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Colour Stability of Tooth-Coloured Restorative Materials

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Abstract - *The colour stability of the surface and in-depth (2 mm) layer of two resin composites, a laboratory second-generation resin composite and a compomer were evaluated after 24 and 360 hours of water aging under dark and UV light conditions. The influence of various polymerization techniques on color changes was also evaluated. Color differences (ΔE*) showed higher color changes under UV light exposure than under dark storage, both at 24- and 360-hour evaluations. Color changes were statistically higher at the 360-hour assessment, in both conditions of maintenance. Compomer was the least color stable of the materials tested. Additional polymerization significantly decreased the colour change of both composite resins.*

KEY WORDS: Colour stability, Resin composites, Compomers, Laboratory resin composites, Additional polymerisation, Photoaging

INTRODUCTION

Increasing demand for aesthetic dentistry has been coupled with rapid rate of development of new aesthetic restorative materials. Colour match and long-lasting colour stability of the material are two of the major factors which influence the failure or the success of an aesthetic restoration. Resin composite and glass-ionomer restoratives have the advantage of the tooth-like appearance. Unfortunately, all these materials are known to be susceptible to various degrees of discolouration on prolonged exposure to the oral environment.

An unacceptable colour match still remains one of the major causes for replacement of anterior restorations[1,2]. Clinical studies have demonstrated that this colour change may take place during the first six months post-treatment, or later[3–7]. Investigators have concluded that most of the glass-ionomer and resin composite restoratives should not be considered as colour stable[8–15].

Discolouration is a multifactorial phenomenon[10,16]. There are external and internal causes for this discolouration. External discolouration, which is due to plaque and accumulated colour pigments, comes about through absorption or superficial chemical degradation and penetration of colourants. Intrinsic discolouration, on the other hand, triggered by environmental attack like UV irradiation or thermal energy, is induced by physico-chemical reactions in the composite matrix. Material-related factors, such as, the polymerisation system, the initiator and inhibitor agents, the type of monomers, the amount and the type of fillers and the conversion rate of carbon-carbon (C=C) double bonds have been associated with the colour changes of the resin composite materials[8,16–18]. Besides, external factors such as the finishing and polishing procedures, the ultraviolet light, the heat and the water have been contributed to colour change in the resin materials[8,10,17,19–21].

However, there are colour-related areas in the resin materials that have not been yet fully investigated. The influences of various additional polymerisation methods of the resin materials on their colour stability and the colour changes in the deep layers of the materials, which are not subjected directly to the aging factors, have not been assessed.

The aim of this study was to investigate the colour changes of surface and in-depth layers of resin restorative materials under various conditions of aging and to evaluate the influence of additional polymerization techniques on these changes.

MATERIALS AND METHODS

Two restorative resin composites, one laboratory second-generation resin composite and one polyacid-modified resin composite (compomer) were tested. Detailed description of the materials is presented in *Table 1.*

Twenty sample discs (6mm in diameter and 2 mm in thickness) were prepared for the materials Belle Glass HP and Compoglass and forty for Heliomolar Radiopaque and Prodigy Condensable. Each specimen was made by packing the material in a transparent plexi glass mould and pressing it between two glass plates. Photopolymerisation of all materials, besides Belle Glass HP, was carried out through the glass, for 40 sec from the upper surface only, using an 8 mm light-curing guide (Elipar Highlight, 3M). Twenty of the specimens of Heliomolar Radiopaque and Prodigy Condensable were subjected to additional polymerisation in the Brilliant DI–500 (Coltene) light/heat curing oven for 7 min at 120°C. Belle Glass HP samples were initially polymerised for 40 sec from the upper surface again, under the Teklite light-curing apparatus (Belle Glass, SDS-Kerr,) and then they were placed for the second cycle of curing, in the Belle Glass HP

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Table 1. *Description of materials tested.*

Material	*Code*	*Manufacturer*	*Batch No*	*Monomer*	*Filler content (% by mass/volume)*	*Filler size (μm)*
Heliomolar Radiopaque[1] (A3 shade)	HR	Vivadent, Ets., Liechtenstein	A16264	UDMA, Bis-GMA, TEGDMA	66,7 / 46	0.04–0.2
Prodigy Condensable[1] (A3 shade)	PC	Kerr/Sybron Co., Romulus, MI, USA	904590	Bis-GMA, TEGDMA	80 / 62	0.04–3
Belle Glass HP[2] (A3 Transluscent Dentin)	BG	SDS-Kerr, Orange, CA, USA	807585	UDMA, TEGDMA	78,7 / 66	0.6 (mean)
Compoglass[3] (A3 shade)	CG	Vivadent, Ets., Liechtenstein	919769	Bis-GMA, TEGDMA, UDMA, DCDMA	71,1 / 51	1.0 (mean)

1: resin composite, 2: laboratory second-generation resin composite, 3: polyacid-modified resin composite

UDMA: Urethane dimethacrylate
Bis-GMA: Bisphenol A-glycol methacrylate
TEGDMA: Triethylene glycol dimethacrylate
DCDMA: Dimethacrylate cycloaliphatic dicarboxylic acid

curing unit (Belle Glass, SDS-Kerr), for 20 minutes (140°C, pressure 60 psi, N2 atmosphere). All specimens were polished flat using moist 600 and 1000 grit SiC abrasive papers, in order to remove any defective polymerization layers caused by oxygen inhibition.

After polishing, all specimens were kept in distilled water at 37±1°C, in total darkness, for 24 hours, after which the initial colour values were measured on the upper (top) and the 2 mm-depth (bottom) surface of the samples. These initial colour values were used as baseline for the colour change measurements.

Specimens of all materials were divided into two groups of ten (group A and B). Specimens of group A were subjected to photoaging by UV light exposure under flooding distilled water in a weathering apparatus (SUNTEST CPS+, ATLAS Material testing Technology BV) and working parameters **BT** (Black Panel Temperature): 55±5°C, **E** (input irradiance): 400W/m^2, **WT** (water temperature): 37±1°C. Two exposure periods, the 24- and 360-hour, were used for specimens in the group A. Total irradiation dose counted was 34.566 KJ/m^2 for 24-hour and 51.8406 KJ/m^2 for 360-hour storage periods. Specimens of group B (control group), after the baseline measurement, were kept for 24 and 360 hours in total darkness, in distilled water at 37±1°C. After the initial baseline measurements further colour measurements were carried out at the 24- and 360-hour storage, for all groups.

Colour measurements were performed with a colourimeter (Dr Lance Micro Color, Braive Instruments) using standard illuminant C with 45° illumination and 0° viewing angle geometry. Data were recorded in CIEL*a*b* colour space system (CIE, International Commission on Illumination, Colorimetry, 1976)[22]. Each specimen was blotted dry with tissue paper and then, placed on a white background during the measurement to prevent potential absorption effects on any of the colour parameters measured. Five measurements were repeated on each specimen and surface, at the same assessment time and the average value was calculated. Colour change values (ΔE^*) between baseline and subsequent measurements were expressed as a distance of two colour positions in the three dimensional colour space and calculated according to the following formula: $\Delta E^* = [\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}]^{1/2}$.

Two-way and one-way analysis of variance (ANOVA) and Student-Newman-Keuls post-hoc tests were used to compare colour changes (ΔE^*) for all materials as a function of storage conditions (dark and UV light) and experimental period (24 and 360 hours). Unpaired t-test was applied to define, for each material, any significant differences of ΔE^* between groups A and B and the paired t-test to detect differences between the 24- and the 360-hour experimental periods. A 95% confidence level was chosen. Statistical analysis was performed by the software SPSS (ver.11) for windows.

RESULTS

Mean and standard deviation values of colour change (ΔE^*) for the top and bottom surfaces of the groups dark/water and photoaging at 24- and 360-hour testing periods are summarised in *Table 2*.

Statistical analysis showed that the aging conditions and times influenced significantly the colour change of each material. Significantly higher colour alterations were detected after UV light exposure compared to the aging under dark conditions for all the materials and times tested, on both surfaces of the specimens. Aging for 360 hours, under both conditions, caused significantly higher colour differences, compared to the 24-hour assessment.

Statistical analysis revealed significant differences among the materials tested (*Tables 3* and *4*). At 24-hour of photoaging, top surface of HR showed the highest ΔE^* values, while HRCH and PCHC analogues, the lowest. There was no significant difference among the top surfaces of PC, BG and CG. On the bottom surface, BG and PCHC presented significantly higher colour stability than the other materials and HR the highest colour change. After 360 hours of photoaging, CG demonstrated the highest ΔE^* level, on both surfaces. HRHC and PCHC groups showed the lowest ΔE^* on the top and BG and PCHC on the bottom surfaces. PC exhibited statistically significant less colour change than HR.

Additional thermo-photopolymerisation of Heliomolar Radiopaque (HRHC) and Prodigy Condensable (PCHC) decreased the ΔE^* values when compared to the corre-

Table 2. *Mean and standard deviation ΔE* values for all groups, aging conditions and surfaces measured.*

Groups	*Surface*	*Dark/Water*		*Photoaging*	
		ΔE (B-24h)	*ΔE (B-360h)*	*ΔE (B-24h)*	*ΔE (B-360h)*
Heliomolar Radiopaque (HR)	T	0.75±0.19	1.44±0.38	2.21±0.58	2.79±0.64
	B	0.85±0.19	1.53±0.51	3.01±0.52	3.76±0.50
Heliomolar Radiopaque-HC (HRHC)[1]	T	0.47±0.21	0.74±0.25	0.70±0.31	1.42±0.26
	B	0.43±0.18	0.69±0.30	1.85±0.39	2.88±0.42
Prodigy Condensable (PC)	T	0.56±0.18	1.06±0.22	1.24±0.55	2.50±0.46
	B	0.80±0.25	1.21±0.30	1.49±0.35	2.63±0.38
Prodigy Condensable-HC (PCHC)[2]	T	0.40±0.09	0.88±0.13	0.89±0.26	1.88±0.41
	B	0.49±0.08	0.95±0.11	1.06±0.23	2.16±0.39
Belle Glass HP (BG)	T	0.66±0.15	1.17±0.34	1.52±0.35	2.89±0.30
	B	0.48±0.15	0.94±0.21	0.99±0.30	1.90±0.36
Compoglass (CG)	T	0.64±0.25	1.91±0.58	1.54±0.33	5.69±1.22
	B	0.62±0.27	2.06±0.43	1.37±0.32	6.09±0.75

1:Heliomolar Radiopaque thermo-photopolymerized 2:Prodigy Condensable thermo-photopolymerised

T: top surface B: bottom surface (2 mm depth)

ΔE (B-24h): ΔE between baseline and 24-hour aging
ΔE (B-360h): ΔE between baseline and 360-hour aging

Table 3. *Material ranking and results of statistical analysis in dark/water groups.*

24 Hours		*360 Hours*	
Top Surface	*Bottom Surface*	*Top Surface*	*Bottom Surface*
PCHC $^{\alpha}$	HRHC $^{\alpha}$	HRHC $^{\alpha}$	HRHC $^{\alpha}$
HCHC $^{a\,b}$	BG $^{\alpha}$	PCHC $^{\alpha}$	BG $^{\alpha\,b}$
PC $^{a\,b}$	PCHC $^{\alpha}$	PC $^{a\,b}$	PCHC $^{a\,b}$
CG $^{a\,b}$	CG $^{a\,b}$	BG $^{a\,b}$	PC ^{bc}
BG ^{b}	PC ^{b}	HR.^{b}	HR ^{c}
HR ^{b}	HR ^{b}	CG ^{c}	CG ^{c}

Group means by time and surface, with the same letter are not statistically different.

Table 4. *Material ranking and results of statistical analysis in photoaging groups.*

24 Hours		*360 Hours*	
Top Surface	*Bottom Surface*	*Top Surface*	*Bottom Surface*
HRHC $^{\alpha}$	BG $^{\alpha}$	HRHC $^{\alpha}$	BG $^{\alpha}$
PCHC $^{\alpha\,b}$	PCHC $^{\alpha}$	PCHC $^{\alpha}$	PCHC ^{a}
PC $^{b\,C}$	CG ^{b}	PC ^{b}	PC ^{b}
BG ^{C}	PC ^{b}	HR ^{b}	HRHC ^{b}
CG ^{C}	HRHC ^{c}	BG ^{b}	HR ^{c}
HR ^{d}	HR ^{d}	CG ^{c}	CG ^{d}

Group means by time and surface, with the same letter are not statistically different.

Table 5. *Comparison of ΔE* top with ΔE* bottom surfaces in 360-hour aging groups.*

Dark/Water	*Photoaging*
HR $^{\alpha}$	HR ^{b}
HRHC $^{\alpha}$	HRHC ^{b}
PC $^{\alpha}$	PC $^{\alpha}$
PCHC $^{\alpha}$	PCHC $^{\alpha}$
BG $^{\alpha}$	BG ^{c}
CG $^{\alpha}$	CG $^{\alpha}$

Materials marked with **a** showed no significant different ΔE^*_{bottom} than ΔE^*_{top}
Materials marked with **b** showed significant $\Delta E^*_{bottom} > \Delta E^*_{top}$
Materials marked with **c** showed significant $\Delta E^*_{bottom} < \Delta E^*_{top}$ ($p<0.05$)

sponding single-photopolymerized groups (HR and PC) under UV light aging, on both surfaces evaluated.

After 24 hours of storage in distilled water under dark conditions, top surface of PCHC and bottom surface of HRHC provoked the lowest ΔE* values. Both, top and bottom surface of HR showed the highest colour changes. There were no significant differences among the bottom surfaces of HRHC, BG, PCHC and CG. At 360-hour dark/water storage, top surface of HRHC and PCHC and bottom surface of HRHC followed by BG and PCHC presented the lowest ΔE* values. CG exhibited the highest ΔE* values.

ΔE* values after 360 hours of photoaging were statistically significant lower at the top than the bottom surfaces for HR, and HRHC groups, while, BG revealed the opposite behavior (*Table 5*). No differences were noted for all materials after dark/water aging.

DISCUSSION

Colour stability is critical to the long-term aesthetics of restorations and has previously been studied in vitro for a variety of aesthetic restorative materials[8–15]. Colour change in resin restorations may be caused by changes in the material structure and formation of coloured degradation products, by change in the surface morphology and by extrinsic staining. Accelerating aging in a weathering chamber appears to model the first two of the mechanisms.

The accelerated aging process simulates the effects of long-term exposure to environmental conditions through light, temperature and humidity. Accelerated aging has been adopted to test the colour stability of resin composites since 1978[23]. It is desirable to determine the equivalence of short-term intervals of accelerated aging in long-term performance under oral circumstances. Various experimental periods have been proposed. It seems that the majority of colour change evoked through accelerated UV aging is elicited in the first 300 hours of the process[13,24]. In the present study, 24 and 360 hours of exposure to UV light under flooding water at 37±1°C were selected. Control specimens were kept in total darkness, in distilled water at 37±1°C, for the same period. As in other studies, the level of the colour changes was depended on the aging time, under both storage conditions[12,13,24].

Discolouration can be evaluated visually and by instrumental techniques. Tristimulus colourimeters are capable of detecting colour differences below the threshold of visual perception. Even though in dentistry, a colour change that is more than one unit (ΔE*>1) can be perceptible[25], the general population can distinguish colour differences of the value ΔE* = 3.3 or higher[26,27]. Discolouration above this level is referred to as clinically unacceptable. At 360-hour of photoaging, where the highest colour changes were demonstrated, the lowest ΔE* value was 1.42. However, out of the resin composites tested only Heliomolar Radiopaque showed ΔE* higher than 3.3 (ΔE* = 3.76). Compoglass though demonstrated a significantly higher colour change (ΔE* = 6.09), which was visually perceptible and clinically unacceptable.

Significantly higher colour alterations were detected after photoaging compared to the aging only under water for all the materials tested, on both surfaces of the specimens. Previous generations of resin composites exhibited higher susceptibility to UV light than to water, as well[16,24,28]. These findings show the strong effect of the UV irradiation on the colour stability of the materials assessed. UV light attacks the polymer matrix by photolysis and photo-oxidation resulting in bond scission and subsequent chemical transformation[29]. Reactions in the polymer matrix and formation of new functional moieties are involved. These structural changes induced by UV light are described as photo-degradation and reflect changes in physical, mechanical and optical properties of the materials. The amount of influence depends on the amount of the UV light exposure, the material composition, the presence of UV stabilizers, the concentration of defects or unsaturated sites and impurities, and the presence of unreacted C=C double bonds which can be oxidised[29]. Water sorption has been associated with colour change of the materials, as well. Water penetrates the polymer matrix manifesting molecules breakdown by hydrolysis, plasticisation, solubilisation and consequent degradation of the material[30,31]. All these structural changes and the surface roughness provoke change of the refractive index leading to colour instability.

The microfilled resin composite Heliomolar Radiopaque showed the greatest colour change of all the resin composites tested in the current study. It is an interesting finding that Heliomolar Radiopaque demonstrated significant colour alteration even after 24 hours of photoaging. Microfilled resin composites have been found to be less colour stable than hybrids[17,32,33]. This behavior has been attributed to the relatively higher resin content, which is less resistant to photolysis, photo-oxidation and to water sorption.

Additional thermo-photopolymerization resulted in higher colour stability for Heliomolar Radiopaque and Prodigy Condensable under all experimental conditions. However, this decrease was much more significant on the upper than the 2 mm-in depth surface. It has been proven that light and heat increase the conversion rate of C=C double bonds[34–37] with sequence improvement in the physico-mechanical properties[38,39], which may include the resistance to colour change. However, the conversion in the outer portion of the heat-cured composites was higher than in the inner portion[37]. The latter may explain the difference recorded between the top and bottom surface, which was mentioned above.

Belle Glass HP revealed a peculiar behavior. This material is a laboratory second-generation resin composite, which, after the initial photopolymerisation, is subjected to additional curing at 140°C under a nitrogen atmosphere. These conditions produce an oxygen-free environment and a higher degree of C=C conversion, which reaches to the 80% value[40,41]. Therefore, it would be suspected that the material could be less susceptible to colour change. However, this was not the case in the present study. On the top surfaces, after 360 hours of photoaging, no differences were verified compared to Heliomolar Radiopaque and Prodigy Condensable even without their additional – thermo-photopolymerisation. On the other hand, ΔE* on bottom surfaces of Belle Glass HP demonstrated the least values of all materials. This behavior might be associated with the conditions of the second cycle of cure subjected the material. Curing under nitrogen pressure may promote nitrogen entrapment, which may change the roughness and porosity of the direct exposed surface (top) and thus higher colour changes may be induced on this surface.

Compoglass was the least colour stable material in the present study. Polyacid-modified resin composites are more colour stable than glass-ionomer cements and resin-modified glass-ionomers and more susceptible to colour change than resin composites[12,42,43]. The acid-base reaction induced in polyacid-modified resin composites by water sorption generates continuous alterations in structure of the materials[44]. As a consequence, possible changes in refraction index and thus, changes in the initial colour can de provided. In 2001, Vargas *et al.*[15] recorded a gradual colour change in the compomer Dyract, which was higher than 3.3 after seven weeks of storage in artificial saliva at 37°C.

Bottom surface colour changes have a remarkable significance since they probably go along with a change in the chemical structure and the physicomechanical properties of the material[45]. Besides, intrinsic colour changes are irreversible. Higher ˜* values of the bottom compared to the top surfaces of the materials may reflect the negative effect of the in-depth cure on the colour stability of the subsurface layers, which also are not directly exposed to photoaging. It can be assumed that insufficient in-depth polymerization may provoke higher colour change in the inner layers of the materials, because of the decreased rate of C=C double bonds. ΔE^*_{bottom} was significantly higher than ΔE^*_{top} only in the microfilled Heliomolar Radiopaque, even after additional photopolymerization. The composition and the optical properties of the material account for the depth of cure[46,47]. It has been shown that the light is transmitted more through hybrid than through microfilled resin composites[48]. Equal colour changes between the top and 2 mm-depth surfaces measured for most of the materials tested, impose that refinishing of the outer surface of the restoration cannot reverse the initial colour of the material.

CONCLUSIONS

According to the results of this study it can be concluded that:

- Aging of the materials under UV light/water conditions facilitates higher colour changes compared to the dark/water analogues.
- Prolonged photoaging led to higher colour changes.
- The polyacid-modified resin composite was the least colour stable of all the materials and the microfilled was less stable than the hybrid resin composite.
- Additional thermo-photopolymerisation decreased significantly the colour change of Heliomolar Radiopaque and Prodigy Condensable.
- Equal colour changes were detected between the surface and the in-depth layer (2 mm) of the materials. Higher in-depth colour changes were recorded only for Heliomolar Radiopaque with and without additional polymerisation.

MANUFACTURERS DETAILS

- Heliomolar Radiopaque, Vivadent, Ets., Liechtenstein
- Prodigy Condensable, Kerr/Sybron Co., Romulus, MI, USA
- Belle Glass HP, SDS-Kerr, Orange, CA, USA
- Compoglass, Vivadent, Ets., Liechtenstein
- Elipar Highlight, 3M Espe, USA.
- DI–500 light/heat curing oven, Coltene, Germany
- Teklite light-curing apparatus, Belle Glass, SDS-Kerr, Orange, CA, USA
- Belle Glass HP curing unit, Belle Glass, SDS-Kerr, Orange, CA, USA
- SUNTEST CPS+, ATLAS weathering apparatus, Material testing Technology BV, Gelnhausen
- Dr Lance Micro Color colorimeter, Braive Instruments, Belgium
- SPSS (ver 11) for windows, SPSS Inc, Chicago, IL, USA

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