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Comparison of microhardness of three interim restorative materials: PEEK, PMMA and indirect composite

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Objectives Interim restorations play a critical role in success of restorative treatments. However, they need to preserve their integrity in the oral environment. Microhardness is an important factor in preservation of the integrity of restorations. This study aimed to compare the microhardness of three interim restorative materials namely polyether ether ketone (PEEK), an indirect composite, and polymethyl methacrylate (PMMA).

Methods This in vitro, experimental study evaluated 10 disc-shaped specimens, measuring 15 mm in diameter and 1 mm in thickness, fabricated from PEEK, an indirect composite, and PMMA. The microhardness of the specimens was measured at three points of each specimen using the Vickers' hardness test before and after water storage for 30 days. Data were analyzed using repeated measure ANOVA, one-way ANOVA and Tukey's test.

Results Indirect composite showed maximum microhardness, which was significantly higher than that of the other two materials (P=0.001). However, no significant difference was noted in microhardness of PEEK and PMMA (P=0.33). The microhardness of the materials did not significantly change after 30 days of water storage (P=0.06).

Conclusion The microhardness of indirect composite was higher than that of PEEK and PMMA. Also, 30 days of water storage had no significant effect on microhardness of the materials.

Keywords Hardness; Polyetheretherketone; Polymethyl Methacrylate; Composite resins

Introduction

Efficient interim restorations play an important role in success of fixed partial dentures. Interim restorations are used aiming to protect the dental pulp, preserve the position of the prepared teeth, prevent over-eruption of the opposing teeth and tipping or movement of the adjacent teeth, and preserve the gingival health. Moreover, interim restorations play an important role as a pattern for the fabrication of final restorations.[1] They also play a critical role in assessment of occlusion and maxilla-mandibular relationship.[2]

Long-term use of interim restorations is imperative in some certain cases, as for maintaining or establishing proper occlusal vertical dimension in patients under dental implant treatment.[3] Also, interim fixed restorations are imperative for soft tissue management and provision of adequate gingival contour for definitive restorations.[4, 5] In such cases, interim restorations should be used in the oral cavity for a relatively long period of time. Thus, they need to be fabricated from materials with high mechanical strength. Mechanical strength is a critical factor for preservation of the integrity of interim restorations.

Microhardness is a clinically important physical property of restorations. It refers to the resistance of a material against indentation by a microhardness tester that applies load for a specific period of time to create indentations in a material. Microhardness is among the main parameters that can predict the clinical success of restorative materials.[6]

Surface degradation and fracture are among the main reasons for replacement of interim restorations, which is time-consuming and costly for both patients and clinicians. Factors affecting the microhardness include the quality and technique of polishing, chemical composition of materials, and long-term effects of water and other storage media.[6] Surface hardness can indicate the density of a material, and it is assumed that a denser material has higher resistance to wear and surface degradation.[7] Thus, type of material used for the fabrication of interim restorations is important, and should be carefully selected.

Polymethyl methacrylate (PMMA) is commonly used for the fabrication of interim restorations. Due to optimal properties such as polishability, easy application, easily reparability, low irritancy, good flexibility, and low cost, PMMA is commonly used for the fabrication of denture base, special trays, and interim restorations.[8-10] However, it also has inherent shortcomings such as high polymerization shrinkage, high brittleness, poor mechanical properties, and low antibacterial activity that limit its application.[11, 12] In an attempt to optimize the properties of acrylic resins, composite resins were introduced as polymer restorative materials reinforced with silica particles.[13] Composite resins have higher mechanical properties than acrylic resins, and have lower coefficient of thermal expansion and subsequently lower dimensional changes during setting reactions. Moreover, composite resins have higher wear resistance and hardness, and consequently superior clinical performance compared with PMMA.[14]

Polyether ether ketone (PEEK) is a semi-crystalline engineered plastic material with excellent mechanical and thermal properties. PEEK has several advantages such as light weight, non-toxicity, bioinert behavior, high corrosion resistance, and a modulus of elasticity close to that of bone.[15-17] At present, it is commonly used in fixed and removable dentures, orthodontics, and dental implant therapy as abutment.[16, 17] Karaokutan et al, and Balkenhol et al. showed that composite resins had superior mechanical properties compared with methacrylate resins.[18, 19] Also, several studies have indicated that water sorption is an important factor affecting the properties of materials such as their hardness.[20, 21] However, studies comparing the microhardness of PEEK, PMMA, and composite resins are limited. Thus, this study aimed to compare the microhardness of PEEK, PMMA, and an indirect composite resin used for the fabrication of interim restorations. The null hypothesis was that the microhardness of the abovementioned three interim restorative materials would not be significantly different, and water storage would have no significant effect on their microhardness.

Methods and Materials

This in vitro, experimental study was approved by the ethics committee of dental faculty of Islamic Azad University (IR.IAU.DENTAL.REC.1399.042). The following three materials were evaluated:

PEEK (BiOHPP-98×20 mm AG; A2-Bredent CO, Senden Brec A.M., Germany), PMMA (Ceramill TEMP-A2-71L20 mm, Amann Girrbach, Germany), and an indirect composite resin (Crea-lign Paste-Dentin A2-Bredent CO-GmbH, Senden, Germany). Table 1 presents the properties of the abovementioned three materials.

Table 1- Properties of PEEK, PMMA and indirect composite

Material	Abbrev.	Type	Manufacturer	Composition	Ref	Lot	Filler%
Biohpp A2	PEEK	Blank	Bredent, Senden, Germany	Ceramic fillers-partially crystalline	54002121	463034	20
Ceramill A2	PMMA	Blank	GmbH- Germany-Amann Girrbach	Polymethyl methacrylate cross link	760323	50915	-
Cera.lign A2	Comp	Paste	Bredent, Senden, Germany	Nano filler ceramic –bis-GMA	CLFNDA30	N173893	60

Thirty disc-shaped specimens (n=10) were fabricated with 15 mm diameter and 1 mm thickness according to ISO4049 as follows:

PEEK and PMMA blanks were obtained and milled in the desired dimensions using a computer-aided design/computer-aided manufacturing (CAD/CAM) machine (Ceramill MAP 400-AMANNGIRBACH, Germany). They were then polished with 1000 and 2000-grit abrasive papers. Impurities and dust were eliminated by using air spray, and the dimensions of the specimens were then measured by a digital caliper with 0.01 mm accuracy (MAX150–Hogetex Co, Netherland) according to ISO standard such that the diameter of specimens was not smaller than 14.8 mm.[22]

To prepare the composite specimens, a ring-shaped mold with the desired dimensions was first made from silicon.[6] Ethyl polyvinyl was used as a separator to enhance the separation of material from the mold. Its formulation is such that it does not interfere with the polymerization of composite resin. A glass slab was placed over the mold to prevent void formation. Next, the composite resin was light-cured using a curing unit (LABO Light LV-III-120W-GC, Japan) with 440-480 nm wave length and 1500 mW/cm^2 light intensity for 40 s.[6] Excess material was removed by polishing with 1000 and 2000-grit abrasive papers. Impurities and dust were removed by air spray. The specimen dimensions were then measured by a digital caliper with 0.01 mm accuracy.[22]

A square-shaped diamond indenter was used for the Vickers' hardness test. This test is similar to the Brinell and Knoop tests with the difference that a 136° conical, diamond indenter applies a certain load to a material and creates a square-shaped indentation. The diameters of the square-shaped indentation are measured, and the Vickers hardness number (VHN) is calculated using the formula below:

VHN=1.854F/ d2

Where F is the load applied in kilograms and d2 is the diameter of the square-shaped indentation in square-millimeters (mm^2).[6]

After specimen preparation and also after their storage, the microhardness of specimens was measured at three points using a digital Vickers hardness tester (VTP6060, Bareiss Co, Germany) with 50 g load applied for 15 s. The mean of the three values was calculated and reported as the final VHN.[6] After measuring the baseline microhardness of specimens, they were incubated in pure distilled water (Pilton shop Co., Iran) at 37°C for 30 days (246M/53/108; Memmert Co., USA). The container and holder of specimens were designed such that the specimens were positioned perpendicular and had a minimum of 3 mm distance from each other according to ISO4049. Also, a minimum of 10 mm3 of water was considered for immersion of each specimen. The container was then sealed. The volume of water was checked daily.[22] After 30 days of incubation, the specimens were rinsed and placed in a desiccator (Simax Co., Czech) for 30 days. The desiccator contained silica and had been recently charged and dried in an oven for 6 h at 130°C.[22] The desiccator door was coated with silicon oil to ensure an airtight seal. It was then connected to a vacuum

pump to create a vacuum with no humidity and reach a constant weight. The secondary microhardness of specimens was then measured. Also the microhardness of specimens was compared before and after water storage.

Statistical analysis: Data were submitted to repeated measures ANOVA and one-way ANOVA for among-group comparison, Tukey's test for between-group comparison, and repeated measures ANOVA to compare the materials before and after immersion using SPSS version 25. All statistical analyses were performed at a significance level of 0.05.

Results

Repeated measures ANOVA and one-way ANOVA showed a significant difference in baseline microhardness of the three materials (P<0.001). The secondary microhardness of the three materials was also significantly different (P=0.018). Moreover, significant differences were noted between the primary and secondary microhardness values (P=0.015).

The Tukey's test showed that the microhardness of indirect composite was significantly higher than that of PMMA and PEEK (before and after 30 days of storage) (P=0.001). The difference between PMMA and PEEK was not significant (P=0.33, Table 2).

Table 2- Intergroup (pairwise) comparison of Vickers hardness number by the Tukey's test

Group Comparison	Mean difference	P value
Before immersion		
PEEK vs PMMA	-1.5333	0.572
PEEK vs COMP*	-21.333	< 0.001
PMMA vs COMP	-19.8000	< 0.001
After immersion		
PEEK vs PMMA	-6.13000	0.331
PEEK vs COMP	-12.49000	0.013
PMMA vs COMP	-6.36000	0.305

Also, assessment of the change in microhardness after immersion of the materials by using repeated measures ANOVA revealed that there was no significant difference in any material (P=0.06, Table 3 and Diagram 1). However, numerically, the microhardness of PEEK and PMMA increased after immersion while the microhardness of composite decreased.

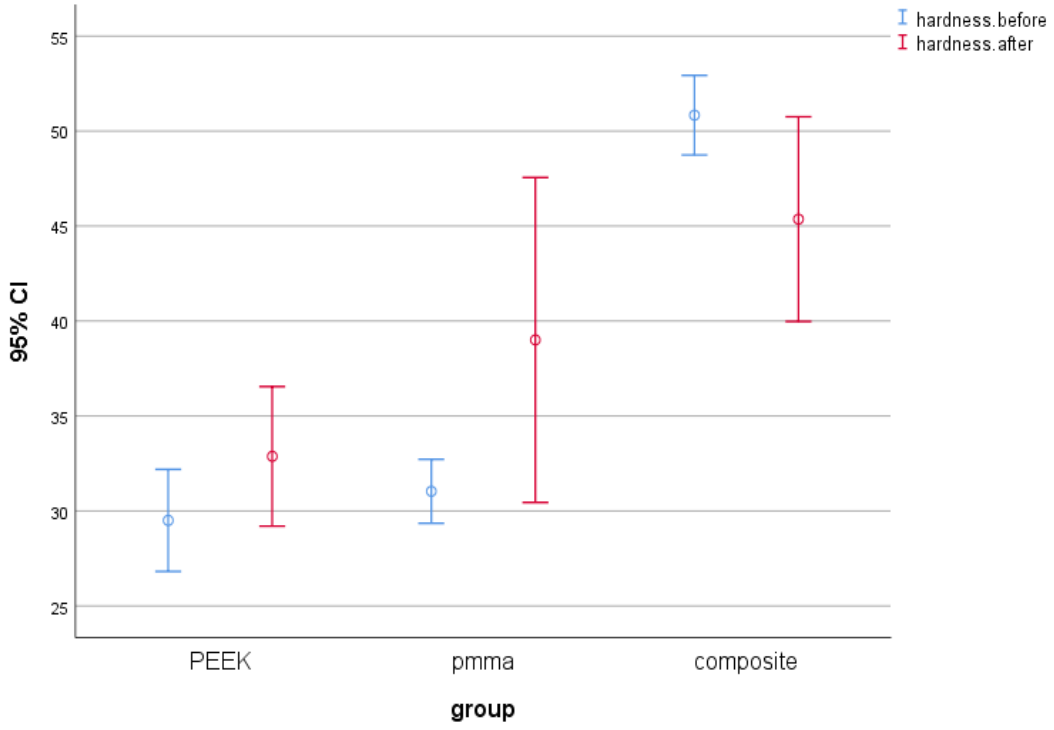

Diagram 1- Comparison of microhardness of the three interim restorative materials before and after immersion

Table 3- Mean (± standard deviation) Vickers micro hardness number (VHN) of interim materials before and after immersion (Kg/mm^2)

Materials	Before immersion	After immersion
PEEK	29.5033±7.19	32.8733±9.83
PMMA	31.0367±4.51	39.0033±22.90
Composite	50.8367±5.60	45.3633±14.43

Discussion

This study evaluated the microhardness (VHN) of PEEK, an indirect composite and PMMA, which are used for interim restorations. The results showed that the microhardness of composite was significantly higher than that of PEEK and PMMA. Thus, part of the null hypothesis was rejected. Also, the results showed that 30 days of water storage had no

significant effect on microhardness of the materials. Thus, the second part of the null hypothesis was accepted.

In this study, Ceraline composite was used, which has a bis-GMA matrix with 60% ceramic nano-filler particles. In general, indirect composites have superior mechanical properties such as compressive strength, tensile strength, hardness, and wear resistance due to a number of reasons such as their increased filler percentage, mode of polymerization, and fabrication by the CAD/CAM technology. CAD/CAM composite resins have easier fabrication and repair process, and have lower fragility and risk of fracture. Also, they are compatible with different types of milling machines and have a higher marginal quality.[23]

Several factors affect the hardness of composite resins such as the organic matrix composition, type, amount and size of filler particles, degree of conversion[24], surface contamination (for example with bonding agent), the degree and intensity of curing, duration of immersion and storage in saliva/water, and polishing of composite surface.[2] Maximum microhardness noted in indirect composite rather than PMMA in our study can be due to the difference in their monomer composition. The resin matrix of indirect composite resins contains multifunctional monomers such as bis-GMA and TEGDMA, which form cross-links with other monomers while auto-polymerized conventional methacrylates contain mono-functional monomers with low molecular weight, which result in lower microhardness and lower wear resistance.[25-27] Moreover, size of filler, and weight, volume and ratio of filler/matrix affect the microhardness. Higher filler percentage increases the microhardness.[28] In the present study, PEEK showed a lower microhardness than the other two materials, which was in agreement with previous findings.[23, 29, 30] PEEK is a thermoplastic, semi-crystalline material devoid of residual monomer in its matrix.[29] PEEK blocks used in this study contained 20% filler content; due to its matrix properties and low filler percentage, PEEK showed lower microhardness than the indirect composite. Its microhardness was almost similar to that of PMMA. Addition of compounds such as carbon fiber or glass to the short chains would improve the mechanical properties and decrease the water sorption and solubility of PEEK.[31]

Alamoush et al.[32] showed that feldspathic porcelain blocks (502 kg/mm^2) had maximum microhardness followed by Vita Enamic (203 kg/mm^2), composite resin (73-121 kg/mm^2) and PEEK blocks (25-27 kg/mm^2). The microhardness of enamel and dentin was reported to be 313 and 62 kg/mm^2, respectively. In general, the mechanical properties such as hardness of dental materials should be preferably in the range of mechanical properties of bone, enamel and dentin .[23, 32]

PEEK has optimal mechanical properties, and its tensile and flexural strength are comparable to those of bone and enamel. Although some studies have reported its acceptable application in fixed and removable dentures[16, 17], many others have reported its low modulus of elasticity.[23, 29, 30] Thus, it is imperative to use PEEK reinforced with other compounds (fillers or TiO2 particles) or ceramics with a small percentage of PEEK filler for permanent prosthetic restorations. The hardness of PEEK is almost equal to that of PMMA, and it also possesses some other optimal properties such as minimal water sorption and solubility. Thus, it is suitable for the fabrication of long-term interim restorations.[30] The results of the present study showed that microhardness of specimens did not significantly change after 30 days of water storage (P=0.06). However, numerically, the VHN of indirect composite decreased by 5 kg/mm^2 while the VHN of PMMA averagely increased by 9 kg/mm^2 and that of PEEK increased by 1.5 kg/mm^2.

In the present study, specimens of all three materials were fabricated by using the CAD/CAM technology. Thus, they had less porosities and higher integrity, resulting in higher mechanical properties. Since the baseline microhardness of PEEK was lower than that of indirect composite and PMMA, its microhardness after immersion was still lower than that of the other two materials. Savabi et al. reported that the microhardness of composite resins used for the fabrication of interim restorations did not significantly change after 7 days of immersion in artificial saliva.[27] However, Negahdari et al.[6] evaluated three nanohybrid composite resins (Kalore, G-aenial, Aura) after 60 days of immersion in water and showed higher solubility of G-aenial, due to its lower filler percentage. G-aenial showed slight reduction in hardness after 7 days; however, its hardness did not significantly change after 60 days. The hardness of the other two composite resins increased over time. Mckinney and Wu[33] reported that composite resins stored in water for 2 weeks experienced an increase in hardness. Controversy in the results of studies can be attributed to the effect of different factors such as the filler percentage, solubility, water sorption, polymerization percentage, type of resin matrix, and duration of water storage. The hardness value is indirectly correlated with the degree of polymerization. Higher degree of polymerization increases the hardness.[34]

Water sorption of the resin matrix that occurs during water storage decreases the mechanical properties. Water sorption causes swelling of the cross-linked polymer, and decreases the frictional forces between the polymer chains. Thus, when the matrix is water-saturated, it becomes stabilized.[35] Also, water sorption at the resin-filler interface degrades the chemical bonding of filler and matrix. Resultantly, the filler particles are leached out from the surface, decreasing the hardness.[36] Moreover, factors such as void formation during material application can cause water sorption and degrade the composite material.[37] The microhardness of PMMA and PEEK did not significantly change after 30 days of water storage; however, they numerically experienced a slight increase in microhardness; this increase was greater for PMMA. In general, polymerization conditions including temperature and curing environment (water/air) affect the oxidation of monomer and microhardness of auto-polymerized PMMA. According to Lee et al.[38] the release rate of residual methyl methacrylate monomer directly

affects the reduction of mechanical properties such as microhardness. In other words, the lower the residual monomer, the higher the mechanical properties would be. Water storage during polymerization eliminates oxygen and its effects, and improves microhardness. Lee et al. [38] added that curing temperature also plays a role in improvement of microhardness, and curing of resin in high-temperature water decreases the amount of residual monomer by 80%, and increases the microhardness by over 50%.

It appears that longer water storage of specimens in the present study might have resulted in significant results. In this study, microhardness was measured by the Vickers hardness test due to its simplicity, popularity and reliability.[23] Factors such as the composition of saliva, oral temperature, and functional loads can significantly affect the microhardness of restorative materials. However, due to some limitations, thermocycling and cyclic loading were not performed in this study. On the other hand, longer water storage would better simulate the clinical setting for intraoral restorations. Thus, the abovementioned factors should be addressed in future studies.

Conclusion

Within the limitations of this study, it may be concluded that indirect composite had a higher microhardness than PEEK and PMMA. Water storage for 30 days did not significantly change the microhardness of interim restorative materials evaluated in this study.

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Conflict of Interest

No Conflict of Interest Declared■

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