

Improving the Mechanical and Physical Properties of Hybrid (Polyether Ether Keton) Composites

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Abstract

Statement of Problem: Polyether ether ketone material is considered as an important thermoplastic material due to its properties. To obtain a high value stress and tougher hybrid PEEK during different dental applications. **Purpose:** In this study, it was aimed to improve some mechanical and physical properties of dental (polyether ether ketone) PEEK. Different mechanical properties will be measured at different time intervals after incubation in the Ringer solution. **Materials and Methods:** A total of 80 samples were produced ($n = 20$) used for each test. 2 groups of different PEEK materials were used; extrusion PEEK and compression PEEK (PPE, PPC). 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, tensile, and hardness tests will be applied. ANOVA and post-hoc tests were used for statistical analysis. **Results:** The results of mechanical strength tests including compression, tensile, bending and hardness tests on PEEK (PPE, PPC) 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. 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 (P -value > 0.05). Mechanical tests showed different results between different PEEK samples at different time storage intervals. **Conclusion:** The measuring parameters (pressure stress, bending stress, tensile stress and hardness value) varied across the study groups (PPE, PPC) and across the four storage conditions/times (dry condition and one day, one week and three weeks in Ringer solution) within the same group.

Keywords

PEEK, Mechanical Properties, PEEK Storage

1. Introduction

Over the last three decades, researchers from various expertise such as physics, engineering, chemistry, biology and medical sciences, have been 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 the applied forces [1] [2] [3].

(PEEK) (Polyether 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 [4] [5].

PEEK has appeared as one of the most important materials and has employment in demanding engineering components such as chemical process industries and medical materials [6] [7]. The persistent demand of developed manufactures for even greater performing materials, has however led to the emergence of PEEK in order to evolve the mechanical, thermal and electrical features of the matrix [8] [9] [10] [11].

PEEK has a high temperature thermoplastic material that is supplied in a range of viscosities according to the processing method [12] (Table 1). It is processed by using different conventional techniques including machining injection molding, extrusion, compression molding, and film and fiber production [13].

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

Table 1. Material used in the study and technical characteristics.

Abbreviation	Brand name	Composition	Manufacturing processes*	Specific gravity
I/PE	Victrex Corp PEEK INCHR New Material China	pure PEEK R10 sheet	Extrusion	1.29 g/cm
II/PC	Light copra PEEK Germany	pure PEEK disk	Compression molding	1.22 g/cm

*Working temperature 350°C - 420°C.

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 [14]. 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 [15]. This process is typically used in the production of extremely thick sections of 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. 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 [16] [17] [18].

There are two 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 discussed in this study is the effect of the storage environment on different PEEK samples; (PPE) and (PPC), mechanical tests showed different results between different PEEK samples at different time storage intervals.

The aim of this study was to examine the effect of compressive strength, bending strength, tensile strength, and micro hardness strength on two different processing types of pure PEEK at different ringer solution storage time intervals. The obtained data were collected and analyzed statistically and different results were evaluated and compared.

2. Materials and Methods

2.1. Materials

Two different types of PEEK were used in this study. The materials (samples) to be examined are given in (Table 1). The number of samples used in this study is based on the parameters explained by Kibuacha, 2021 to determine sample size and how to calculate the sample size with yield smaller margins of error and be more representative.

A total of 80 samples were produced ($n = 20$) for each test. Then these two groups were divided into four subgroups ($n = 10$) according to sample storage status. They were tested at dry storage as a control group for each subgroup and after incubation in Ringer's solution (Polifleks® Poliforma, Polifarma İlaç Sanayi Ve Tic., Türkiye) at different time intervals; 1 day, 1 week, and 3 weeks at 37°C (Table 2).

2.2. Methodology

1) Samples preparation:

Table 2. 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
			Total No. = 80

The tested samples were cut according to ISO standards that were specific for each mechanical 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).

For compression strength test; the PEEK samples were cut 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). For the bending test, the PEEK samples were cut according to ISO 178: 2010 (Plastics—Determination of Flexural Properties) with specimen dimensions of 80 × 10 × 4 mm (ISO 178: 2010 Plastics). The dog-bone shape tensile testing specimens 90 × 5 × 4 mm were cut according to ISO 527-2 standard (2012 Plastics—Determination of Tensile Properties). For microhardness 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) (Figures 1(a)-(d)).

2) Samples Storage:

The different PEEK samples; PPE, PPC 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 temperature (25°C) before testing (Figure 2).

3) Mechanical test measurements:

Compression test was taken by electromechanical universal testing system (INSTRON, 5982, Bluehill® 2 Software, UK), with 10 KN load cell and constant displacement of 1.3 mm/min static testing systems, three-point bending test taken by electromechanical universal testing system (INSTRON, 5982, Bluehill® 2 Software, UK), with fixture length = 50 and at speed of 1 mm/min. Each test took about 6 min to reach a permanent deformity phase. Ductility also was valued within the recorded diagram. For tensile test measurements the PEEK samples

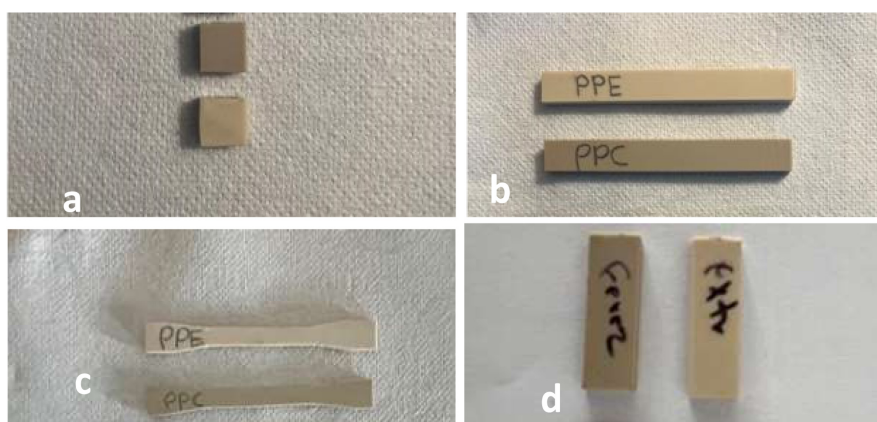


Figure 1. PEEK samples for (a) Pressure test, (b) Bending test, (c) Tensile test, (d) Hardness test.

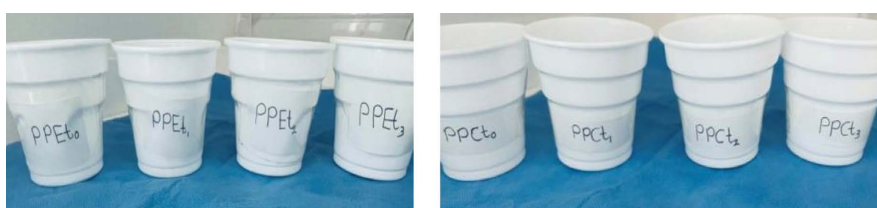


Figure 2. PEEK group samples at different storage time.

were examined with a device (INSTRON, 5982, Bluehill® 2 Software, UK) at speed of 1 mm/min. Sample elongation was also valued within the recorded diagram (**Figures 3(a)-(c)**). The Vickers Hardness Tester (Universal Motion, Inc. No. 208, R Cube, S. No. 116/5/1, Warje Highway, Near Atul Nagar, Pune-411052, Maharashtra) with 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° .

Within each group, the pressure stress increased significantly after one day and one week of storage in the solution, the mean of pressure stress value within the 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**). According to repeated measure ANOVA, there was no significant ($P\text{-value} > 0.05$) difference in pressure stress within PPE group across different storage conditions/times (**Table 3**).

The mean pressure stress value within the 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. 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 (**Figure 4**). According to repeated measure ANOVA and post-hoc tests, there were five significant

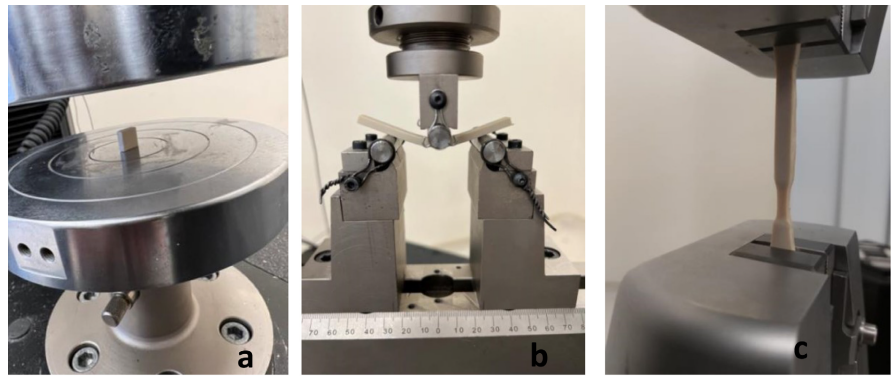


Figure 3. PEEK samples on electromechanical universal testing system (a) Pressure test; (b) Bending test; (c) Tensile test.

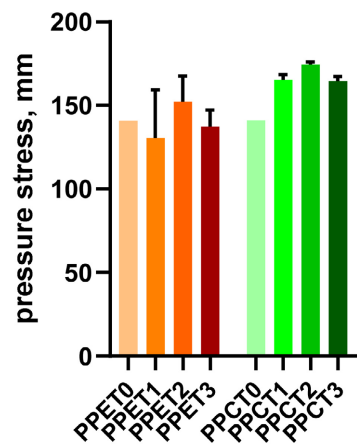


Figure 4. The difference in the pressure stress value across four different storages for PPE, PPC samples.

Table 3. 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	<u>130.6</u>	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	<u>152.2</u>	-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

*ns = non 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). The mean 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 (Figure 5). In other words, bending stress within the 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 the 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.

ANOVA and post-hoc tests 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 values between dry condition from one side and one day and one week in the solution from the other side (Table 5).

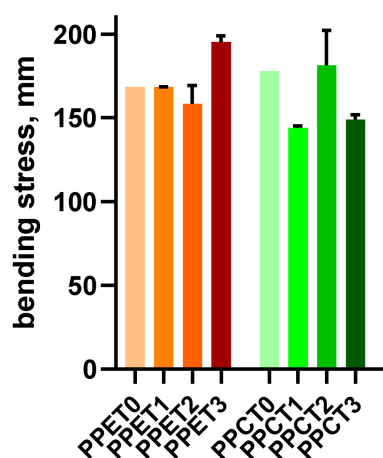


Figure 5. The difference in the bending stress value across four different storages for PPE, PPC samples.

Table 4. 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

*Significant (P -value < 0.05) according to post hoc test (Turkey's multiple comparisons). ns = non significant.

The mean of bending stress value within the PPC group ranged from 143.9 mm after one day to 181.4 mm after one week in the Ringer solution (**Figure 5**). 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 6**).

The mean of tensile stress value within the Pure Peek Extrusion (PPE) group ranged from 85.8 mm after one week in the Ringer solution to 92.0 mm in the control (dry) condition (**Figure 6**). The tensile stress reduced gradually after storing in the Ringer solution. According to repeated measure ANOVA and post-hoc tests, there were two significant ($P > 0.05$) differences in tensile stress

Table 5. The differences in the bending stress 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	158.3	10.16	6.394	No	ns	0.5309
PPET0 vs. PPET3	168.5	195.2	-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

*Significant (P -value < 0.05) according to post hoc test (Turkey's multiple comparisons). ns = non significant.

Table 6. The difference in the bending stress value across different storage conditions/times within the same group (pure Peek compression).

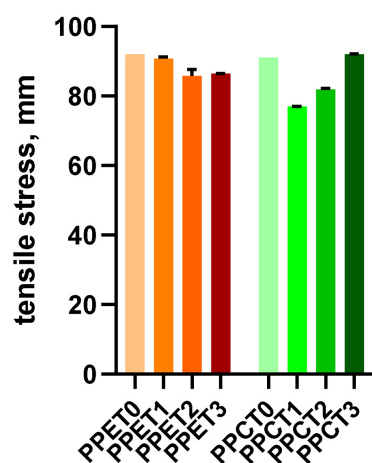
repeated measure ANOVA P-value	0.0908						
Tukey's multiple comparisons test	Mean 1	Mean 2	Mean Diff.	SE of diff.	Significant?	Summary	Adjusted P-Value
PPCT0 vs. PPCT1	178	143.9	34.07	0.744	Yes	*	<0.0001
PPCT0 vs. PPCT2	178	181.4	-3.401	12.06	No	ns	0.99
PPCT0 vs. PPCT3	178	149	28.91	1.631	Yes	*	0.0082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

*Significant (P -value < 0.05) according to post hoc test (Turkey's multiple comparisons). ns = non significant.

Table 7. 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 (Turkey's multiple comparisons). ns = non significant.

**Figure 6.** The difference in the tensile stress value across four different storage for PPE, PPC samples.

within PPE group across different storage conditions/times (**Table 7**).

The mean of tensile stress value within the Pure Peek Compression (PPC) group ranged from 76.98 mm after one day in the Ringer solution to 92.0 mm after three weeks in the solution (**Figure 6**). The tensile stress initially (after one day) reduced after storing in the Ringer solution but increased after a 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.

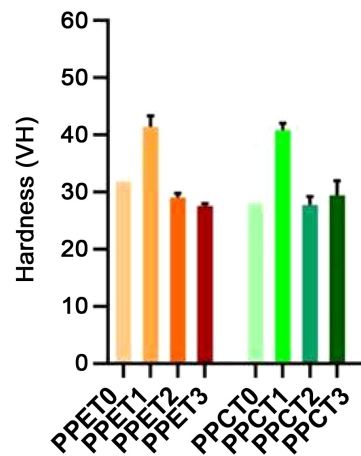
The tensile stress means after one day and one week in the Ringer solution were significantly lower than that in control (dry) condition and after three weeks in the solution (**Table 8**).

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 (**Figure 7**). The storage condition after one day in the Ringer solution had the

Table 8. 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).

**Figure 7.** The difference in the hardness value across four different storages for PPE, PPC samples.

highest hardness value while after three weeks in the same solution had the lowest hardness value within the PPE group. According to repeated measures ANOVA, there was a 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 the 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 the Ringer solution within the same group (PPE) (Table 9).

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 solu-

tion (Figure 7). The storage condition after one day in the Ringer solution had the highest hardness value while after one week in the same solution had the lowest hardness value within the 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 tests (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 10).

3. Discussion

There are two hypotheses in this study with its 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 discussed in this study is the effect of the storage environment on different PEEK samples; Mechanical tests showed different results between different PEEK samples at different time storage intervals.

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 [19] [20]. Compressive, bending, Tensile and hardness forces were carried out in laboratory settings 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

Table 9. 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	<u>41.4</u>	-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	<u>27.56</u>	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 (Turkey's multiple comparisons).

Table 10. The difference in the hardness 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 (Turkey's multiple comparisons).

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. For the bending stress there was a significant difference in the dry environment (control group) between PPE and PPC group. PPC registered a 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. 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 PPE group and PPC group were found with almost equal shape of bending in the middle of the sample without fracture after being subjected to bending test at different storage environments/time.

According to the tensile strength test results there was 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 the 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.

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 a number of researches [21] [22]; due to the difference in crystallinity 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 and 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 [23].

These variations also were explained by 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 [24].

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 [25] [26].

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 [27] [28] [29]. On the other hand, processing, structure, and properties of PEEK semicrystalline was investigated and showed that under normal processing conditions for high performance composites, the mechanical properties may not be strongly affected by different levels of crystallinity [30]. 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 [17] [31] [32] [33].

Findings related to different storage time intervals of 1 day, 1 week, 3 weeks and for the different PEEK types used; PPE, PPC 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, that's 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 [34] [35] [36].

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 $30 \times 10 \times 2.5$ 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 measure. Future studies are suggested to study the effect of PEEK different temperature cycling degrees and its effect on various PEEK mechanical and biological properties and to evaluate

the effect of PEEK different temperature storage on its mechanical behavior. Also, it's suggested for future researchers to use different types of PEEK material within the same dental treatment like using two types of PEEK material in fixed or removable prosthodontics appliances and evaluating each mechanical bearing inside the patient mouth.

4. Conclusions

1) The four measuring parameters (hardness, tensile stress, pressure stress and bending stress) varied across the study groups (PPE, PPC) 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, PPE group has high levels of bending and tensile stress while low levels of hardness and pressure stress.

3) Across all the study groups (PPE, PPC) the mean of tensile stress was significantly higher in the dry condition than that in the Ringer solution (P-value < 0.05). Within each group, the tensile stress is reduced after storing in the solution.

4) (PPE, PPC) showed the mean of pressure stress was significantly highest after one week in the Ringer solution.

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

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

References

- [1] Mushtaq, A., Sakshi, Ch., Naila, P. and Radhika, G. (2021) PEEK: A Futuristic Dental Material in Pediatric Dentistry. *Pediatric Dental Journal*, *7*, 72-74. https://doi.org/10.14744/JPD.2021.04_37
- [2] Harting, R., Marius, B., Thomas, B., Regina, S.P. and Svea, P. (2017) Functionalization of Polyetheretherketone for Application in Dentistry and Orthopedics. *Bionanomaterials*, *18*, Article ID: 20170003. <https://doi.org/10.1515/bnm-2017-0003>
- [3] Pourkhalil, H. and Maleki, D. (2022) Fracture Resistance of Polyetheretherketone, Ni Cr, and Fiberglass Postcore Systems: An *in Vitro* Study. *Journal of Dental Research*, *19*, 20. <https://doi.org/10.4103/1735-3327.338783>
- [4] Papageorgiou, D., Cristina, V., Robert, J.Y., Ian, A.K., *et al.* (2019) Hybrid Poly(Ether Ether Ketone) Composites Reinforced with a Combination of Carbon

- Fibres and Graphene Nanoplatelets. *Composites Science and Technology*, **175**, 60-68. <https://doi.org/10.1016/j.compscitech.2019.03.006>
- [5] Elawadly, T., Iman, A.W., Amr, El. and Reham, B. (2017) Can PEEK Be an Implant Material? Evaluation of Surface Topography and Wettability of Filled versus Un-filled PEEK with Different Surface Roughness. *Journal of Oral Implantology*, **43**, 456-461. <https://doi.org/10.1563/aaid-joi-D-17-00144>
- [6] Liebermann, A., Timea, W., Patrick, R.S., Harry, S., Patrick, L.Ö., Malgorzata, R., Bogna, S. and Dipl, I. (2016) Physicomechanical Characterization of Polyetheretherketone and Current Esthetic Dental CAD/CAM Polymers after Aging in Different Storage Media. *Journal of Prosthetic Dentistry*, **115**, 321-328. <https://doi.org/10.1016/j.prosdent.2015.09.004>
- [7] Nobre, M., Carlos, M., Ricardo, A., António, S. and Nuno, S.J. (2020) Hybrid Polyetheretherketone (PEEK)-Acrylic Resin Prostheses and the All-on-4 Concept: A Full-Arch Implant-Supported Fixed Solution with 3 Years of Follow-Up. *Journal of Clinical Medicine*, **9**, Article No. 2187. <https://doi.org/10.3390/jcm9072187>
- [8] Altstaedt, V., Philipp, W. and Jan, S. (2003) Rheological, Mechanical and Tribological Properties of Carbon-Nanofiber Reinforced Poly(Ether Ether Ketone) Composites. *Polímeros: Ciência e Tecnologia*, **13**, 218-222. <https://doi.org/10.1590/S0104-14282003000400005>
- [9] Ma, R. and Tang, T.T. (2014) Current Strategies to Improve the Bioactivity of PEEK. *International Journal of Molecular Sciences*, **15**, 5426-5445. <https://doi.org/10.3390/ijms15045426>
- [10] Lai, Y.H., Kuo, M.C. and Hu, J.C. (2007) On the PEEK Composites Reinforced by Surface-Modified Nano-Silica. *Materials Science and Engineering*, **458**, 158-169. <https://doi.org/10.1016/j.msea.2007.01.085>
- [11] Wang, L., D'Alpino, P., Lopes, L. and Pereira, J. (2003) Mechanical Properties of Dental Restorative Materials: Relative Contribution of Laboratory Tests. *Journal of Applied Oral Science*, **11**, 162-167. <https://doi.org/10.1590/S1678-77572003000300002>
- [12] Muhsin, S.A. (2016) Evaluation of Poly(Etheretherketone) for Use as Innovative Material in the Fabrication of a Removable Partial Denture Framework. Doctoral Dissertation, University of Sheffield, Sheffield.
- [13] Roeder, R.K. (2019) PEEK Biomaterials Handbook. 12: Bioactive Polyaryletherketone Composites. Elsevier Inc., Amsterdam, 203-227. <https://doi.org/10.1016/B978-0-12-812524-3.00012-0>
- [14] Hoskins, T.J. (2015) The Mechanical and Tribological Properties of PEEK Gears. Doctoral Dissertation, University of Birmingham, Birmingham.
- [15] Kurtz, S.M. (2012) An Overview of PEEK Biomaterials. In: *PEEK Biomaterials Handbook*, Elsevier Inc., Amsterdam, 1-7. <https://doi.org/10.1016/B978-1-4377-4463-7.10001-6>
- [16] Wang, Y., Müller, W., Rumjahn, A. and Schwitalla, A. (2020) Parameters Influencing the Outcome of Additive Manufacturing of Tiny Medical Devices Based on PEEK. *Materials*, **13**, Article No. 466. <https://doi.org/10.3390/ma13020466>
- [17] Zhang, T., Liu, H., Zhang, D. and Chen, K. (2019) Mechanical and Wear Properties of Polyetheretherketone Composites Filled with Basalt Fibres. *Science and Engineering of Composite Materials*, **26**, 317-326. <https://doi.org/10.1515/secm-2019-0016>
- [18] Perng, L.H., Tsai, C.J. and Ling, Y.C. (1999) Mechanism and Kinetic Modelling of PEEK Pyrolysis by TG/MS. *Polymer*, **40**, 7321-7329.

- [https://doi.org/10.1016/S0032-3861\(99\)00006-3](https://doi.org/10.1016/S0032-3861(99)00006-3)
- [19] Wilson, N.H.F. (1990) The Evaluation of Materials: Relationships between Laboratory Investigations and Clinical Studies. *The Open Dentistry Journal*, **15**, 149-155.
- [20] Gonzalez, D., Rusinek, A., Jankowiak, T. and Arias, A. (2015) Mechanical Impact Behavior of Polyether-Ether-Ketone (PEEK). *Composite Structures*, **124**, 88-99. <https://doi.org/10.1016/j.compstruct.2014.12.061>
- [21] Ji, S., Sun, C., Zhao, J.I. and Liang, F. (2015) Comparison and Analysis on Mechanical Property and Machinability about Polyetheretherketone and Carbon-Fibers Reinforced Polyetheretherketone. *Materials*, **8**, 4118-4130. <https://doi.org/10.3390/ma8074118>
- [22] Yang, C., Tian, X., Li, D., Cao, Y., Zhao, F. and Shi, C. (2017) Influence of Thermal Processing Conditions in 3D Printing on the Crystallinity and Mechanical Properties of PEEK Material. *Journal of Materials Processing Technology*, **248**, 1-7. <https://doi.org/10.1016/j.jmatprotec.2017.04.027>
- [23] Siddiq, A. and Kennedy, A.R. (2020) Compression Molding and Injection over Molding of Porous PEEK Components. *Journal of the Mechanical Behavior of Biomedical Materials*, **111**, Article ID: 103996. <https://doi.org/10.1016/j.jmbbm.2020.103996>
- [24] Rokaya, D., Srimaneepong, V., Sapkot, J., Siraleartmukul, J.Q. and Iriwongrungson, V. (2018) Polymeric Materials and Films in Dentistry: An Overview. *Journal of Advanced Research*, **14**, 25-34. <https://doi.org/10.1016/j.jare.2018.05.001>
- [25] Kurtz, S.M. (2011) Synthesis and Processing of PEEK for Surgical Implants. In: Kurtz, S.M., Ed., *PEEK Biomaterials Handbook*, Elsevier, Amsterdam, 9-22. <https://doi.org/10.1016/B978-1-4377-4463-7.10002-8>
- [26] Yu, W., Zhang, H., Yang, S., Zhang, J., Wang, H., *et al.* (2020) Enhanced Bioactivity and Osteogenic Property of Carbon Fiber Reinforced Polyetheretherketone Composites Modified with Amino Groups. *Colloids and Surfaces B*, **193**, Article ID: 111098. <https://doi.org/10.1016/j.colsurfb.2020.111098>
- [27] Li, T., Song, Z., Yang, X. and Du, J. (2023) Influence of Processing Parameters on the Mechanical Properties of Peek Plates by Hot Compression Molding. *Materials*, **16**, Article No. 36. <https://doi.org/10.3390/ma16010036>
- [28] Offringa, A.R. (2010) In New Thermoplastic Composite Design Concepts and Their Automated Manufacture. *Aeronautics*, **58**, 45-49.
- [29] Rinaldi, M., Cecchini, F., Pigliaru, L., Ghidini, T., Lumaca, F. and Nanni, F. (2021) Additive Manufacturing of Polyether Ether Ketone (PEEK) for Space Applications: A Nanosat Polymeric Structure. *Polymers*, **13**, Article No. 11. <https://doi.org/10.3390/polym13010011>
- [30] Seferis, J.C. (1986) Polyetheretherketone (PEEK): Processing-Structure and Properties Studies for a Matrix in High Performance Composites. *Polymer Composites*, **7**, 158-169. <https://doi.org/10.1002/pc.750070305>
- [31] Hager, M.D., Bode, S., Weber, C. and Schubert, U.S. (2015) Shape Memory Polymers: Past, Present and Future Developments. *Progress in Polymer Science*, **49**, 3-33. <https://doi.org/10.1016/j.progpolymsci.2015.04.002>
- [32] Aguirre, C., Franco, F., Samsudin, H., Fang, X. and Auras, R. (2016) Poly(Lactic Acid)-Mass Production, Processing, Industrial Applications, and End of Life. *Advanced Drug Delivery Reviews*, **107**, 333-66. <https://doi.org/10.1016/j.addr.2016.03.010>
- [33] Zalaznik, M., Kalin, M. and Novak, S. (2016) Influence of the Processing Temperature on the Tribological and Mechanical Properties of Polyether-Ether-Ketone

(PEEK) Polymer. *Tribology International*, **94**, 92-97.

<https://doi.org/10.1016/j.triboint.2015.08.016>

- [34] Rajagopal, S., Priya, V., Sameul, Ch., Sarassri and Abraham, P. (2021) Peek Material and Its Technical Consideration in Dental Application—A Systematic Review. *International Journal of Recent Advances in Multidisciplinary Research*, **2**, 2582-7839.
- [35] Hirschey, J., Gluesenkamp, K.R., Mallow, A. and Graham, S. (2018) In Review of Inorganic Salt Hydrates with Phase Change Temperature in Range of 5°C to 60°C and Material Cost Comparison with Common Waxes. *Conference: 5th International High Performance Buildings Conference*, Purdue, 9-12 July 2018, 1-10.
- [36] Yu, K., Liu, Y. and Yang, Y. (2021) Review on Form-Stable Inorganic Hydrated Salt Phase Change Materials: Preparation, Characterization and Effect on the Thermo-physical Properties. *Applied Energy*, **292**, Article ID: 116845.
<https://doi.org/10.1016/j.apenergy.2021.116845>