ORIGINAL RESEARCH

Comparative Evaluation of Surface Roughness and Color Stability of Polyetheretherketone with Conventional Interim Prosthetic Materials: An *In Vitro* Study

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Abstract

Aim: The aim of this *in vitro* study was to compare the surface roughness and color stability of polyetheretherketone (PEEK) with those of conventional interim prosthetic materials like polymethylmethacrylate, bis-acrylic composite, and rubberized diurethane dimethacrylate, following immersion in solutions of varying pH value.

Materials and methods: A total of 320 circular discs with 10 mm diameter and 2 mm height were divided based on the fabrication ($n = 80$)—group A: polymethylmethacrylate; group B: bis-acrylic composite; group R: rubberized diurethane; and group P: hot-pressed PEEK—and were subjected to baseline measurement of roughness ($n = 40$) and color ($n = 40$) using 3D profilometer and UV-Vis spectrophotometer, respectively. Later, 10 samples from each group were immersed in distilled water, black coffee, green tea, and Pepsi, respectively, for 120 days, and measurements of roughness and color were repeated. The differences in roughness (ΔR^a) and color change(ΔE) were calculated and statistically analyzed with a significance level of p-value < 0.05.

Result: Irrespective of the immersion solution, the highest mean difference in the roughness values was shown by rubberized diurethane specimens: $\Delta R^a = 3.574880$ (0.0048350) in carbonated beverages, and lowest difference was shown by bis-acrylic composite: $\Delta R^a = 0.29004$ (0.0017473) in distilled water. The greatest color stability was exhibited by PEEK. The type of interim material and immersion solution had a statistically significant effect on change in color and roughness values.

Conclusion: The immersion in solutions of varying pH had a significant effect on surface roughness and color stability of all the tested materials. The R^a value of all specimens after immersion was still within the clinically acceptable range. Polyetheretherketone was the most color stable material in all solutions, except in green tea.

Clinical significance: This study will provide guidance to dentists and patients regarding the selection of interim material for long-term use, depending on the effect of beverage consumption on its color stability and roughness.

Keywords: Color stability, Interim prosthetic material, Polyetheretherketone, Profilometer, Spectrophotometer, Surface roughness.

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Introduction

Interim restorations are an essential component of all fixed prosthodontic rehabilitations, ranging from single crowns to full mouth rehabilitation with implants. Once in place, interim prostheses are expected to maintain esthetics, optimal gingival health, relationship between abutments, and function. They also play a vital role in improving and maintaining the gingival contour.[1–3]

When provisional restorations are indicated for longer duration, as in implant integration, extensive dental treatments involving occlusal rehabilitation, change in vertical dimension, or treatment of temporomandibular disorders, the property of surface roughness and esthetics become even more important in the selection of a provisional material. Many of these situations require a minimum of 3 to 4 months of provisionalization, and perceptible change in the color of the material during this period can lead to esthetic complications and possible refabrication.[4] Also, longer the provisional is in place, greater will be the impact of its surface roughness on the gingival architecture, which may adversely affect the success of the future permanent restoration.[5,6]

Materials commonly used for fabrication of provisional restorations are polymethylmethacrylate (PMMA) and bis-acrylic composite.[6,7] Rubberized diurethane dimethacrylate, an improved rubberized resin, is another direct esthetic crown material, which claims to have good impact and fracture resistance, good marginal adaptation, and enhanced esthetics.

Polyetheretherketone (PEEK) is a thermoplastic monochromatic semi-crystalline polymer, which is being increasingly used in dentistry. Some of its short-term applications in fixed prosthodontics include fabrication of interim restorations, temporary abutments for implants, resin-bonded fixed dental prostheses veneered with composite, and customized healing abutments.[8]

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Table 1: Experimental groups

Group code	*Trade name*	*Manufacturer*	*Shade*	*Type*
Gp A	Semident SC-10	Samit Dental Products, Jhandewalan, New Delhi, India	No. 3	Autopolymerized polymethylmethacrylate
Gp B	Protemp™ 4	3M India Ltd., Bengaluru, Karnataka, India	A 2	Bis-acrylic composite
Gp R	Tuff-temp™ Plus	Pulpdent Corp., Watertown, Massachusetts, USA	A 2	Rubberized diurethane dimethacrylate
Gp P	BioHPP®	Bredent, Chesterfield, Derbyshire, UK	White	Polyetheretherketone

Polyetheretherketone is favored over metal in areas where esthetics is the major concern.[9] Also, favorable mechanical properties combined with the lower elasticity of PEEK, which reduces transfer of stresses to the abutment teeth, could make it a good choice as a long-duration provisional material in situations where abutments are not periodontally sound.[8]

The modified form of PEEK, BioHPP, has excellent polishability, low plaque affinity, and good wear resistance.[10] Polyetheretherketone is available in both hot pressed and milled variants. Although studies on pressed PEEK have reported a larger marginal gap than the milled variant, it is still within the clinically acceptable limits.[11,12] The lesser cost of pressed PEEK makes it a viable option for provisional restorations.

Since beverages of different pH are regularly consumed by vast majority of the population, their effect on the color stability and roughness of long-term provisional materials must be investigated so that the appropriate material can be selected or necessary lifestyle modification can be suggested to the patient during the period of provisionalization.

Currently, there is a lack of evidence of the effect of immersion in solutions of varying pH on the surface roughness and color stability of hot-pressed PEEK and how it compares with conventional interim prosthodontic materials, in global literature. Hence, this study was undertaken to compare the surface roughness and color stability of four interim prosthodontic materials after immersion in beverages of varying pH. The null hypothesis was that immersion in solutions of varying pH will not have a significant effect on surface roughness and color stability of interim prosthetic materials.

Materials and Methods

Study Design and Study Setting

This experimental *in vitro* study was conducted in the Department of Prosthodontics, Government Dental College, Kozhikode, Kerala, India, and has been approved by the Institutional Ethical Committee (IEC no.: 193/2020).

A total of 320 circular discs of diameter 10 mm and thickness 2 mm were fabricated using four interim prosthetic materials and divided into four experimental groups (n = 80)—group A (Gp A): autopolymerized PMMA, group B (Gp B): bis-acrylic composite, group R (Gp R): rubberized diurethane dimethacrylate, and group P (Gp P): hot-pressed polyetheretherketone (Table 1).

Fabrication of Samples

For groups A, B, and R, the materials were mixed as per the manufacturer's instructions.

While hand mixing was used for Gp A specimens, Gp B and Gp R utilized an automix system (IDS Denmed dispensing gun, IDS Denmed Pvt. Ltd., New Delhi, Delhi, India). The mix was then packed into a stainless-steel cylindrical mold with an inner diameter of 10 mm and a height of 2 mm. The mold was covered and compressed with a glass plate to pack the mix. Since the curing mechanism of the materials was different, Gp A specimens were allowed to cure at room temperature and Gp B specimens were cured using a dental curing light (Woodpecker i-LED Curing Light, Guilin Woodpecker Medical Instrument Co. Ltd., Guilin, Guangxi, China) for 60 seconds. Gp R specimens with a dual cure mechanism were polymerized as per the manufacturer's instructions, removed from the mold after 4 minutes and 45 seconds from the beginning of the mix and wiped with alcohol.

Gp P specimens were fabricated by hot pressing an ingot at 400° C in For2Press system (Bredent Medical GmbH & Co., Weissenhorner, Senden, Germany) and sectioning with a carbide disc to get specimens of 10 mm diameter and 2 mm thickness.

All the specimens were examined and those with voids were discarded. The remaining specimens were polished with silicon carbide abrasive paper (Performer Silicon Carbide Abrasive Paper, AE Abrasives Edge Pvt. Ltd., New Delhi, Delhi, India) of increasing fineness (1,000, 1,200, and 1,500). To ensure standardization, each specimen was polished for 30 seconds, using circular movements under constant water irrigation, followed by cleaning in an ultrasonic water bath (Durasonix 3.2 L Ultrasonic Cleaner, China) for 5 minutes.

Measurement of Baseline Values

Half of the specimens of each group (n = 40) were subjected to baseline roughness measurement and the remaining half (n = 40) were measured for color values using 3D profilometer (InfiniteFocusG5, Bruker Alicona, Raaba, Austria) and UV-Vis spectrophotometer (UV-2600i, Shimadzu Corp., Nakagyo-ku, Kyoto, Japan), respectively.

The non testing side of specimens, subjected to roughness measurements, were notched with a small bur to reorient the specimen on 3D profilometer, for repeated measurement.

The 3D profilometer had a numerical aperture of 0.3 and a vertical scanning speed of 1,000 to 3,000 nm per minute, and was capable of non-contact three-dimensional (3D) surface roughness measurements with 10× magnification, at three different locations of each specimen: at center and at 2 mm anterior and 2 mm posterior to the central point. The mean roughness value, S^a (3D), and R^a (two-dimensional) were calculated and entered.

Baseline absorption and transmittance values for the entire range of visible light wavelengths from 400 to 700 nm were measured using an UV-2600i UV-Vis spectrophotometer, with a double beam, single monochromator optical system which functions at a wavelength range of 185–900 nm with a wavelength accuracy of ±0.1 nm, scanning speed of 4000–0.5 nm per minute using a light source of 50-Watt halogen lamp. Color Calculation Software was used to calculate the Commission Internationale del'Eclairage L*a*b*(CIELAB) color values of specimens based on absorption spectra. The color estimations were repeated three times for each specimen, and the baseline color parameters were recorded.

Following baseline measurements, color stability and roughness of the specimens were evaluated using four beverages of varying pH as given in Table 2.

Table 2: Comparison of change in two-dimensional roughness (ΔR_a) of experimental groups in different solutions

Group code	*Solution 1**	*Solution 2**	*Solution 3**	*Solution 4**
Gp A	0.73316 (0.0027664)[a]	1.25688 (0.0012458)[a]	0.324300 (0.0012450)[a]	0.731620 (0.0024519)[a]
Gp B	0.29004 (0.0017473)[b]	0.52158 (0.0093687)[b]	0.340800 (0.0037068)[b]	0.547700 (0.0027377)[b]
Gp R	2.39502 (0.0301034)[c]	2.4518 (0.0043824)[c]	1.953520 (0.0013828)[c]	3.574880 (0.0048350)[c]
Gp P	0.54780 (0.0025952)[a,b,c]	0.643 (0.0257425)[a,b,c]	0.471000 (0.0031662)[a,b,c]	0.418320 (0.0019588)[a,b,c]

Values given are all mean(standard deviation) values. *Between groups *p*-value statistically significant. [a,b,c]Pairwise comparison between groups using Bonferroni test. Different superscripts indicate statistically significant difference

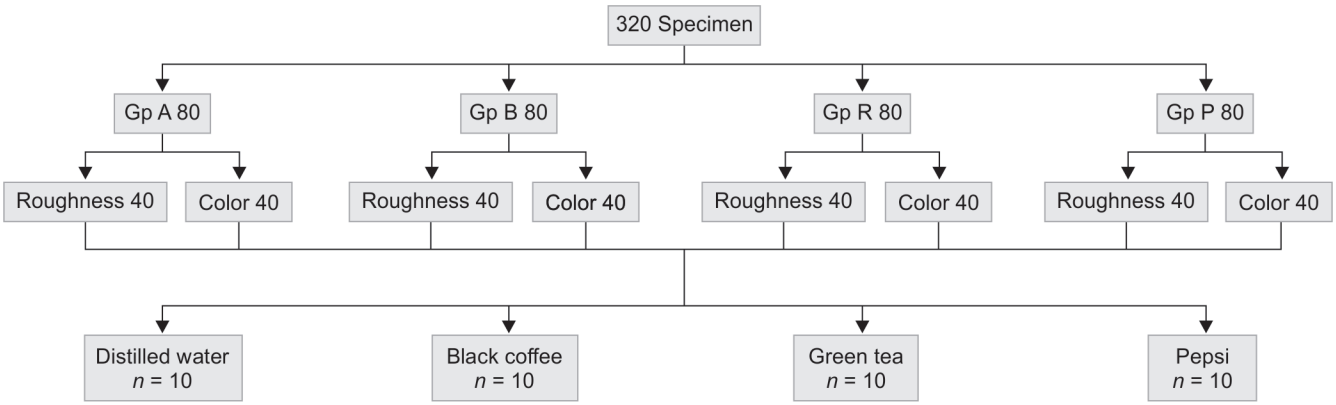

Fig. 1: Schematic representation of allocation of samples to various subgroups

Preparation of Immersion Solutions

Solution 1 was distilled water (Bepure Distilled Water, Bepure Tech Pvt. Ltd., Mumbai, Maharashtra, India) with pH 7 and acted as the control medium.

Solution 2 was black coffee with pH 5 and was prepared by adding 2.8 gm of coffee powder (Nescafe Classic, Nestle India Ltd., Gurugram, Haryana, India) to 150 mL of boiling water. After stirring the solution for 15 seconds every 2 minutes, it was filtered using a filter paper on cooling.

Solution 3 was green tea with pH 5.6 and was prepared by pouring 100 ml of boiled water to the 1.3 gm tea bag (Lipton Green Tea Clear & Light, Hindustan Unilever, Mumbai, Maharashtra, India) placed in a container followed by removal of the tea bag after 2 minutes from the solution.

Solution 4 was a commercially available carbonated soft drink (Pepsi, PepsiCo India Holdings Pvt. Ltd., Gurugram, Haryana, India), with a pH 2.5.

Thus, each group was further divided into four subgroups based on the immersion solution ($n = 10$). A schematic representation of allocation of samples into various subgroups is shown in Figure 1.

Each sample was immersed daily for 45 minutes, in 20 ml of their corresponding solutions, in small cylindrical closed containers for 120 days. The solutions were renewed, and their pH was verified using pH meter (Wellon pH meter, Super Tek Ro System Pvt. Ltd., New Delhi, Delhi, India) every day.

Measurement of Final Values

After 120 days, the specimens were cleaned with distilled water and dried using tissue paper. The specimens belonging to the roughness group were once again subjected to roughness measurement using the same 3D profilometer, and the color values of the specimens belonging to the colorimetry group were measured using the same UV-Vis spectrophotometer. All these measurements were done under the exact conditions and the same manner as for the baseline measurements.

The difference in roughness in two dimension and 3D, ΔR^a and ΔS^a, respectively, were calculated. The calculation of the color difference, ΔE was done using the below formula:

$$\Delta E = [(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2]^{1/2},$$

where L* is the perceptual lightness and a* is the red/green coordinate, and b* is the yellow/blue coordinate.

The SPSS software version 25 was used for statistical analysis in this study. Two-way analysis of variance (ANOVA) was used to evaluate the effect of the type of interim material and immersion solution, on changes in color and roughness values. Since ANOVA showed statistically significant difference between the groups, it was followed by Bonferroni *post hoc* test. The correlation between change in roughness (ΔS^a) and change in color values (ΔE) was measured using Pearson correlation coefficient. Significance level was set at $p < 0.05$.

Results

The initial R^a values of the specimens ranged from 0.18 to 0.37 µm. The mean R^a value of PMMA and bis-acrylic composite were 0.18 and 0.19 µm, respectively, whereas rubberized diurethane and PEEK exhibited an R^a value of 0.223 and 0.37 µm, respectively. Even after immersion in various solutions, the R^a values of all specimens were within 3.8 µm, with rubberized diurethane showing the maximum R^a value. After 120 days, changes were observed in the R^a values of different interim materials across the various immersion solutions. Table 2 shows the change in R^a values (ΔR^a) of different experimental groups after immersion.

The initial S^a values, on the contrary, were lowest for Gp B (0.21–0.28 µm) and highest for Gp P (0.44–0.54 µm). For Gp A and Gp R, the S^a roughness values ranged from approximately

Table 3: Comparison of change in three-dimensional roughness of experimental groups (ΔS_a) in different solutions

Group code	*Solution 1**	*Solution 2**	*Solution 3**	*Solution 4**
Gp A	1.0269 (0.1745082)[a]	1.381200 (0.1281369)[a]	1.038900 (0.1209733)[a]	0.956740 (0.0092689)[a]
Gp B	0.525100 (0.0038321)[b]	0.810700 (0.0078533)[b]	0.980800 (0.0053465)[b]	0.598700 (0.0021599)[b]
Gp R	3.595 (0.0488834)[c]	1.909700 (0.0070551)[c]	2.879100 (0.0031329)[c]	4.852100 (0.0100856)[c]
Gp P	0.808200 (0.0012728)[a,b,c]	0.745200 (0.0028931)[a,b,c]	0.734500 (0.0013191)[a,b,c]	0.987700 (0.0043134)[a,b,c]

Values given are all mean(standard deviation) values. *Between groups *p*-value statistically significant. [a,b,c]Pairwise comparison between groups using Bonferroni test. Different superscripts indicate statistically significant difference

Table 4: Comparison of change in color of experimental groups (ΔE) in different solutions

Group code	*Solution 1**	*Solution 2**	*Solution 3**	*Solution 4**
Gp A	2.160 (0.0452769)[a]	16.21 (0.0761577)[a]	7.453 (0.0671491)[a]	2.599 (0.0649115)[a]
Gp B	1.6080 (0.0068920)[b]	9.43 (0.0924662)[b]	20.23 (0.1537856)[b]	11.700 (0.1282576)[b]
Gp R	1.4650 (0.0204450)[c]	9.59 (0.0651920)[c]	20.11 (0.0764853)[c]	4.750 (0.0430116)[c]
Gp P	0.2370 (0.0033912)[a,b,c]	0.88 (0.0148155)[a,b,c]	6.462 (0.0567450)[a,b,c]	0.8480 (0.0223495)[a,b,c]

Values given are all mean(standard deviation) values. *Between groups *p*-value statistically significant. [a,b,c]Pairwise comparison between groups using Bonferroni test. Different superscripts indicate statistically significant difference

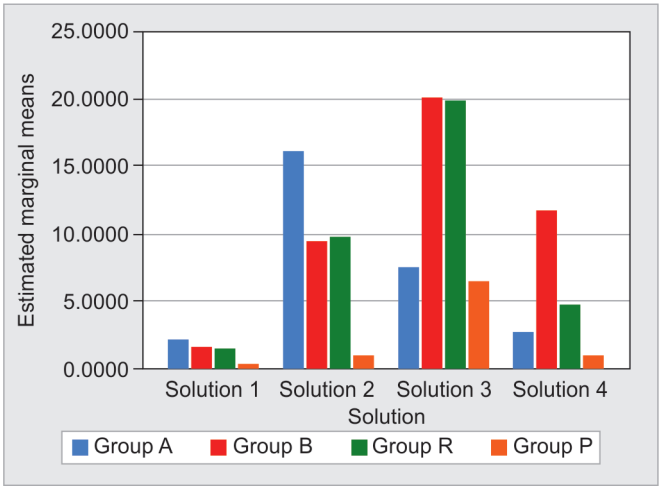

Fig. 2: Color change of experimental groups in different immersion solutions

0.25 to 0.45 µm. The change in S^a values (ΔS^a) following immersion in various solutions for a time period of 120 days are shown in Table 3.

Irrespective of the immersion solution, the highest difference in the roughness values (R^a and S^a) was shown by Gp R. The lowest difference was shown by Gp B in distilled water and highest by Gp R in carbonated soft drink.

The mean and standard deviation values of change in color (ΔE) of various groups are presented in Table 4. When comparing the ΔE of the various interim prosthetic materials in different solutions, the greatest color stability was exhibited by Gp P, irrespective of the immersion solution. The maximum color change was seen following immersion in green tea for all groups except Gp A, which showed maximum color change in black coffee. A bar chart showing the color change of experimental groups in different immersion solutions is shown in Figure 2.

According to two-way ANOVA, the type of interim material and immersion solution had a statistically significant effect ($p < 0.05$) on change in color and roughness values. Bonferroni *post hoc* test was done to identify exactly which groups and solutions differ from each other. The *post hoc* test showed that the difference in ΔR^a and ΔE between all the groups and solutions were significant. However, the difference in ΔS^a within Gp A was not significant ($p < 0.05$) between solution 1 and solution 3, solution 1 and solution 4, and between solution 3 and 4.

Pearson correlation showed a high positive correlation between initial roughness (S^a) and change in color values (ΔE) in Gp P and medium negative correlation in Gp B ($p < 0.05$) (Table 5). The correlation in Gp A and Gp R were not significant.

Thus, it can be inferred that immersion in solutions had the highest effect on the roughness values of rubberized diurethane and the least effect on bis-acrylic composite and PEEK. Also, among the materials tested, PEEK is the most color stable except when immersed in green tea. The initial surface roughness significantly affected the color stability of PEEK.

Discussion

Based on the results of the study, the null hypothesis was rejected, since immersion in solutions of varying pH had a significant effect on surface roughness and color stability of all the tested interim materials. In this study, the surface roughness was measured using both R^a and S^a. S^a represents the arithmetic mean in 3D for the height of the peaks and valleys of surface roughness. Since sufficient data regarding the relation between S^a values and plaque accumulation is not available, the R^a values were also recorded in this study to understand the clinical relevance of the recorded roughness values.

Of all the interim materials evaluated, rubberized diurethane dimethacrylate showed the highest change in roughness values in all immersion solutions. The S^a values of bis-acrylic composite and PEEK were least affected by immersion in various solutions. The initial mean R^a value of PMMA and bis-acrylic composite were lower than the plaque accumulation thershold of 0.2 µm.[13] According to Kaplan et al.,[14] any value within 10 µm was clinically undetectable and hence acceptable. Even after immersion in various solutions, the R^a values of all specimens were within the clinically acceptable range.

The soaking time had a significant effect on the color stability of provisional material. Since PEEK is not usually used for short periods of provisionalization, in this study, a single immersion period of

Table 5: Correlation between initial roughness and color change

Group code	*Correlation coefficient*	*p-value*
Gp A	0.117	0.624
Gp B	−0.447*	0.048
Gp R	0.074	0.757
Gp P	0.830*	0.000

*Pearson correlation coefficient is significant at the 0.05 level (two-tailed)

120 days was selected for all materials. According to Guler et al.,[15] coffee consumption (one dose) lasts for 15 minutes, and the average consumption of coffee is 3.2 cups per day. The oral clearance rate of other beverages also was in the range of 10–15 minutes as per Hans et al.[16] Hence, in this study, a daily immersion for 45 minutes was done to simulate clinical conditions. Irrespective of the immersion solution, the color stability of PEEK was superior to other interim materials. Except for immersion in green tea, PEEK showed a clinically imperceptible color change, i.e., ΔE. Polyetheretherketone is a hydrophobic material, and its water absorption and solubility value are low when compared with those of acrylic and composite materials which in turn results in lesser absorption of colorants into its matrix and hence resistance to stainability by various beverages.[17–19] Similar staining profile was seen for BioHPP in a study conducted by Porojan et al.,[20] where maximum staining of BioHPP was seen in tea.

Except for PMMA, which showed maximum color change in coffee, all the interim prosthetic materials showed the maximum color change when immersed in green tea. As per this study, bis-acrylic composite was more color stable in coffee than methyl methacrylate. This result contradicts the findings of Rutkunas et al.[21] and da Fonsêca Costa and Lima.[22] Water absorption in autopolymerizing acrylic resin is influenced by residual monomer and air bubble inclusion during mixing.[23] Since hand mixing was used for manipulation of PMMA, it could have resulted in increased presence of residual monomer and air bubble inclusion which could have, in turn, led to increased water inclusion, adversely effecting its color stability even in distilled water, than bis-acrylic, which was automixed. Autopolymerizing resins also exhibit higher solubility and inferior color stability due to oxidation of the amine accelerator they contain.

Also, according to Türker et al.,[24] higher discoloration is caused by fluid pigment from food and beverages in the more porous acrylic resins than the resin composites. The presence of modified Boven resin in Protemp 4 makes it hydrophobic, which could have reduced water absorption, resulting in its low color change in distilled water compared with PMMA.[25]

Composite showed maximum discoloration in green tea, which could be due to the adsorption or absorption of tannins, which deeply penetrate the composite matrix.[26] The yellow color causing material present in coffee has low polarity. Compared with coffee, tea has components having a higher polarity. The adsorption of these polar colorants from tea at the surface of composite resin materials could have led to greater staining in them.[27] Although the carbonated beverage Pepsi had the lowest pH, it may not have resulted in a severe discoloration due to the lack of yellow colorants.[28]

The surface roughness and color stability were investigated together because the optical property of a restoration is affected by surface roughness.[15,29] A strong positive correlation was seen between the surface roughness and color stability of PEEK. Hence, establishing a better surface finish could result in better color stability.

The strength of the study includes comparison of hot-pressed PEEK, a relatively newer material with the two most commonly used interim materials, autopolymerized PMMA and bis-acrylic composite. The materials were evaluated over a longer period (120 days), which is usually the minimum period of provisionalization indicated for extensive dental restorations including implants. Also, in addition to R^a value, the commonly used roughness parameter, S^a value, which gave a more comprehensive idea about the surface characteristics of the tested materials was recorded and compared.

This study has many limitations. Since evaluation was done only after immersion corresponding to 4 months, effect of storage time on surface roughness and color stability at various time intervals cannot be ascertained from this study. Both cleaning solutions and cleaning methods like tooth brushing may affect the staining of the materials, which have not been considered in this study. The exact simulation of the clinical situation was not possible since it was an *in vitro* study. To better understand the performance of interim material over varying time periods, clinical studies of different duration of provisionalization, with emphasis on the effect of cleaning methods should be conducted in the future to understand how these materials perform *in situ*.

Conclusion

The immersion in solutions of varying pH values has a significant effect on surface roughness and color stability of all the tested interim materials. The R^a value of all specimens after immersion was still within the clinically acceptable range. Polyetheretherketone was the most color stable interim material in all solutions, except green tea, which warrants its use in situations requiring long-term provisionalization. Green tea showed the maximum staining potential among the tested solutions for all interim materials except PMMA.

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