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COLOR STABILITY OF THREE PROVISIONAL MATERIALS AFTER EXPOSURE TO TWO CHROMATOGENS BEFORE AND AFTER THERMOCYCLING –AN IN VITRO STUDY

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Abstract:

Aim *- The present investigation was done with an aim to evaluate and compare the effect of two chromatogens on color stability of three provisional materials before and after thermocycling.*

Materials and methods *- Three commercially available provisional materials were chosen - DPI heat cure, Protemp™4 chemical cure and the relatively newer Luxatemp Solar dual cure material. Flat circular metallic dies were prepared of 22 mm diameter and 2 mm thickness. Total 150 samples were prepared from the materials using these dies. The samples were finished and polished using standardized methods. They were divided into five groups. Two groups from each material were subjected to a standardized thermocycling regimen. The samples were immersed in two staining solutions - coffee solution and sambhar curry solution for 30 days. Artificial saliva was used for the control group. The solutions were prepared using a standardized method and were changed every day. The color measurements were done twice - once before thermocycling and staining and once after, in CIE L*a*b* color system using a reflectance spectrophotometer.*

Results *– Statistical analysis was done in SPSS version 20.0 using One-way ANOVA and Tukey's post-hoc test. p value <0.05 was considered statistically significant.*

Conclusions *– New material Luxatemp Solar showed least color stability, followed by Protemp™4 whereas DPI showed maximum color stability. Sambhar curry showed higher staining ability. The color changes seen with coffee and control were not clinically perceptible. Thermocycled samples showed more color change than non thermocycled samples.*

Keywords: Color stability, provisional materials, coffee solution, sambhar curry solution, thermocycling.

CLINICAL IMPLICATIONS *- DPI provisional restorative material showed better color stability than Protemp™4 and Luxatemp Solar. However, the values of color change were clinically acceptable with the control and coffee group for all materials. Hence, patients with a simple or a health conscious diet, or the ones regularly consuming coffee, can be given prosthesis using any provisional material. If the diet of the patient includes typical Indian curries, it is advisable to use DPI heat cure provisional material as it showed clinically acceptable color change with sambhar, which was the representative of Indian curries.*

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Introduction

Provisional restorations are an essential part of fixed prosthodontic treatment.[1] They are designed to enhance esthetics, stabilization and/or function for a limited period of time, after which they are to be replaced by a definitive prosthesis.[2] These may be required to be placed in the patients' mouth for a few days to few weeks. Occasionally, interim treatment has to function for extended intervals and provide long-term tooth protection and stability while adjunctive treatment is accomplished,[3] like in the midst of the Covid pandemic where most dental clinics performed only emergency treatments globally or other emergency situations where patient cannot report to the clinic. Amongst all its functions, esthetics of the provisional restoration is of prime importance especially in cases where the provisional restorations are going to be used for a long period of time and or are in the esthetic zone.[4] Discoloration of provisional restorations may result in patient dissatisfaction and an additional expense for their replacement[5], adding to the number of visits and costs.

Regardless of their chemistry, most provisional restorative materials are subject to sorption, a process of absorption and adsorption of liquids. As a result, color changes may occur over time when these provisional restorations are subjected to various staining agents.[6] Indian food consists of various chromatogenic substances such as tea, coffee, colas, turmeric powder, red chilli powder, spices, oil, curry etc. which are consumed on a daily basis and can adversely affect the color of the provisional restorative material.[7] With globalization, Indian food is now relished on a regular basis in most parts of the world. A number of studies have investigated the color stability of provisional materials in various chromatogens. However, the effect of commonly consumed chromatogens by the Indian population on provisional restorative materials has not been studied much.

Intraorally, temperature changes are seen induced by routine eating and drinking. Thermocycling can be done to simulate this clinical situation in the laboratory.[8] However, limited data is available about the color stability of provisional resins on temperature changes subjected to thermocycling.

Today, with increased dental awareness amongst patients and their improved standard of living, it is imperative for the Prosthodontist to provide a prosthesis which not only functions efficiently, but also maintains its appearance over the entire period of service.

Considering these facts, the present study was undertaken to evaluate the color changes that occurred when DPI heat cure, Protemp™4 chemical cure and the relatively newer Luxatemp Solar dual cure provisional restorative materials, were subjected to immersion in coffee and sambhar curry solution, which are common Indian chromatogens, for a period of 30 days, before and after thermocycling, which represented temperature changes (Table1). The null hypothesis was that there is no significant difference in the color stability of tested provisional materials when exposed to chromatogens or thermocycling.

Material and Methods

The details of all materials are given in Table 1. The methodology was divided as follows:

A. Preparation of samples from provisional materials (Fig 1) - A total of 150 samples (50 samples from 3 different provisional materials) were prepared. 6 disc shaped brass dies were fabricated, 22 mm in diameter and 2 mm in thickness. These were used for preparation of a gypsum mold (for enabling heat curing of DPI samples) and a silicone mold (easy retrievability of Protemp™4 and Luxatemp Solar samples). (Fig 2)

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i. Preparation of DPI heat cure samples (Fig 3)

50 samples were fabricated of shade A according to manufacturer's instructions and ADA specifications no. 27. A standard medium sized mix of 0.8±0.1gm was used per sample (4.8±0.1 gm. for 6 samples) as per ADA specifications no. 27.[9] Thus, pre-weighed 3.6 gms of polymer and 1.2 ml of monomer was used for preparing 6 samples in the gypsum mold. The curing was done in clean water bath in acrylizer, at 100 °C for 1 hour.[10]

ii. Preparation of Protemp™4 chemical cure samples and Luxatemp Solar dual cure samples (Fig 4,5)

50 samples were fabricated of shade A2 according to manufacturer's instructions. The material was dispensed into the silicone mold through the dispensing gun (10:1 mixing ratio). A glass slab was placed on top of the assembly along with a 5 kg weight to extrude the excess material. The Protemp™4 material was allowed to polymerize by chemical curing for 5 minutes for final setting, while the Luxatemp Solar

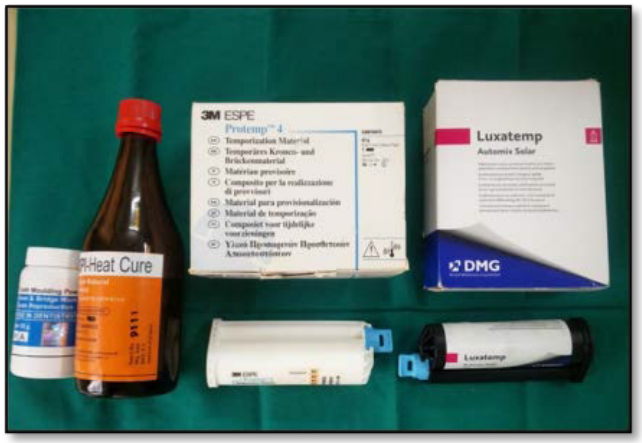

Fig. 1. Provisional materials used

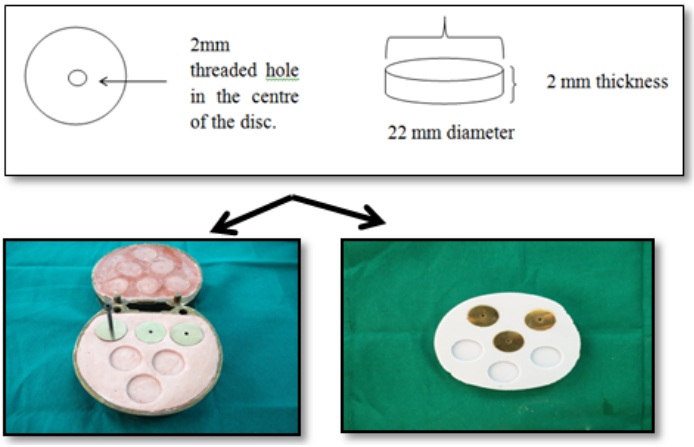

Fig. 2. Preparation of gypsum and silicone mold from dies

TABLE 1 Details of materials and solutions

SR. NO.	TYPE OF MATERIAL	TRADE NAME	MANUFACTURER	SHADE/ BATCH NO.	
1	Heat-cure polymethyl methacrylate	DPI Tooth molding C & B Material	Dental Products of India, The Bombay Burmah Trading Corporation Ltd., Mumbai – 01	A	3136
2	Chemical-cure bis acrylic resin composite	Protemp™4	3M ESPE, 3M Deutschland GmbH Dental Products, Germany	A2	604604
3	Dual-cure bis acrylic resin composite	Luxatemp Automix Solar	DMG, ChemischPharmazeutischeFabrik GmbH, Hamburg, Germany	A2	742313
4	Artificial saliva	Artificial Saliva	MP Sai Enterprise, Mumbai-53	-	
5	Coffee solution	Nescafe´ Original 3in1 premix	Nestle´, Lampung 35243, Indonesia	52714787LA	
6	Sambhar Solution	Everest Sambhar Masala	S. Narendrakumar& Co., Mumbai – 83	38E 6071	

material was allowed to polymerize initially by chemical curing for 2 minutes and then it was light cured at intervals of 20 seconds for 1 minute on both the sides of the sample for final setting. The samples were then retrieved, cleaned with ethanol to remove the oxygen inhibiting layer and polished.

All the samples were polished using 15 second application of coarse pumice applied with a moist muslin wheel on a dental lathe (Unident, India Pvt. Ltd) operating at 1500 rpm. They were then rinsed with distilled water to remove any debris and stored in distilled water for 24 hours at 37°C. This rehydration simulated the first day of service for provisional materials in the oral environment.[11,12,13]

TABLE 2 – Experimental groups and subgroups

Group A (DPI)	**Number**	**Solution**
A1	10	Artificial saliva (Control)
A2	10	Stained with Coffee solution
A3	10	Stained with Sambhar solution
A4	10	Thermocycled and stained with Coffee solution
A5	10	Thermocycled and stained with Sambhar solution
Group B (Protemp4)	**Number**	**Solution**
B1	10	Artificial saliva (Control)
B2	10	Stained with Coffee solution
B3	10	Stained with Sambhar solution
B4	10	Thermocycled and stained with Coffee solution
B5	10	Thermocycled and stained with Sambhar solution
Group C (Luxatemp Solar)	**Number**	**Solution**
C1	10	Artificial saliva (control)
C2	10	Stained with Coffee solution
C3	10	Stained with Sambhar solution
C4	10	Thermocycled and stained with Coffee solution
C5	10	Thermocycled and stained with Sambhar solution

TABLE 3 Pair wise comparison of mean ΔE between subgroups of Group A (DPI)

Comparison	Absolute Mean Difference	P-Value*
A1 vs. A2	0.261	0.7571(NS)
A1 vs. A3	1.426	< 0.001(HS)
A1 vs. A4	0.019	0.9999(NS)
A1 vs. A5	2.862	<0.001(HS)
A2 vs. A3	1.686	<0.001(HS)
A2 vs. A4	0.279	0.7055(NS)
A2 vs. A5	3.122	<0.001(HS)
A3 vs. A4	1.406	<0.001(HS)
A3 vs. A5	1.436	<0.001(HS)
A4 vs. A5	2.842	<0.001(HS)

*Obtained using Tukey test; HS: Highly Significant; S: Significant; NS: Not significant

TABLE 4 Pair wise comparison of ΔE across subgroups of Group B (Protemp4)

Comparison	Absolute Mean Difference	P-Value*
B1 vs. B2	0.0047	0.9999(NS)
B1 vs. B3	13.907	<0.001(HS)
B1 vs. B4	0.885	0.3243(NS)
B1 vs. B5	15.455	<0.001(HS)
B2 vs. B3	13.912	<0.001(HS)
B2 vs. B4	0.890	0.3191(NS)
B2 vs. B5	15.459	<0.001(HS)
B3 vs. B4	13.022	<0.001(HS)
B3 vs. B5	1.547	0.0136(S)
B4 vs. B5	14.569	<0.001(HS)

*Obtained using Tukey test; HS: Highly Significant; S: Significant; NS: Not significant

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50 samples in each group were thus prepared. (Fig 6) All the samples of each group were divided into five subgroups, 10 samples in each group. (Table 2) The discs was randomly picked and serially numbered with an indelible marker.

b. Thermocycling procedure - 20 samples from each group were subjected to the thermocycling procedure. Thermal cycles were simulated in an automated orbital shaker (REMI, Model S - 24BL, Rivotek, India) in a distilled water bath. Each cycle consisted of thermal variation at 5°C and 55°C with a 30-second dwell time and a 15 second transportation time. Hence, each cycle took 75 seconds to complete.[14] The procedure was conducted at 150 rpm and consisted of 1000 cycles.[15,16]

c. Staining procedure - The solutions (Fig 7) were prepared using a standardized method. Coffee solution was prepared using commercially available coffee powder (Nescafe Original 3-in-1 premix). 30 gm. powder (two pre weighed sachets of powder) was added to 300 ml boiling distilled

Fig. 3. Materials for preparation of DPI Heat cure samples

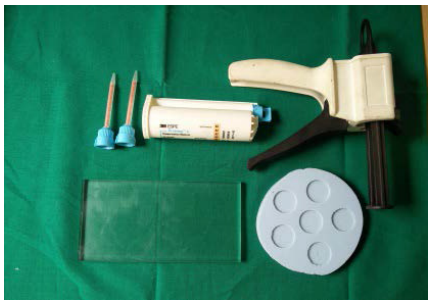

Fig. 4. Materials for preparation of Protemp 4Self cure samples

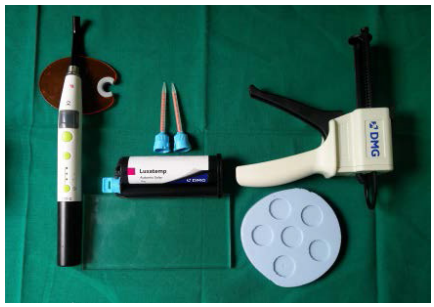

Fig. 5. Materials for preparation of Luxatemp Solar dual cure samples

TABLE 5 Pair wise comparison of mean ΔE across subgroups of Group C (Luxatemp solar)

Comparison	Absolute Mean Difference	P-Value*
C1 vs. C2	1.389	0.0020(S)
C1 vs. C3	19.683	<0.001(HS)
C1 vs. C4	2.158	<0.001(HS)
C1 vs. C5	24.708	<0.001(HS)
C2 vs. C3	18.294	<0.001(HS)
C2 vs. C4	0.768	0.1923(NS)
C2 vs. C5	23.319	<0.001(HS)
C3 vs. C4	17.526	<0.001(HS)
C3 vs. C5	5.025	<0.001(HS)
C4 vs. C5	22.551	<0.001(HS)

*Obtained using Tukey test; HS: Highly Significant; S: Significant; NS: Not significant

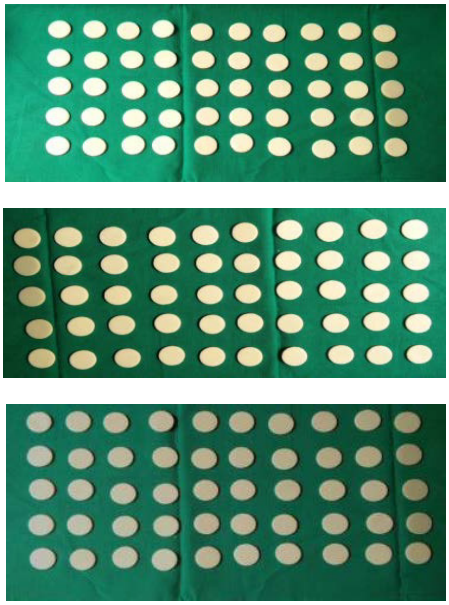

Fig. 6. Preparation of samples – DPI, Protemp4, Luxatemp Solar

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water and stirred for uniform mixing as per the manufacturer's instructions. Sambhar solution was prepared using commercially available sambhar powder (Everest Sambhar Masala). 30 gm. powder (two tablespoons with powder flattened) was added to 300 ml boiling distilled water and allowed to simmer for 5 minutes.

Both solutions were cooled down to room temperature and divided for staining of the 5 subgroups. Both were then diluted with artificial saliva in the ratio of 1:2[6,17]. Solution for control group was prepared using commercially available artificial saliva.

All samples were stored in their respective solutions at 37 °C in a thermostatically controlled incubator for the whole day for 30 days.[18,19] (Fig 8) Fresh solutions were supplemented everyday[20] and the color change after 30 days was observed. (Fig 9)

d. Color measurements of the samples - For the measurements of color, the samples were removed from the solutions and rinsed with distilled water for 30 seconds and gently

Fig. 7. Solutions used

Fig. 8. Samples stored in the incubator in their respective staining solutions

TABLE 6 - Comparison of ΔE of samples from all groups

Materials	Treatment groups: Mean ± SD of ΔE				
	Control	Stained with Coffee	Stained with Sambhar	Thermocycled and Stained with Coffee	Thermocycled and stained with Sambhar
Group A	0.68±0.37	0.42±0.31	2.11±0.64	0.70±0.18	3.54±0.72
Group B	0.93±0.88	0.93±0.39	14.84±1.71	1.82±0.83	16.39±0.89
Group C	0.44±0.17	1.83±0.54	20.12±1.03	2.60±0.68	25.15±1.08
F-value	F(2,27)=1.907	F(2,27) =28.14	F(2,27) =585.4	F(2,27) =23.25	F(2,27) =1426
P-value*	0.168 NS	<0.001 HS	<0.001 HS	< 0.001 HS	< 0.001 HS

*Obtained using one-way ANOVA; NS: Not Significant

cleansed with a soft bristle toothbrush to remove any loose sediments.[6,17] The samples were then blotted dry with tissue paper. Thereafter, the samples were subjected to spectrophotometric analysis twice – once before thermocycling and staining (baseline measurements of color) and once after 30 days (measurements for color change). The color measurements were done using a reflectance spectrophotometer (SpectraScan 5100, Premier Colorscan) with integrating sphere for solid samples. Value of color change was recorded in CIE L*A*B* color system. Color differences (ΔE*) were determined using the following equation:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

Values ΔE > 3.7 was considered as clinically not acceptable.[21]

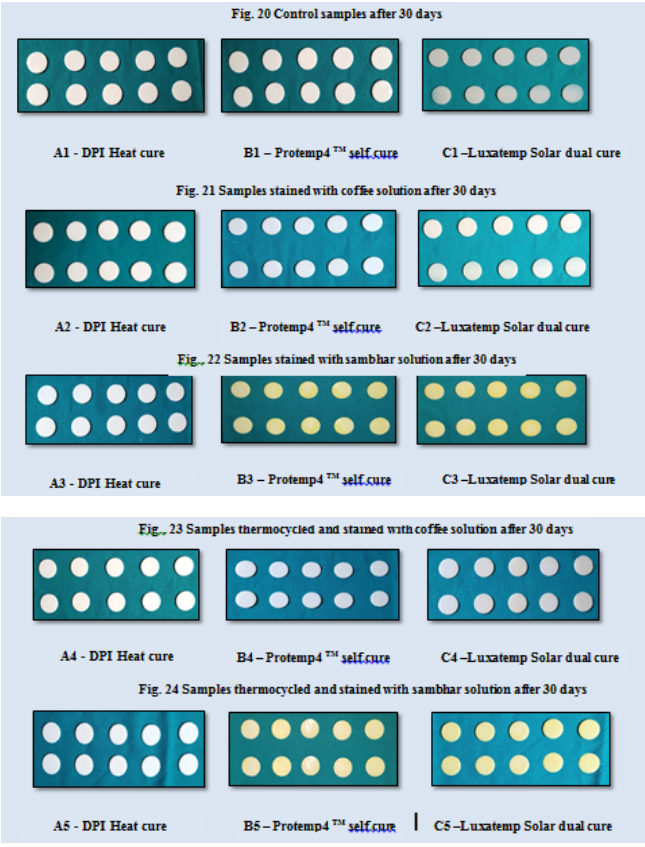


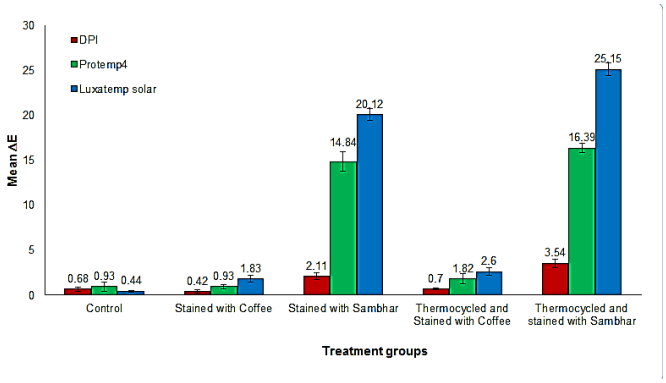
Fig. 9. Samples after 30 days showing color change

Results

The data of the color measurements was obtained and subjected to statistical analysis. The analyses were performed using SPSS version 20.0 (SPSS Inc.). The comparison of mean color stability across groups was performed using one-way analysis of variance (ANOVA). The pair wise analysis was performed using Tukey's post-hoc test. The p value <0.05 was considered as statistically significant while p value <0.001 was considered as highly significant.

It is evident from the comparison of mean ΔE for all the materials and treatments per sub-groups of DPI, Protemp™ 4 and Luxatemp Solar (Graph 1) that for all the groups, the maximum color change was seen with the thermocycled samples stained with sambhar curry solution and minimum discoloration was seen with control samples and those stained with coffee.

The intragroup analysis showed that for DPI and Protemp™ 4 groups, all the paired comparisons had statistically significant differences of mean color stability except comparisons between control samples vs. samples stained with coffee, controls vs. samples thermocycled and stained with coffee and samples stained with coffee vs. samples thermocycled and stained with coffee (Table 3, 4). For the Luxatemp Solar group, the



GRAPH 1 Bar chart showing comparison of mean ΔE for all the materials and treatments

only non-significant comparison was between samples stained with coffee vs. samples thermocycled and stained with coffee, while rest were statistically significant. (Table 5).

The intergroup analysis showed that in the comparison of samples of all experimental subgroups across different materials (Table 6, Graph 1), the mean for Luxatemp solar samples was significantly higher than that of other two treatment groups, as indicated by P-value < 0.001. In control samples, the mean for Protemp 4 was maximum, followed by DPI and then Luxatemp solar. The difference in the means, however, was statistically insignificant.

Discussion

Perceptible color change of the provisional material may compromise its acceptability. The provisional restorative materials chosen were commonly used ones, with the exception of Luxatemp Solar dual cure material, a relatively new material with very limited references in literature as regards stain resistance.

The spectrophotometer used in this study provided large area view i.e. a 25.4 mm port size that had a 22 mm view area for the color measurement of a sample. Hence, 22 mm diameter was selected as the size of the samples. Thickness of 2 mm was selected as it is generally the maximum facial or occlusal thickness of a provisional crown and it also allowed ease of manipulation and polishing.[19] Crispin and Caputo[23] found that the color of specimens with rough surfaces significantly changed. In order to standardize the procedures, the samples were finished using coarse pumice as it is routinely used in clinics for polishing of the restorations.[24]

The staining solutions, namely coffee solution and sambhar curry solution were those which are commonly consumed by the Indian population and those that have strong potential of staining. Both solutions were prepared in a standardized, quantifiable manner. The sambhar solution is a curry that contains most of the Indian chromatogenic spices that are added in the food routinely like turmeric powder and red chili powder.[7] It also contains a mixture of many Indian spices used – coriander, cumin, Bengal gram, black gram, pigeon pea, fenugreek, rice, common salt, curry leaf, tamarind, cassia and asafoetida, all of which have staining properties. This solution was the representative liquid of Indian food having multiple spices and condiments added and it showed the maximum discoloration in all the materials. Gupta[25] stated that the yellow-orange color of turmeric is due to a conjugated diarylhepnoid like Curcumin (3%), which is an active substance, also known as Natural Yellow. The uptake of this colorant by the resins causes staining. Munde and Radke [7] found that sambhar curry solution has the highest staining potential, followed by tea solution and then tobacco solution. The combination of various strong chromatogens and spices in sambhar, commonly found in Indian curries, could be the reason for the highly significant discoloration produced by this solution.

Guler et al[12] has shown that the addition of the sugar and milk powder in beverages results in increased color change and the differences were found to be significant. In the present investigation, pre weighed, commercially available sachets were used as it contained coffee, sugar and milk powder in fixed amounts for standardisation. The coffee solution showed less discoloration compared to sambhar curry solution and the discoloration caused was clinically acceptable ($\Delta E < 3.7$). Chan, Fuller and Hormati[26] found that coffee caused more discoloration than tea and cola beverages. In contrast to these findings, Um and Ruyter[27] reported that tea caused more discoloration than coffee after 48 hours of storage. Absorption and penetration of colorants into the organic phase of the resin-based material is probably due to

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compatibility of the polymer phase with the yellow colorants of coffee. Smaller molecular size of coffee coupled with the absorption phenomenon is said to be the cause of the staining potential of coffee.[20] Coffee also contains large amounts of staining agent like gallic acid which could be another reason for its staining capacity.[28]

The solutions were diluted with artificial saliva with a ratio of 1:2[6,17] and stored at 37 °C in a thermostatically controlled incubator to simulate intra oral environment.[18,19] Samples of the control group were dipped in commercially available artificial saliva as provisional restorations are bathed in saliva in the mouth. This group also represented the population whose diet was simple and did not include coffee or curries. Discoloration produced by this group was clinically not perceptible ($\Delta E < 3.7$) and intergroup comparisons were non-significant.

Thermal stress may affect the surface and structural integrities of resin materials and render the restorations more susceptible to staining and discoloration.[14] In the present study, thermocycled samples showed more discoloration than the non-thermocycled ones in all the three material groups. The results may be explained by **Strohaver and Mattie [28]** who stated that thermal energy, being sufficiently capable of causing decomposition of the organic components present in the resins, leads to the significant chromatic changes after thermal cycling. Thermocycling promotes volumetric contraction and expansion of materials, leading to degradation. **Oliveira et al[15]** found that thermocycling increased the surface roughness in most resins, which may also be the cause of increased discoloration seen with thermocycled samples in the present study.

Chemical discoloration has been attributed to the oxidation of polymer matrix or oxidation of unreacted double bonds in the residual monomers and subsequent formation of degradation products from water diffusion.[27] In the present study, methyl methacrylate material (DPI) was more color stable than the bisacryl composite materials (Protemp™ 4 and Luxatemp Solar). According to **Haselton, Diaz-Arnold and Dawson[6],** bis-acryl resins showed lesser color stability as compared to polymethyl methacrylate (PMMA) since bis-acryl polymers are more polar than PMMA polymers and therefore have greater affinity towards water and other polar liquids. Yannikakis et al[30] found that composite based resins can absorb water at a higher rate because of the high diffusion coefficient in comparison to methyl methacrylate. Hence, the present study is in agreement with these studies.

Strohaver and Mattie [28] showed that the degree of polymerization is critical to clinical performance of any resin system and that the degree of polymerization is not only dependent on type of resin used, but also the method of polymerization. They also found fewer voids in heat cure specimens than in self cured and light cured specimens. The use of heat for curing produced a higher degree of polymerization. Khokar et al[31] found that air voids in the resin material may lead to inhibition zones of unpolymerized material, resulting in lower color stability. This may be the reason of the lower color stability of dual cure (Luxatemp Solar) and chemical cure (Protemp™ 4) provisional materials as compared to the heat cure provisional resin (DPI).

The color stability of the tested provisional restorative material thus depends on the chemical composition of that material, the type of polymerisation and the environment it is subjected to.

Limitations

1. The oral hygiene habits may reduce the extrinsic stains on the restorative material but were not considered in the study.
2. Clinically, provisional restorations have curved surfaces but the samples in the

present study had flat surfaces as extraoral spectrophotometers can evaluate only flat surfaces.

Scope for further Studies

1. Studies considering oral hygiene measures taken by the patient.
2. In vivo studies to confirm the results of this in vitro study.

Conclusions

Within the limitations of the study, following conclusions can be drawn:

1. DPI heat cure provisional material is the most color stable.
2. Luxatemp Solar dual cure provisional material is the least color stable.
3. Protemp™4 chemical cure provisional material has intermediate color stability between heat cure and dual cure provisional material.
4. Amongst the staining solutions, sambhar solution showed the maximum staining ability followed by coffee solution.
5. Discoloration by coffee solution was not clinically perceptible. ($\Delta E \leq 3.7$)
6. Thermocycling affected the color stability of the all the provisional materials tested, causing an increased color change.

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