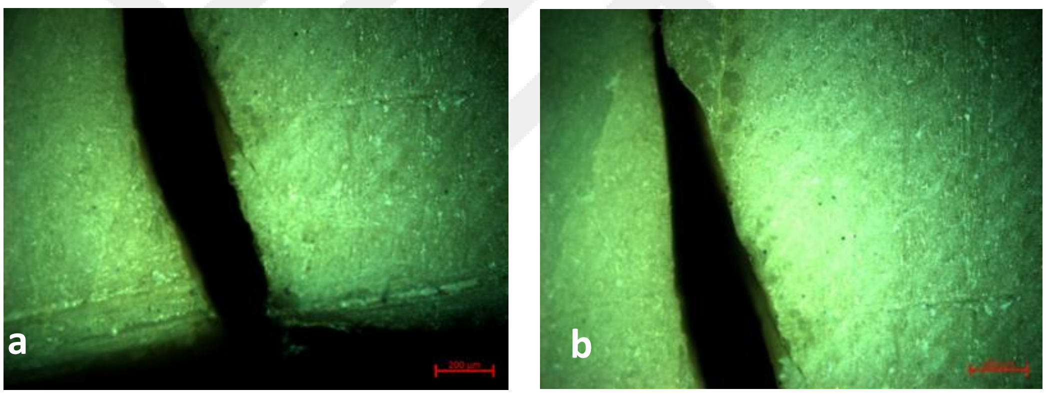


**Figure 4.8(a,b)** : Fracture line pattern of PPC samples

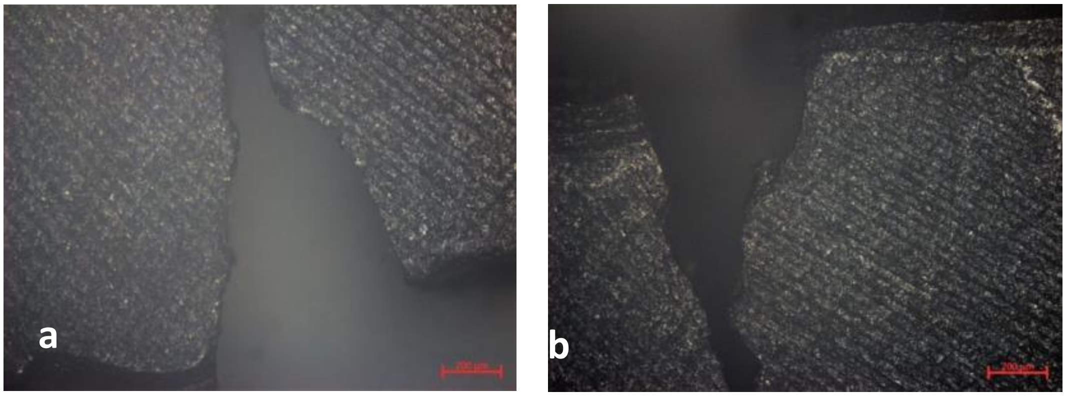


**Figure 4.9(a,b)** : Fracture line pattern of <u>CFP</u> samples

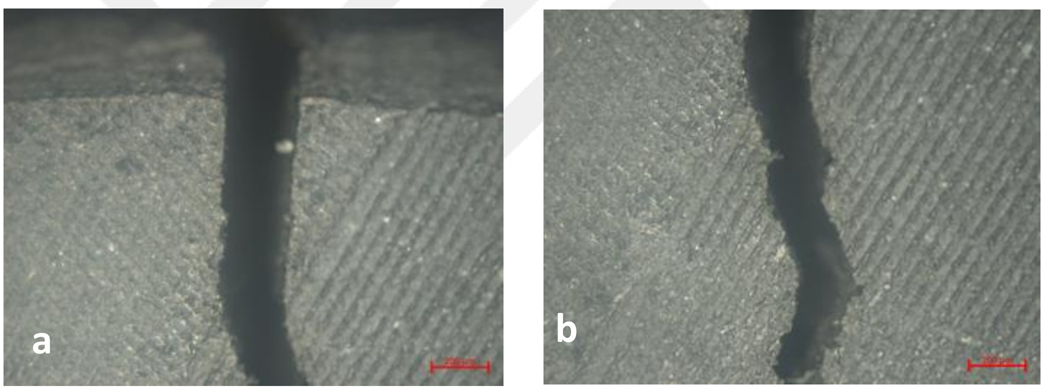


**Figure 4.10(a,b)** : Fracture line pattern of <u>GFP</u> samples

# 5. DISCUSSION

There are three hypotheses in this study. The first assumption is; pure PEEK compression groups (PPC) show higher mechanical stress degrees than other pure PEEK extrusion groups (PPE) while the stress and strain values showed no significant difference between the two pure PEEK groups.

The second hypothesis is; the effect of PEEK fiber reinforcement on stress and strain strength values on different mechanical properties compared to the pure PEEK group; in this study, we used two types of fiber-reinforced PEEK; glass fiber and carbon fiber.The reinforcement fiber PEEK types showed higher rigidity and less ductility scheme than pure PEEK types.The third hypothesis discussed in this study is the effect of storage environment on different PEEK samples; pure PEEK and fiber PEEK. Mechanical tests showed different results between different PEEK samples at different time storage intervals.

Advances in dental materials and technology captured increased attention because of their individual properties and structures (Fernandez and Col., 2021). Development of dental materials has strengthened different applications in dentistry and polymers are that class of material that showed intelligibly improved properties, such as strength, dimensional stability, electrical conductivity, barrier performance, biocompatibility, low plaque affinity and good aesthetics (Dunlop and Bissessur, 2020). PEEK is an emerging type of thermoplastic engineering material with outstanding mechanical and thermal properties as suitable substitute to traditional Implantation in the dental fields with features compatible with those of tooth tissue (Kurtz and Devine, 2007). Compared to pure PEEK considerable studies has recently been directed toward developing PEEK reinforced fibers to improve various physical, mechanical and barrier properties to allow their application to restorative dentistry (Sandler and Col., 2002; Rahmitasari and Col., 2017). Various reinforced PEEK composites have been developed, such as carbon fiber-reinforced PEEK (CFR-PEEK) and glass fiber-reinforced PEEK (GFR-PEEK) with varying fiber lengths and orientations. CFR-PEEK has been of interest to the medical implant community due to its excellent mechanical properties, and biocompatibility. This material can be manufactured in several shapes with various physical, mechanical, and surface properties (Schwitalla and Col., 2015; Wiesli and Col., 2015; Chun and Col., 2014).

In this thesis study, we performed in vitro examinations to discuss the improvement of different mechanical tests applied to pure PEEK and reinforced PEEK materials. PEEK material will be investigated after applying two alternative types of reinforcing fibers. With this experimental work, it was aimed to strengthen some mechanical properties of PEEK and improve their advanced dental applications. In the oral environment, restorations are subjected to stresses from mastication action. These forces act on teeth and/ or material producing different reactions, the knowledge and interpretation of how these materials behave under such forces are relevant to understand the performance of the material (Wilson, 1990; Gonzalez et al., 2015).

Compressive, bending, tensile and, hardness forces were carried out in laboratory setting and they showed an important comparison between the different PEEK samples at different storage environments.

PPE and PPC samples showed no significant difference in compression stress between them within the dry condition storage although the PPC type registered higher pressure stress values than PPE group but still both showed the same deformation and elongation shape after applying pressure force.

PPE revealed a fluctuation over the four-time measurements related to the pressure strength test, it has reduced after one day storing in the Ringer solution then increased after one week and decreased again after three weeks of storage in the solution.

For PPC there were five significant differences in the pressure stress across different storage conditions/times. The pressure stress in the dry condition and after three weeks in Ringer solution were significantly lower than after one day and one week in the solution. More specifically, the pressure stress in the control (dry) condition within PPC group was significantly less than all the three measurements after the storing in the Ringer solution. According to the tensile strength test the results there were no significant difference between PPE group and PPC group within the dry condition storage although the PPE type registered

a higher tensile stress value than PPC groups.Tensile strength first increases and then converges to a stable level of 90±2 MPa. The elongation behavior at break also showed an increasing trend at first and then decreased. The tensile strength of PEEK gradually increases and tends to stabilize after, with fracture line position which was in the middle of the samples for PPE groups and on the sides of the samples for the PPC groups at the different storage time intervals.

For PPE the tensile stress reduced gradually after storing in Ringer solution from dry condition to 1 day, 1 week, and 3 weeks respectively. While for PPC the tensile stress initially (after one day) reduced after storing in Ringer solution but increased after longer period of storing in the solution. The tensile stress means after one day and one week in Ringer solution was significantly lower than that in control (dry) condition and after three weeks in the solution .More specifically, the tensile stress in the control (dry) condition within the PPC group was significantly higher than all three measurements after the storing in the Ringer solution.

For the bending stress there was a significant difference at dry environment (control group) between PPE and PPC group. PPC registered high bending strength value of 178 MPa in comparison to 168.5 MPa for PPE groups. PPE displayed significantly higher values after three weeks in the solution compared to after one week in the solution. However, there were no significant changes in the bending stress between dry condition from one side and one day and one week in the solution from the other side. For PPC there was fluctuation in the bending stress over the four-time measurements. More specifically, the bending stress reduced after one day storing in the Ringer solution then increased after one week and decreased again after three weeks of storage in the solution. More specifically, the mean of bending stress was significantly lower after one day and three weeks in the solution than that in the dry condition. Both of PPE group and PPC group found with almost equal shape of bending in the middle of sample without fracture after subjected to bending test at different storage environment/ time.

For hardness test PPE showed four significant differences in the hardness value across different storage conditions/ times. The mean value was higher in PPE than PPC with no significant difference between them at dry environment storage (control group) with 30±2 VHN. Both of pure PEEK samples showed increase in the penetration of the test indenter to the surface of material and by consequence reduce the hardness of the tested material. The hardness of PPE after one day in Ringer solution was higher compared to the other three storage conditions: control (dry) group and storage in Ringer solution after one week and three weeks. Additionally, the hardness at control (dry) condition was significantly higher compared to the hardness after three weeks in Ringer solution within the same group (PPE). For hardness test evaluation there was a valuable difference in hardness value within PPC group across different storage conditions/ times. The hardness of PPC after one day in Ringer solution was higher compared to the other three storage conditions: Control (dry) group and storage in Ringer solution after one and three weeks. These both behavior of PPE and PPC groups revealed almost uniform shape of sample indentation after subjected to Vickers micro hardness test.

All these findings related to pure PEEK types used in our study explained that PPE group and PPC group had presented uneven reactions with every mechanical test for each time interval at ringer storage environment. These variations were explained by number of researches; (James et al, 1986; Yang et al, 2017) due to the difference in crystallity pattern between PPC and PPE groups that affect the matrix behavior toward applied mechanical forces. The effect of processing, structure, and properties of PEEK thermoplastics showed that under normal processing conditions for high performance composites, the mechanical properties may not be strongly affected by different levels of crystallinity was discussed (Siddiq and Kennedy, 2010) . These variations also were explained by Roeder. 2019 who established the resultant microstructure of PPC as fully dense and maximum in crystallinity and lower molecular weight PEEK with a smaller particle size resulting in improved mechanical properties (Roeder et al., 2019). The thermal processes involved in extrusion PEEK type and annealing result in slight variation in mechanical property while the small powder size of PPC granules resulted in more uniform property in the final shape (Kurtz, 2011; Yao & Lou, 2020).

The PPE and PPC exhibit a brittle fracture and the load decreased to a constant value until the material failed at the fracture point. This fracture behavior comes in agreement with the explanation of Gao et al, 2022. The ultimate tensile strength was the value of the maximum tensile load, and the tensile curves proved that the pure PEEK is a ductile material. The PPE and PPC samples and the elongation at break is relatively small, this mechanical behavior was agreed with the pattern of elongation and fracture of tensile stress strength (Li et al, 2023; Offringa, 2010; Rinaldi et al, 2021). The effect of processing temperature and cooling of extruded PEEK have a pronounced influence on the crystallization process and therefore affect the properties of finished objects to a large extent (Yang et al, 2017). Conducted experiments on extruded and compression types of PEEK stated increas in mechanical properties (14% increases in compression strength) but is not able to decrease the undesired porosity formed during PEEK processing (Basgul et al, 2020). The smooth powder grade of PEEK polymer has been recommended for the compression method though this process may produce higher crystallinity and compression strength (Wang et al, 2020; Zanjanijam et al, 2020; Perng et al, 1999).

On the other hand processing, structure, and properties of PEEK semicrystalline was investigated showed that under normal processing conditions for high performance composites, the mechanical properties may not be strongly affected by different levels of crystallinity (Seferis et al., 1986).

CFP and GFP groups exhibited a valuable difference in pressure stress between in dry environment (control groups). GFP samples showed the higher pressure stress value and more stable shape behavior in comparison to the other PEEK samples measured and showed more stable shape in comparison to CFP groups who showed elongation and sample deformation after submitted to compression test. The pressure stress for CFP reduced after one day storing in the Ringer solution then increased after 1-3 weeks of storage in the solution and it was significantly higher after 1-3 weeks in the solution compared to the dry condition and after one day in the solution. Whereas for GFP groups pressure stress showed a fluctuation over the four-time measurements. In particullary, the pressure stress reduced after one day storing in the Ringer solution then increased after one week and decreased again after three weeks of storage in the solution. The mean of pressure stress was significantly lower after three weeks in the solution than that in the dry condition and after one week in the solution.

For tensile test GFP group registered lower tensile stress value than CFP group (control dry environment) but still both GFP and CFP revealed almost same pattern of fracture line at the side of specimens with fractured immediately when the load attained the ultimate tensile stress with almost no elongation shape rather than fracture in comparison to the PPE and PPC. CFP group appeared with significantly reduced values after one day in Ringer solution compared to dry condition, their values reduced sharply after storing in Ringer solution with significant differences across different storage conditions/ times.

More specifically, the tensile stress in the dry condition within CFP group was higher than all the three measurements after the storing in the Ringer solution. While for GFP group they almost showed the same results of CFP group with stress values reduced sharply after storing in Ringer solution with significant differences in tensile stress within GFP group across different storage conditions/ times. Stress values significantly reduced after one day in Ringer solution compared to dry condition. Both of CFP and GFP groups showed no elongation with fracture line on the sides of the tested samples rather than in the middle.

The bending stress GFP group and CFP was with low values at dry condition than the pure PEEK groups, they revealed an immediate fracture at low bending stress values of 142± 2 MPa.  CFP group showed inconstancy over the four-time measurements. The bending stress increased sharply after one day storing in the Ringer solution compared to dry condition then reduced over time in the solution. The bending stress means in the dry condition and after three weeks in Ringer solution were significantly lower than that after one day in the solution. Moreover, the bending stress in the dry condition was higher than that after three weeks in the solution.  For GFP group bending stress there was a variation over the four-time measurements. The bending stress means in the dry condition was lower than the three measurements after storing in the Ringer solution.

CFP groups and GFP groups improved their excellent hardness values which were higher than the PPE and PPC groups Though CFP groups scored higher hardness values than GFP. They showed decrease in the penetration of the test indenter to the surface of  material and by consequence raise in the hardness of the tested material in comparison to the PPE and PPC groups. The hardness test for CFP group with storage condition after one week in Ringer solution demonstrated the highest hardness value, while after three weeks in the same solution he showed the lowest value for hardness stress. There were three significant differences in the hardness value across different storage conditions/ times within the CFP group. The hardness stress after three weeks in Ringer solution was lower compared to the other three storage conditions; control (dry) group and storage in Ringer solution after one and two weeks.

Discussing our results with all these variations in the different mechanical tests applied for reinforcement PEEK types used in this study; CFP group and GFP group have been interpreted as diversified outcomes related to manufacturing process with difference in mechanical behavior between GFP and CFP which were improved by Janda et al., who found the reason (Janda et al. 1990). The elevated rates of both reinforcement PEEK samples used in our study came with the similarity of most researches conducted the effect of applying carbon fibers and glass fibers on the hardness of PEEK materialit was stated stated that  the hardness of carbon/glass fiber far exceeded that of the pure PEEK matrix (Gao et al, 2022). The hardness and modulus of the PEEK matrix were enhanced by the fiber due to its excellent mechanical properties, and the reinforcement was improved with the increased mass fraction of fiber.

This rigidity of reinforced PEEK groups with low elasticity attitude explained normally as the fibers of carbon and glass increased the stiffness toward the bending strength application.

In further researches, they found that the molding temperature, molding method for PEEK blends manufacturing process will affect the mechanical properties and different material ratios and deformation temperatures have a significant impact on the shape force of the sample during measurement process (Zhang et al, 2021; Hager et al, 2015; Aguirre et al, 2016; Zalaznik et al, 2016). GFP showed high stress values than PPE which agreed with most of the research that preceded it; a good fiber combination in the PEEK matrix provides the best compressive strength and higher ductility. These finding were also revealed by who stated that the application of glass fibers to the PEEK matrix improved their load bearing capacity (Sfondrini et al, 2014). Other improvement came in agreement to our findings who found that the reinforcement PEEK groups with CFP showed the same compression stress values with pure PEEK groups with no statistical differences and our results came in agreed with the finding of Han et al. with no statistical difference between the carbon fiber PEEK and pure PEEK materials in compressive strength (Han et al, 2019). Another research by the study by (Han et al, 2019) who observed that reinforced carbon fiber had significantly better strengths than pure PEEK with no statistical difference  between them. This may be explained as that both of PPE and CFP groups had treated with the same range of processing temperature expressed as uniform mechanical pattern behavior. On the same issue a study came in agreements with our thesis result; they found that application of more than 5% of glass fiber to the PEEK matrix had showed a decrease in tensile strength by 38% with increase of fracture stress (Georgiou et al, 2015). It was stated that glass and zirconium nanoparticles improve the ability of PEEK to less surface deformity behavior (Peng and Li, 2014; Gladson et al., 2019). The low tensile stress values registered in our study was also showed by a study (Cao et al, 2009) which stated that glass/basalt reinforced sheets under elevated temperatures up to 200◦C showed 40% tensile strength reduction (Cao et al, 2009). Other finding reported 80% strength reduction of woven GF composites at 300 ◦C (Gibson et al, 2010).

The tensile length of the material fracture and ultimate tensile stress became smaller as the glass fiber mass fraction increased. These findings agreed with the ones explained by (Hou et al, 2018; Quan, & Yan, 2001). It was explained that the bending stress linearly decreased as the displacement increases until break occurred and the bending stress dropped rapidly to zero with sharp fracture morphology although they registered higher values of bending modulus due to the use of high cooling rate mechanism (Xu et al, 2017) . CFP is a brittle fracture at the stress concentration when applying bending strength rather than net PEEK (Dai et al, 2022; He et al, 2017). On the other hand our findings disagreed with the results of (Han et al, 2019) who explained that reinforced carbon fiber had significantly better strengths than the pure PEEK in the bending tests ($P < 0.05$).

These differences related to the printing manufacturing technique applied by Han; he stated that the use of post heat treatment (tempering) resulted in excellent bending properties.

Our findings for both pure peek types used in this research agreed with the ones improved by (Negahdari et al, 2018) evaluated three nano hybrid composite resins including PEEK showed slight reduction in hardness after 7 days.Another explanation found that the chemical bonding of filler and matrix resultantly leached out from the surface decreasing the hardness (Ferracane and Marker, 1992).

Song et al., registered higher values in micro hardness test for both carbon fiber PEEK and glass fiber PEEK in comparison to the net PEEK type (Song et al, 2017). Ji et al., also discussed the reinforcement fiber PEEK type with hardness test they improved that mean hardness, and mean load of the PEEK are smaller than those of the GF/PEEK. PEEK-reinforced 30 wt% glass fiber can get higher stiffness, hardness, and resistance to deformation and also lead to poor uniform property (Ji et al, 2015). Mohamed et al., also discussed the effect of adding glass fibers on the micro hardness on thermosetting polymers as the values of the reinforcing glass fibers with 20% volume fraction lead to a remarkable increase in the micro-hardness values, Vickers micro-hardness properties of these samples were evaluated by carrying out hardness testing (Mohamed et al, 2018).

The mean of pressure stress on day 1 in the Ringer solution ranged from 102 mm for CFP group and 165.3 mm for PPC group. In other words, the CFP had the lowest pressure stress while the PPC group had the highest-pressure stress after one day in the solution. The mean of pressure stress after one week in the Ringer solution ranged from 152.2 mm for PPE group to 188.6 mm for GFP group. In other words, the PPE had the lowest pressure stress while the GFP group had the highest-pressure stress after one week in the solution. The mean of pressure stress after three weeks in the Ringer solution ranged from 137.3 mm for PPE group to 185.8 mm for CFP group. In other words, the PPE had the lowest pressure stress while the CFP group had the highest-pressure stress after three weeks in the solution.

The mean of tensile stress on day 1 in the Ringer solution ranged from 49.7 mm for CFP group and 90.7 mm for PPE group. In other words, the CFP had the lowest tensile stress while the PPE group had the highest tensile stress after one day in the solution. The mean of tensile stress after one week in the Ringer solution ranged from 57.5 mm for CFP group and 85.8 mm for PPE group. Like after one day in the solution, the CFP had the lowest tensile stress while the PPE group had the highest tensile stress after one week in the solution. The mean of tensile stress after three weeks in the Ringer solution ranged from 62.0 mm for GFP group and 92.0 mm for PPC group. In other words, the GFP had the lowest tensile stress while the PPC group had the highest tensile stress after three weeks in the solution. The mean of bending stress on day 1 in the Ringer solution ranged from 143.9 mm for PPC group and 169.7 mm for CFP group. In other words, the CFP had the highest bending stress while the PPC group had the lowest bending stress after one day in the solution. The means of bending stress after one week in the Ringer solution ranged from 136.3 mm for CFP group to 181.4 mm for PPC group. In other words, the CFP had the lowest pressure stress while the PPC group had the highest- bending stress after one week in the solution. The means of bending stress after three weeks in the Ringer solution ranged from 130.7 mm for CFP group to 195.2 mm for PPE group. In other words, the CFP had the lowest bending stress while the PPE group had the highest- bending stress after three weeks in the solution. The mean of hardness value on day 1 in the Ringer solution ranged from 40.7 for PPC group and 54.8 for CFP. In other words, the PPC had the lowest hardness value while the CFP group had the highest hardness value after one day in the solution. The mean of hardness value on week 1 in the Ringer solution ranged from 27.7 for PPC group and 50.4 for CFP. In other words, the PPC had the lowest hardness value while the CFP group had the highest hardness value after one week in the solution. The mean of hardness value on week 3 in the Ringer solution ranged from 27.6 for PPE group and 42.7 for GFP. In other words, the PPE had the lowest hardness value while the GFP group had the highest hardness value after one week in the solution.

From all these findings related to different storage time intervals of 1 day, 1 week, 3 weeks and for the different PEEK types used; PPE, PPC, CFP, GFP who submitted to four different mechanical tests; compression, tensile, bending, and hardness tests we figured that each PEEK group showed his own specific behavior in ringer solution storing. PPC and GFP revealed better compression strength with minimum shape deformation in comparison to CFP who showed better values than PPE group,so to improve the compression strength for PPE, PPC, CFP, GFP it's better to keep the samples for one week in ringer solution to raise their compression strength rates. PPE and PPC groups exposed better tensile strength in comparison to CFP and GFP groups but still it's better not to store any of the PEEK samples in ringer solution at any time interval because it showed a clear decline in tensile strength rates. The bending values for PPE have been improved after 1 day and 3 weeks storing in ringer solution but for CFP and GFP 1day storing in ringer solution was improving the bending strength rates than 1 week and 3 weeks' time period.

For hardness test PPE, PPC, CFP GFP all revealed higher values after 1 day storing in ringer solution. There is no doubt that the presence of fibers reinforcement inside the PEEK matrix has improved their various mechanical properties (Ji eta l, 2015; Abbood etal, 2021; Dai et al, 2022; Bazli & Abolfazli, 2020). But still the numbers of researches related to the effect of salty solution storage on different PEEK types are limited. Overall, studies of polymer solubility in molten inorganic salt hydrates are still in their infancy. However, most of the available studies, only determined the reduced mechanical properties of resins after aging in artificial saliva, alcohol, and distilled water. From the results of our study we found that the storage in ringer solution at 1 day has improved the hardness strength rates for all of PPE, PPC, CFP, and GFP groups while the storage in ringer solution for 1 week has upgrade the compression stress values. For tensile stress and bending stress rates there was different behavior pattern for each type of PEEK groups at different storage time intervals.

Pure PEEK types of PPE and PPC groups proved their tensile and bending stress strength after 1 week and 3 weeks of storing in ringer solution and that can be explained as molecular interactions between the salty particles and surface molecules of PEEK samples as ionic concentration and this increased the values of both mechanical tests applied (Rajagopalan et al, 2021; Hirschey et al, 2018; Yu et al, 2021).

PPC and PPE types with better elasticity behavior in comparison to CFP and GFP rigid types so the immersion in salty solution exceeded the rigidity to small levels. Liebermann et al., found that the highest impact on solubility was found for storage in physiological saliva, followed by NaCl, and distilled water so PEEK can be recommended for long-term restorations, he also found that non- or low-filled materials like PEEK showed the lowest hardness values, whereas the highest values were found for materials with high percentages of filler particle and these results came in agreement with the findings of our work that CFP and GFP types showed higher hardness rates and bending rates than PPE and PPC types (Liebermann et al, 2016). Pure PEEK-brands with identical molecular weight are easily to be differentiated in their plastic deformation behavior. Hence, increasing crystallinity favors an energy impact of the E-module, resulting from decreasing resilience.The higher the rate of densely packed crystals the harder and brittle the component will become. PPC has shown better improvement in tensile and bending stress values after storing in salt solution than PPE type; that can be illustrated as compression powder particles are evenly distributed in the matrix on the one hand, while on the other, they performed the intended bonding with it and this including molecular bonding with salt ions (Janda, 1990). These are in contrast to the findings of some authors who have already emphasized the outstanding stability of PEEK. They pointed out its trait to remain unaffected even after being exposed to 1000h in contact with 250 ◦C hot water steam .Accordingly, no measurable influence on the results might be expected to appear after incubation at 37 ◦C. Thus, this unexpected effect has to be object of examination in the future (Stober et al, 1984; Searle & Pfeiffer, 1985; Boinard et al, 2000).

Increasing salt concentration will also induce shrinkage of the polymer molecule in solution, with the shielding of electrostatic fields around charged functionalities by counter-ions resulting in chain coiling. On other hand CaCl2 were found as suitable salts to be stabilized inside the polymer matrix. The wetting behavior of the salt determines the salt distribution inside the polymer matrix and this improves the performance and cyclic lifetime inside polymers matrix (Aarts et al, 2023).

The limitations of this study have been summarized as this in vitro study cannot demonstrate many factors related to the oral environment, especially the evaluation scheme. Also hardness samples were made according to rectangular package dimensions of 30×10×2.5 mm according to ISO 20795-1 standard in proportion to the original dimensions of PEEK samples came from factory in China, and for compression mechanical test the measurements evaluated the pressure strength in accordance to the PEEK samples dimensions rather than pressure modulus which need a different samples measures.

Future studies are suggested to:

1. Studying the effect of PEEK different tempreture cycling degrees and its effect on various PEEK meachanical and biological properties.
2. Studying numbers and different lengths of fibers used for reinforecemnt PEEK material.
3. Evaluating the effect of PEEK different tempreture storage on its meachanical behavior.
4. Studying different PEEK features with another storage solutions like; pure water, tea, or acidic fluid.
5. Discussing the possibility of using different types of PEEK material within the same dental treatment like using two types of PEEK material in fixed or removable prosthodontics appliance and evaluating each mechanical bearing inside patient mouth.

# 6. CONCLUSION

1. The four measuring parameters (hardness, tensile stress, pressure stress and bending stress) varied across the four study groups (PPE, PPC, CFP and GFP) and across the four storage conditions/ times (dry condition and one day, one week and three weeks in Ringer solution) within the same group.
2. Each material has strength in certain parameters and weakness in others. For example, the CFP group has high levels of hardness (54.79) VHN and pressure stress (185.8) MPa while it has low levels of bending stress and tensile stress. In contrast, PPE group has high levels of bending and tensile stress while low levels of hardness and pressure stress.
3. The CFP group had the highest hardness value while both PPE and PPC had the lowest hardness value among the four-study group. The mean hardness value was the highest after one day in the solution across the four groups then reduced over time.
4. In general, PPE and PPC groups had higher tensile stress compared to that in CFP and GFP group particularly after storing in the solution. Across all the four study groups (PPE, PPC, CFP and GFP), the mean of tensile stress was significantly higher in the dry condition than that in the Ringer solution ($P<0.05$). Within each group, the tensile stress reduced after storing in the solution.
5. Across all the four study groups (PPE, PPC, CFP and GFP), the mean of pressure stress was significantly highest after one week in the Ringer solution.

6. Within each group, the pressure stress increased significantly between day 1 and week 1 of storage in the solution.

7. No clear pattern existed for bending stress across the four groups. The bending stress of GFP group was the lowest at dry condition compared to the three Ringer solution measurements. In contrast, the bending stress of CFP group was the lowest after three weeks in the solution compared to the three measurements. Within CFP and GFP groups, the pressure stress increased significantly between dry condition and after one day in the solution. The bending stress mean was the highest after one day for CFP group, after one week for PPC group and after three weeks for PPE group in the solution compared to other three measurements/storage conditions.

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# EKLER

## EK-1. ETİK BİLDİRİM VE İNTİHAL BEYAN FORMU

SAĞLIK BİLİMLERİ ENSTİTÜSÜ
Graduate School of Health Sciences

**ETİK BİLDİRİM VE İNTİHAL BEYAN FORMU[1]**

| | |
|---|---|
| Öğrencinin Adı ve Soyadı | Reem Abdulrahim Nsaif |
| Öğrencinin Numarası | |
| Ana Bilim Dalı | **Protetik Diş Tedavisi** |
| Öğrencinin Kayıtlı Olduğu Program Türü | Doktora |

**Yukarıda bilgileri verilen tezin intihal tespit yazılımıyla (Turnitin) yapılan tarama sonucunda elde edilen benzerlik oranları aşağıdaki gibidir. Beyan edilen bilgilerin doğru olduğunu, aksi hâlde doğacak hukuki sorumlulukları kabul ve beyan ederiz.**

| Bölümler | Benzerlik Oranı | Maksimum Benzerlik Oranları |
|---|---|---|
| **I.** Giriş | %13 | % 15 |
| **II.** Genel Bilgiler | %32 | % 35 |
| **III.** Materyal ve Metod | %17 | % 35 |
| **IV.** Bulgular | %11 | % 15 |
| **V.** Tartışma | %17 | % 20 |

***Not:*** *Yedi kelimeye kadar benzerlikler ile Başlık, Kaynakça, İçindekiler, Teşekkür, Dizin ve Ekler kısımları tarama dışı bırakılabilir. Yukarıdaki azami benzerlik oranları yanında tek bir kaynaktan olan benzerlik oranlarının %5'den büyük olmaması gerekir.*

# EK-2. ETİK KURUL ONAY FORMU

T.C
ATATÜRK ÜNİVERSİTESİ
DİŞ HEKİMLİĞİ FAKÜLTESİ DEKANLIĞI
ETİK KURULU

**Oturum Tarihi: 23/03/2022**
**Oturum Sayısı: 05/ 2022**

**KARAR**

| | |
|---|---|
| **SORUMLU ARAŞTIRMACI** | ***Prof. Dr. Funda BAYINDIR*** <br> ***Arş. Gör. Dt. Reem NSAIF*** |
| **Araştırmanın Açık Adı** | **Karbon Fiber ve Cam Fiber ile Güçlendirilmiş Hibrit (Poli Eter Eterketon) Kompozitlerinin Mekanik Ve Fiziksel Özeliklerinin İncelenmesi -[*Improve The Mechanical and physical Properties of Hybrid (Poly Ether Etherketone) Composits Reinforced With Carbon Fibers and glass fibers*]** |
| **Karar No** | **36.** |
| **Alınan Karar** | Prof. Dr. Funda BAYINDIR danışmanlığında Arş. Gör. Dt. Reem NSAIF'in birlikte hazırladıkları **"Karbon Fiber ve Cam Fiber ile Güçlendirilmiş Hibrit (Poli Eter Eterketon) Kompozitlerinin Mekanik Ve Fiziksel Özeliklerinin İncelenmesi** -[*Improve The Mechanical and physical Properties of Hybrid (Poly Ether Etherketone) Composits Reinforced With Carbon Fibers and glass fibers*]" başlıklı Doktora tezi Sağlık Bakanlığı tarafından yayımlanan 19 Ağustos 2011 tarih ve 28030 sayılı "Klinik Araştırmalar Hakkındaki Yönetmelik" hükümlerine bağlı kalınarak yapılmak şartıyla; kabul edilmesinde bilimsel ve etik açıdan sakınca olmadığına oybirliği ile karar verildi. |