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A Comparative Evaluation of Surface Properties and Water Sorption of Polyetheretherketone (PEEK) With Different Polymers in Different Aging Media

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ABSTRACT

Context: Different pH affects surface properties and water sorption of prosthesis. Polyetheretherketone (PEEK) is an emerging material and very scarce data is available for effect of pH on it.

Aims: This invitro study was conducted to evaluate and compare the surface properties and water sorption of Polyetheretherketone (PEEK) with Polymethylmethacrylate (PMMA) and Indirect Resin Composite (IRC) in different aging media.

Settings and Design: Invitro analytical study

Methods and Material: 90 disks of CAD/CAM milled PEEK, PMMA and laboratory processed IRC were fabricated (n=30). Specimens from each group were aged in aging media with different pH twice a day for 10 minutes. Initial and final surface roughness, gloss and water sorption of each specimen was measured after 180 days of aging and difference was calculated.

Statistical analysis used: Multivariate CRD ANOVA was performed for pair wise comparison. Level of significance was set at $p \le 0.05$.

Results: With respect to groups, there was statistically significant difference between the groups for change in surface roughness and water sorption of all groups. With respect to aging media there was statistically significant difference in water sorption. Statistical analysis of subgroups showed significant difference for water sorption.

Conclusions: Within the limitation of the present study it can be concluded that PEEK exhibited better properties in terms of change in surface roughness and water sorption than PMMA and IRC test specimens. pH of aging media has no effect on change in surface roughness and loss of gloss.

Keywords: PEEK, PMMA, IRC, Surface roughness, Gloss, Water sorption, pH, Aging media and CAD/CAM **INTRODUCTION**

Polymers is a group of material commonly used in applications such as artificial teeth, tooth restorations, cements, orthodontic space maintainers and elastics, crown and bridge facings, obturators for cleft palates, inlay patterns, implants, impressions, dies, temporary crowns, endodontic fillings and athletic mouth protectors.¹ Polymers used commonly for dental prosthesis fabrication include polymethylmethacrylate and BISGMA based resins. Over the years there have been constant development and modification in these materials to suit the restorative/prosthetic needs required for their desired use.

Computer-Aided Design (CAD)/Computer Aided Manufacturing (CAM) technologies have been applied in the fabrication of PMMA prosthesis. CAD/CAM PMMA shows superiority in terms of many properties, including its hardness, flexural strength, flexural modulus, and impact strength.² Polymerization shrinkage of the resin is eliminated and the fit of prosthesis is greater than conventionally fabricated one.

Composite materials are increasingly being used for the restoration, due to excellent esthetic and better bonding, but they are associated with demerits like risk of microleakage and secondary caries, need for meticulous oral hygiene maintenance, lower fracture toughness, and polymerization shrinkage effects.³

Based on these demerits newer laboratory processed indirect resin composites (IRC) were introduced which have twice the filler content than that of organic matrix which improves mechanical properties and reduces polymerization shrinkage. Indirect resin composite also has added advantage of better esthetics and is relatively less prone to postoperative sensitivity. It is dimensionally stable and has much reduced microleakage.

PEEK is a synthetic, tooth colored polymeric material, characterized by high mass-based stability, strong resistance against temperature loads, chemical and physical and radiological stress and corrosion.

Surface properties play an indisputably central role in the clinical success of prosthesis. The roughness of the prosthetic surfaces is important, since the adhesion of microorganisms to a surface is a prerequisite for the colonization of that surface.⁴

Prosthetic materials used in oral rehabilitation are subjected to complex and changing humid and wet environment, which is physiologically oral characterized by natural saliva and its component. Potential deleterious effects on surfaces of prosthesis may arise from pH changes due to cariogenic biofilms in the oral ecology, diet intake, and different enzymes. This can lead to not only esthetic problems in terms of water absorption and discoloration but also to the mechanical weakening of the materials, surface changes, decreased fracture resistance, increased wear etc.⁵ Properties of the material that are mainly affected by pH of food are surface roughness, gloss, water sorption etc.

Limited research and studies have been done on surface properties and water sorption properties of restorations fabricated using PEEK and newer acrylic and composite materials. Therefore, the present study was taken up to evaluate and compare change in surface properties and water sorption of polyetheretherketone (PEEK) with CAD/ CAM polymethylmethacrylate (PMMA) and indirect resin composites (IRC) after aging in media with different pH.

MATERIAL & METHODOLOGY:

30 specimens were prepared from each test material (PEEK, PMMA and IRC) with diameter of 20mm and thickness of 3mm. (Figure 1)





The surface roughness (Ra, μ m) for all specimens was analyzed with the help of profilometer. (Figure 2A) A reading was obtained by the needle passing across 0.8mm length at 0.5mm/s, to the nearest 0.01 μ m. Surface roughness was measured at 2 positions, radially across each specimen, and a final Ra average were then calculated for that specimen. Gloss measurements for all specimens were obtained using glossmeter (Figure 2B). The gloss was measured at 60degree angle from normal to the surface. Measurements were repeated 3 times for each specimen and the mean values were calculated. Initial weight of each specimen was measured using electronic weighing machine (analytical balance) (Figure 2C). Specimens were placed in balance pan and thereafter transparent glass door of the electronic weighing machine was closed. Measurements were repeated 3 times for each specimen and the mean values were calculated.



Figure 2: (A) Surface profilometer, (B) Gloss meter, (C) Electronic weighing machine

After initial evaluation, the specimens were stored in media with different pH. 10 specimens of each group were immersed in Cola, turmeric and tea solutions respectively. (Figure 3 A-C)



Figure 3: Specimens immersed in aging media (A) Cola solution- acidic media, (B) Turmeric solution- basic media, (C) Tea solution- neutral media

Each specimen was immersed for 10 minutes twice a day in aging solutions for 180 days. The specimens were stored in artificial saliva for the remaining time in between immersion cycles. After removing the specimen from aging media after a total of 360 cycles of immersion, the samples were cleaned in ultrasonic cleaner and wiped with dry clean cloth and air dried at room temperature. After immersion into aging media with different pH, final surface roughness, gloss and weight of all specimens was measured for the same surface as done prior to immersion. Difference between initial and final surface roughness, gloss and weight was calculated.

RESULTS:

The present study was conducted to evaluate and compare surface properties and water sorption of

polyetheretherketone (PEEK) with different polymers in different aging media. The study was divided into 3 groups, Group I [Polyetheretherketone (PEEK)], Group II [Polymethylmethacrylate (PMMA)] and Group III [Indirect resin composite (IRC)]. Specimens from each group were aged in acidic, basic and neutral media and each group was further sub-divided into 3 subgroups; Group IA, Group IB and Group IC (PEEK specimens aged in acidic, basic and neutral media respectively); Group IIA, Group IIB and Group IIC (PMMA specimens aged in acidic, basic and neutral media respectively); Group IIIA, Group IIIB and Group IIIC (IRC specimens aged in acidic, basic and neutral media respectively). Data was tabulated and subjected to statistical analysis. CRD ANOVA of mean values was performed. The level of significance was set at $p \le 0.05$.

On subjecting the specimens of Group I to aging the change in surface roughness observed was 0.019±0.009um in acidic media, 0.021±0.005um in basic media and 0.028±0.027µm in neutral media respectively; Loss of gloss observed was 33.50±5.602GU in acidic media, 36.40±6.432GU in basic media and 34.40±7.456GU in neutral media respectively; Water sorption observed was 0.025±0.015mg in acidic media, 0.025±0.024mg in basic media and 0.022±0.028mg in neutral media respectively.

On subjecting the specimens of Group II to aging the change in surface roughness observed was 0.032±0.022µm in acidic media, 0.025±0.012 µm in basic media and 0.048±0.026µm in neutral media respectively; Loss of gloss observed was 38.20±7.554GU in acidic media, 34.70±12.365GU in basic media and 36.30±6.617GU in neutral media respectively; Water sorption observed was 0.055±0.043mg in acidic media, 0.101±0.058mg in basic media and 0.114±0.041mg in neutral media respectively.

On subjecting the specimens of Group III to aging the change in surface roughness observed was $0.116\pm0.049\mu$ m in acidic media, $0.128\pm0.050\mu$ m in

basic media and 0.153±0.063µm in neutral media respectively; Loss of gloss observed was 31.90±7.880GU in acidic media, 31.30±8.641GU in basic media and 33.80±6.373GU in neutral media respectively; Water sorption observed was 0.079±0.039mg in acidic media, 0.189±0.092mg in basic media and 0.093±0.043mg in neutral media respectively.

On subjecting the values to CRD ANOVA, with respect to groups, there was statistically significant difference between the groups for change in surface roughness and water sorption of all groups ($p \le 0.05$). However, there was no statistically significant difference in loss of gloss of all groups. (p > 0.05)

With respect to aging media there was statistically significant difference in water sorption ($p \le 0.05$), whereas there was no statistically significant difference for change in surface roughness and loss of gloss. (p>0.05)

For two-way interaction between groups and aging media there was significant difference for water sorption ($p \le 0.05$). (Table 1) However there was no statistically significant difference observed for change in surface roughness and loss of gloss. (p > 0.05)

Groups	N	Subset for alpha =0.05				
		1	2	3	4	5
IC	10	0.02				
IA	10	0.03	0.03			
IB	10	0.03	0.03			
IIA	10		0.06	0.06		
IIIA	10			0.08	0.08	
IIIC	10			0.09	0.09	
IIB	10				0.10	
IIC	10				0.11	
IIIB	10					0.19

Table 1: Summary of pair wise statistical analysis of water sorption for two-way interaction between groupsand aging media

* Level of Significance $p \leq 0.05$.

DISCUSSION:

With the recent increased interest in prosthesis fabricated using computer aided technology, a range of CAD/CAM polymethylmethacrylate (PMMA) based polymers has been introduced in recent few years. CAD/CAM PMMA based polymers that are utilized for the fabrication of the prosthesis provides a superior fit and strength in comparison to the conventionally prosthesis.⁶ processed Polyetheretherketone (PEEK) is the latest inventory of dentistry and is claimed to have better properties in parallel with existing materials. Polyetheretherketone is resistant to hydrolysis, non-toxic and has one of the best biocompatibility. Proper contour, smoothness and high gloss can produce the desired appearance of natural tooth structure desired by patients. A rougher surface texture can lead to decreased gloss and increased discoloration of the material surface which can affect the restorations esthetics. Therefore, it is of paramount importance to obtain smooth and glossy surfaces. Water sorption of material indicates adsorption and absorption of water when in service. The water absorbed by the resin can act as plasticizer and causes softening, discoloration and loss of mechanical properties of resin such as hardness, transverse strength and fatigue limit.

In the present study the evaluation of surface properties and water sorption was performed on 30 discs of diameter 20mm and 3mm in thickness of each PEEK, PMMA and IRC respectively. These dimensions were used as it is compatible size for performing tests for surface roughness, gloss and water sorption. This was similar to the dimensions used by Rukiye Durkan et al⁷ in their study.

Specimens of each group were aged in acidic, basic and neutral pH media for 10mins twice a day. Anja Liebermann et al⁵ found that daily 20 mins of immersion in any media is needed for specimens to exhibit change in surface properties. Cola solution, turmeric solution and tea solution were selected as Acidic, Basic and Neutral media respectively because these are commonly consumed by Indian population.⁸

Aging procedure was carried for 180 days (6 months) to find out the long-term effects of different pH media on surface roughness, gloss and water sorption of specimens. After removing the specimen from aging media after a total of 360 cycles of aging, the samples were cleaned in ultrasonic cleaner and wiped with dry

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clean cloth and air dried at room temperature. Contact profilometer was used in this study to measure change in surface roughness because the stylus is in continuous contact with the surface and this method is not sensitive to surface reflectance or color unlike used in optical profilometer. Ra is a mathematical average of the absolute values of the measured profile height of surface irregularities, measured from a mean line within the preset length of specimen. A reading was obtained by needle passing across 0.8mm length at 0.5mm/sec, to the nearest 0.01 µm. The gloss was measured at 60-degree incidence angle from normal to the surface. Measurement angle refers to the angle between the incident light and the perpendicular. The glossmeter provides a quantifiable way of measuring gloss intensity ensuring consistency of measurement by defining the precise illumination and viewing conditions. For measuring water sorption, the measurement of initial and final weight of each specimen was measured using electronic weighing machine (analytical balance). The use of a mechanically vented balance safety enclosure, which has uniquely designed acrylic air-foils, allows a smooth turbulence-free airflow that prevents balance fluctuation and the measure of mass down to 1 µg without fluctuations or loss of product. The specimen's weight was measured at room temperature to prevent natural convection from forming air currents inside the enclosure from causing an error in reading. Calculation of water sorption was done using formula [WS=m2-m1] where, WS= Water sorption; m1= Specimens weight before placing in media (Initial weight) and m2= Specimens weight after placing in media (Final weight). The result of the present study indicates that there was statistically significant difference between all groups for change in surface roughness. In the present study PEEK test specimens exhibited least change in surface roughness followed by PMMA and IRC test specimens respectively. There was statistically significant difference between PEEK & PMMA, PEEK & IRC and PMMA & IRC test specimens respectively. This property of PEEK test specimen for least change in surface roughness can be attributed to chemical homogeneities in its structure. In the present study Acidic and Basic media exhibited equal change in surface roughness followed by Neutral media. There was no statistically significant difference between aging media for change in surface roughness. In the

current study, it was found that the surface roughness significantly increased when soaked in neutral pH media. This could be attributed to the fact that the pH values can affect the degradation rates of the polymers where the breaking strength of the polymer was found to depend markedly on the pH and was found to be highest at neutral ph. Result of the present study indicates that there was no statistically significant difference for loss of gloss for all groups. In the present study IRC test specimens exhibited least loss of gloss followed by PEEK and lastly PMMA test specimens respectively. The results showed that all materials had high initial gloss. However, in all test materials gloss decreased after aging. Gloss is influenced by a variety of factors, such as the filler size distribution, the index of refraction of the fillers present in the plastic, and the viscosity of the resin matrix components and effect of extrinsic factors on them. In the present study Basic media exhibited least loss of gloss followed by Acidic media and neutral media respectively. However, there was no statistically significant difference observed between different media with respect to loss of gloss. The loss of gloss in different media occurred due to pH changes which caused microscopic and macroscopic roughness that causes incident light to be diffusely reflected, thus reducing gloss of the surface of test specimens. In the present study PEEK test specimens exhibited least water sorption followed by PMMA and IRC test specimens respectively. There was statistically significant difference for water sorption between PEEK & PMMA. PEEK & IRC and PMMA & IRC test specimens. PEEK specimens showed least water sorption due to more homogenous matrix in the structure thus leading to less water absorption. The results of the present study support the concept that water absorption generally increases with higher amounts of resin matrix and a lower amount of filler particles. In the present study Acidic media exhibited least water sorption followed by Neutral and Basic media respectively. There was statistically significant difference between Acidic & Basic media, Acidic & Neutral media and Basic & Neutral media. This can be attributed to the influence of basic medium by its interaction with OH- ions during the hydrolysis process. Thus, an accelerated degradation is expected in a medium with an excess of hydroxyl ions.

The current study signifies that surface properties and water sorption of dental material are two properties that are of paramount importance but commonly neglected by dental clinicians before making choice of dental material for prosthesis. PEEK is undeniable the new emerging material whose properties are superior, favorable and unmatched to currently established polymers in dentistry i.e. PMMA and IRC. With respect to this context PEEK can be used in fabrication of wide variety of prosthesis including dental post structures, removable or fixed framework of prosthesis, wide application in implantology for fabrication of implants, abutments and implant supported prosthesis.^{9,10}

CONCLUSION:

Within the limitation of the present study following conclusions can be drawn:

- Polymers tested in the study i.e. PEEK, PMMA and IRC exhibited changes in surface properties and water sorption when aged in media with different pH.
- PEEK test specimens exhibited least change in surface roughness followed by PMMA and IRC test specimens. Aging of test specimens in Acidic and Basic media exhibited equal change in surface roughness followed by Neutral media.
- Initial gloss of IRC was better than PEEK and PMMA. PEEK, PMMA and IRC test specimens did not exhibit significant change for loss of gloss. Aging of test specimens in Basic media exhibited least loss of gloss followed by Acidic and Neutral media
- PEEK test specimen exhibited least water sorption followed by PMMA and IRC test specimens. Aging of test specimens in Acidic media exhibited least water sorption followed by Neutral and Basic media.
- All the changes in surface roughness, loss of gloss and water sorption by material and media were well within acceptable limit.
- PEEK exhibited better properties in terms of change in surface roughness and water sorption than PMMA and IRC test specimens.
- Aging media with different pH had no effect on change in surface roughness and loss of gloss. Water sorption was found to be considerably affected by aging media with different pH.

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- PEEK is a supreme material and has become extremely widespread in dental field. This suggests its wide application in fabrication of FDP and CPD framework, in implantology for fabrication of implant abutments, implant supported prosthesis framework etc.
- Further research is advised on evaluating properties of Polyetheretherketone (PEEK) invivo.

CLINICAL IMPLICATIONS OF THE STUDY:

PEEK material provides excellent surface properties and low water sorption rate and hence will lead to low biofilm formation rate which in turn aids in less plaque retention on the surface of the prosthesis. Combined with other favorable physical and mechanical properties it can be used in fabricating frameworks for fixed and removable prosthesis and in implantology for fabrication of implant abutments, implant supported prosthesis framework etc.

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