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Colour Stability, Opacity and Cross-Link Density of Composites Submitted to Accelerated Artificial Aging

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The study evaluated the influence of accelerated artificial aging on colour stability, opacity and cross-link density of resin-based composites (RBCs). Seven specimens were obtained of five RBCs (Heliomolar, 4 Seasons, Tetric Evo Ceram, SR Adoro), which were submitted to colour stability and opacity analysis and cross-link density evaluation. All tests were performed before and after aging. After statistical analysis (one-way ANOVA; Tukey; $p<0.05$), it was observed that QuiXfil and SR Adoro presented colour alteration values above those that are clinically acceptable ($\Delta E=5.77$ and 4.34 respectively) and the variation in opacity was lowest for SR Adoro. There was an increase in the cross-link density of all studied materials after aging.

KEY WORDS: Composite, Colour stability, Opacity, Cross-links, Aging.

INTRODUCTION

The aesthetic challenge facing restorative materials is to reproduce the functional characteristics of natural dentition. Therefore, restorations must imitate the optical properties of teeth. Unfortunately, in spite of recent advances and improvements in restorative materials, colour stability continues to be a problem. Failure in aesthetics is one of the commonest reasons for re-intervention[1].

There are many factors that influence the colour stability of light-activated resin-based composites (RBCs), such as the photo-initiation system, the resin matrix, the type of filler particle used, the type of light curing unit used for activation[2] and the exposure time[3,4]. According to Hörsted-Binslev and Mjör[5], RBC discoloration can occur in three ways: I) external discolorations due to plaque accumulation and stains; II) surface alterations with slight penetration and reaction of colouring agents with the surface layer of resin composite (adsorption); or III) intrinsic discolorations due to physical-chemical reactions in the deep portions of the restoration.

The formulation of RBCs, and the characteristics of the particles have a direct impact on the resin surface smoothness[6,7] and consequently on its susceptibility to extrinsic staining[8]. The conversion degree and its chemical characteristics play an important role in the appearance of stains on the RBC surface, and when this is insufficient, it foments the adsorption of some colouring substances and inhibition of polymerization by the oxygen of the restoration surface[9]. The formation of cross-links also plays an important role in the physical and mechanical properties of dental RBCs. Inadequate formation of these cross-links can result in a RBC with deficient physical-mechanical properties, such as poor wear resistance and colour stability, adverse tissue reactions, increase in the water sorption rate, solubility and early failures in the restoration[10,11]. As regards internal discoloration of RBCs, these can be altered by aging, due to the various physical-chemical conditions such as visible light and ultraviolet irradiation, temperature and humidity[12,13].

The aim of this study was to evaluate the colour stability, opacity and cross-link density of different RBCs submitted to accelerated artificial aging (AAA). The hypothesis tested was that the AAA procedure alters the physical and chemical properties of RBCs, according to their composition, promoting colour alteration, increasing in their opacity and diminishing the cross-link density.

MATERIAL AND METHODS

Preparation of specimens

Five dental RBCs, A2 shade, were selected (Table 1). The specimens (n=7) were obtained using a Teflon matrix (12x2mm) by the incremental technique, with insertion of three increments of 2 mm. A glass slide was placed onto the last increment to press the material against the matrix to allow excess material to flow. Photoactivation was performed by a light-emitting diode curing unit (LED - 500mW/cm^2, wavelength in the range between 450 and 480nm), for 40 seconds, as recommended by the manufacturers. For SR Adoro system, cure was performed using Targis Power Upgrade, at 104ºC. After this, the surface test specimens were polished manually, with sand-paper disks assembled in micromotor, following a descending order of granulation. To prevent overheating and surface change, polishing was done in a wet environment and using soft alternate movements. All the test specimens were dry storage at 37ºC in the dark, until the initial colour, opacity and cross-link density evaluation.

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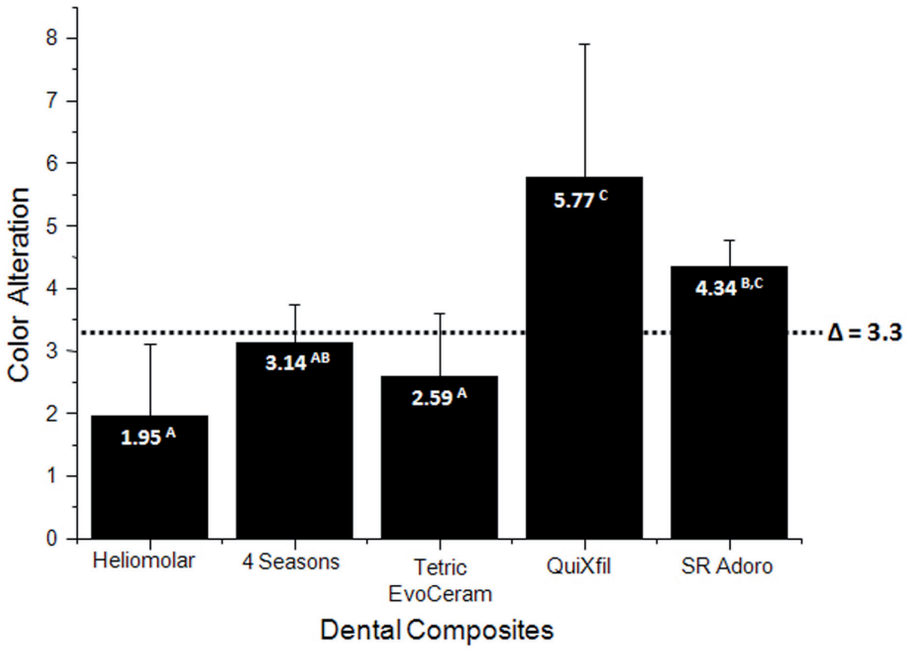

Figure 1 - *Mean Values of ΔE of the studied RBCs. Different letters indicate statistically significant difference (p<0.05)*

Table 1. RBCs used in the study

RBC	*Classification*	*Mean particle size*	*Composition (% by volume)*	*Manufacture/Lot*
Heliomolar	Microparticles	0.04 a 0.2 µm	Bis-GMA (14.3%); UDMA (4.4%); DM-DMA (3.3%); SiO2 highly dispersed (20.2%); Copolymers (47%); YbF3, (10.6%); Stabilizers, catalyzers and pigments (0.2%) Inorganic Load: 64%	Ivoclar Vivadent AG (Schaan, Liechtenstein) Lot: F57760
4 Seasons	Hybrid with fine particles	0.1 to 0.7 µm	Bis-GMA (14.3%); UDMA (4.4%); TEGDMA (3.3%); Silicone dioxide, highly dispersed (20.2%); Copolymers (47%); Ytterbium trifluoride, (10.6%); Stabilizers, catalyzers and pigments (0.2%) Inorganic Load: 55-58%	Ivoclar Vivadent Lot: G20541
Tetric EvoCeram	Microparticles	0.6 µm (0.04 – 3.0)	Bis-GMA (20%); Bis-EMA and UDMA (<20%); Silanized barium glass (30-40%); Ytterbium trifluoride (1-5%); Mixed spheroid oxides (5-10%) Copolymers (30-40%); Stabilizers, catalyzers and pigments Inorganic Load: 53-55%	Ivoclar Vivadent Lot: H29941
QuiXfil	Hybrid	1 and10 µm	UDMA; TEGDMA; di- and trimethacrylate resins; Dimethacrylate modified with carboxylic acid; BHT; UV Stabilizer; CQ; Ethyl-4-dimethylaminobenzoato; Silanized silicate glass, aluminum strontium fluoride phosphate sodium Inorganic Load: 66.4%	Dentsply Detrey GmbH (Konstanz, Germany) Lot: 0510000449
SR Adoro	Indirect Composite	1 and 10 µm	Bis-GMA (20-42%); TEGDMA (6-10%); UDMA (1-20%); DM-DMA (0-7%); SiO2 highly dispersed (19.8%); Copolymers (62.9%); Stabilizers and catalyzers (0.4%); pigments (0.1-0.3%) Inorganic Load: 46-47%	IvoclarVivadent Lot: H22136

Bis-GMA - bisphenol A-diglycidyl dimethacrylate; Bis-EMA - Bisphenol A dimethacrylate ethoxylate; UDMA - Urethane Dimethacrylate; TEGDMA - triethyleneglycol dimethacrylate; DM-DMA - Decamethyl dimethacrylate; YbF_3 - Ytterbium trifluoride ; BHT - Butylated hydroxytoluene; CQ - Camphoroquinone; SiO_2 - silicone dioxide; Ba - Barium.

Table 2. Mean Initial Opacity values (IO), Final Opacity (FO), and variation in opacity (ΔOP) of the studied RBCs

RBC	*IO*	*FO*	*ΔOP*
Heliomolar	96.82 (0.005)	97.70 (0.005)	0.88
4 Seasons	91.32 (0.010)	93.77 (0.005)	1.75
Tetric EvoCeram	90.65 (0.024)	92.40 (0.018)	2.45
QuiXfil	86.35 (0.005)	91.47 (0.015)	5.12
SR Adoro	95.15 (0.005)	95.78 (0.004)	0.63

Table 3. ΔKNH Values before and after AAA

RBC	*ΔKNH-Before*	*ΔKNH-After*
Heliomolar	54.3	4.1
4 Seasons	25.5	8.3
Tetric EvoCeram	30	19.4
QuiXfil	15	0.9
SR Adoro	16.2	9.4

Colour stability analysis

For colour measurements, a Spectrophotometer was used against a white standard background, as described by Pires-de-Souza *et al.*[2]. This system defines colour in terms of 3 coordinate values (L*, a*, b*), which locate the colour of an object within a 3-dimensional colour space. The L* coordinate represents the brightness of an object represented on the y axis, the a* value represents the red (positive x axis) or green (negative x axis) chroma, and the b* value represents the yellow (positive z axis) or blue (negative z axis) chroma[14].

After the initial colour measurement, the test specimens were submitted to AAA for 384 hours, in cycles of 4 hours exposure to Ultraviolet B irradiation (UV-B) at 50ºC and 4 hours of condensation at 50ºC. After this period, they were once again submitted to colour measurements, using the same method as previously described. The colour stability (ΔE) of the materials was calculated using the formula: $\mathbf{D}E^* = [(\mathbf{D}L^*)^2 + (\mathbf{D}a^*)^2 + (\mathbf{D}b^*)^2]^{½}$ where ΔE represents the colour change in all dimensions (L*a*b*) and **D**L*, **D**a* **D**b* represent colour changes along the individual axes[15]. Values of ΔE ≥ 3.3 were considered clinically unacceptable[16].

Opacity analysis

For opacity evaluation, the specimens were submitted to colour measurements against the white and black standard background, and only the L* coordinate was considered[17]. The opacity was calculated according to the formula: Opacity = L*p / L*b, where L*p corresponds to the reading of coordinate L* against the black background and L*b against the white background[18]. This procedure was performed before (Initial Opacity - IO) and after (Final Opacity - FO) AAA, from which it was possible to calculate, in percentage, the difference in opacity between the two periods.

Cross-link density

To measure the cross-link density, the specimens were submitted to the Knoop hardness test under a static vertical load of 500g applied for 15 seconds. Three readings for each specimen were taken and these were averaged to form a single value considered the initial microhardness value (KHN1). Next, the test specimens were stored in a 75% ethanol solution for 24 hours at ambient temperature. In this solution, the monomer is softened, with the linear polymers being softened in comparison with the cross-links[19]. After this period, new microhardness readings were taken (KNH2). The cross-link density was calculated, by the indirect method (ΔKNH) using the equation: ΔKNH = KNH1 – KNH2[20]. High ΔKNH values indicate low cross-link density.

As in the colour stability analysis, the opacity and cross-link density were measured before and after the action of AAA. All the values obtained were submitted to statistical analysis (one-way ANOVA - Tukey; 95% level of significance).

RESULTS

The colour stability measurements (Fig. 1) indicated that the RBCs Heliomolar, 4 Seasons and Tetric Evo Ceram presented colour change values (ΔE) within the clinically acceptable limits (ΔE<3.3), without statistically significant difference among them (p>0.05). However, the RBCs QuiXfil and SR Adoro presented values of ΔE>3.3, considered clinically unacceptable, differing statistically from the RBCs Heliomolar and Tetric Evo Ceram (P < 0.05) however, statistically similar among them (p>0.05). 4 Seasons presented statistically significant difference (p<0.05) in comparison with QuiXfil, which presented the highest ΔE values.

In Table 2, it was observed that the initial opacity (IO) and final opacity (FO) values, before and after AAA, follow a decreasing order as regards the RBCs: Heliomolar< SR Adoro< Tetric EvoCeram< 4 Seasons< QuiXfil.

The opacity alteration (ΔOP) was lower for the indirect RBC (SR Adoro) and a greater change occurred for QuiXfil, in spite of this RBC presenting lower final opacity in comparison with the others. As greater the opacity change (ΔOP), the more opaque the RBCs became after AAA, i.e., the aging procedure altered this optical property of the materials.

For cross-link density evaluation, the values of ΔKNH before and after AAA are described in Table 3.

These values are interpreted as indicating that the greater the numerical difference between ΔKNH "before" and ΔKNH "after" AAA, the greater was the action of this procedure on the cross-link density. The ΔKNH values "before" aging are always numerically higher than the ΔKNH values "after" aging for all the RBCs. Therefore, a lower cross-link density was observed in the non-aged samples in comparison with the samples that underwent the AAA process.

DISCUSSION

The manufacturers of esthetic restorative materials seek to provide dental RBCs with greater chemical stability, resulting in stability of the mechanical and optical properties. The latter characteristic will enable better esthetic results

with RBCs, such as, for example: colour stability, adequate translucence, opalescence and fluorescence[17,18].

Differences in the chemical structure of RBCs, such as the type of oligomers or monomers used; concentration/type of activators, initiators, inhibitors; double carbon bond oxidation; size/type of particles and particle/resin matrix bond system can interfere in colour stability[21]. According to some authors[22,23], dental RBCs with low concentration of load particles would present higher ΔE values. However, the results presented in the present study contradict this information, since the higher the percentage of load by weight in the RBCs, the greater was the colour alteration, results that corroborate the studies of Vichi *et al.*[15], according to whom, hybrid RBCs presented lower colour alteration when compared with the microhybrid and microparticle types, and the latter presented the greatest colour alteration among the previously mentioned RBCs.

In the present study, the threshold of clinical acceptance adopted for the esthetic restorative materials was ΔE<3.3; a value adopted by other authors[24]. Thus, all the ΔE values of RBCs QuiXfil and SR Adoro were higher than 3.3 indicating less colour stability when compared with the other RBCs, and according to the limit determined, this indicated replacement of the restoration for aesthetic reasons.

Dental literature indicates that RBCs allow solvents to penetrate the resinous matrix or at the matrix/particle interface[25]. According to Ferracane[26], as greater the volume of particles in the composition of the RBC, as lower the degree of conversion presented. Consequently the RBC formed will have a larger quantity of remaining double links and lower quantity of formed links. Therefore, this RBC will be more predisposed to the action of the solvent (water), as there will be a greater free volume for the action of the water, which will penetrate into the resinous matrix, causing "swelling" or relaxation of these links, in an effect known as plasticization[26]. The solvent inside the resinous matrix can cause it and the particle/matrix interface to deteriorate. This phenomenon can explain the greater colour alteration in the RBC after AAA[27].

As regards the composition of the resinous matrix, all the dental RBCs used in the present study presented Bis-GMA and UDMA in their composition. QuiXfil, 4 Seasons and SR Adoro also presented TEGDMA in their compositions. This monomer is more predisposed to water sorption than UDMA increasing the solubility of the polymer formed[28,29]. Greater solubility of RBCs in water provides them with lower colour stability, due to the increase in free volume of the polymer formed, and consequently, larger space for the water molecules to diffuse into the polymeric structure. Therefore, one can relate the greater colour alteration of the RBCs QuiXfil and SR Adoro.

The size of the inorganic particles is correlated with colour alteration, in which RBCs with large particles are more susceptible to water sorption and colour alteration. According to Kawaguchi *et al.*[29], hybrid RBCs present a lower coefficient of light transmission due to the various sizes of their particles. In the present study, the RBCs with larger sized particles were QuiXfil and SR Adoro (mean of 10μm) which could explain the greater colour alteration. The RBC Heliomolar presented the smallest particle size among those studied (0.04 to 0.2μm), thus presenting lower colour alteration.

According to Johnston and Reisbick[30], in the majority of dental RBCs, relatively high variations in translucence are found after AAA. This can be explained, as the scattering of incident light in the sample is influenced by the particle size of the dental RBC[29]. The larger-sized load particles make the dispersion of light by the dental RBC difficult, resulting in greater opacity[16]. Microparticle RBCs present greater light scattering than hybrid RBCs, that is to say, less light passes through the material due to greater reflection, which justifies the higher initial and final opacity values for Heliomolar in comparison to Tetric EvoCeram and 4 Seasons[29].

As regards cross-link density, the conversion of methacrylate monomers of the RBCs during and after polymerization does not occur completely, and free double carbon bonds remain which interfere in the density of cross-links through the reaction of free radical propagation[20]. When this radical reacts with a free double link of its own chain, linear chains are formed. When this link is to a different chain, the cross link occurs[20]. When the RBC is immersed in an ethanol solution, there is softening of the linear polymers, due to the solvent/polymer interaction. The solvents form secondary links with the polymeric chain, penetrating and replacing the secondary inter-chain links, and thus, dissolving the linear polymers. However, the solvent is not capable of acting on the cross-links. Thus, the polymers with high cross-link density are less soluble to the action of the solvent by presenting a smaller space between the polymer molecules, due to its reticular aspect, limiting diffusion of the solvent between its molecules[20]. Thus a high cross-link density indicates that the RBC is more resistant to degradation[26]. For the studied RBCs it was observed that the ΔKNH values were higher for the non aged samples, that is to say, an increase in the cross link density was observed in the aged samples. This suggests that AAA helped in the post-polymerization of these RBCs, and consequently there was greater conversion of the monomer, and increase in the density of the cross links. These data corroborate the study of Lee and Powers[23], who affirmed that the high temperature of AAA causes an increase in the degree of polymerization of RBCs.

From the above discussion and analysis of the results, the hypothesis of this study must be partially accepted, since the action of AAA altered the colour and increased the opacity of the studied RBCs. However, there was an increase in the cross-link density of the materials. Further studies are necessary in order to predict the optical properties of esthetic restorative materials over the course of time, remembering that in the oral environment, there are enzymes, such esterase, and other solvents that could cause greater deterioration of the resinous matrix, and consequently, alter the physical-mechanical properties of the dental RBCs [26].

CONCLUSIONS

AAA caused colour alteration in all the studied RBCs, whether discreet or clinically unacceptable (ΔE≥3.3). The opacity of the material is related to the size of the particles of which it is composed, so that RBCs with larger particles present greater variation in opacity than materials with microparticles. AAA produced an increase in cross-link density in the polymers of the studied RBCs.

MANUFACTURERS DETAILS

- LED (Ultraled XP, Dabi Atlante, Ribeirao Preto, Sao Paulo, Brazil)
- Targis Power Upgrade (Ivoclar/Vivadent AG, Schaan, Liechtenstein)
- Sand-paper disks (Sof-Lex, 3M Dental Products, St. Paul, MN, USA)
- Micromotor (N270, Dabi Atlante, Ribeirão Preto, SP, Brazil)
- Spectrophotometer (PCB 6807, Byk Gardner, Geretsried, Germany, CIE L*a*b* system)
- White standard background (White Standard for 45º/0o Reflectance and Color Gardner Laboratory Inc., Bethesda, MD, USA)
- Artificial Accelerated Aging Machine (C-UV Adexim Comexim, Sao Paulo, SP, Brazil)
- White and Black standard background (White and Black Standard for 45º/0o Reflectance and Color Gardner Laboratory Inc., Bethesda, MD, USA)
- Knoop Microhardness Machine (Shimadzu HMV-2 Series, Shimadzu Corporation, Kyoto, Japan)
- 75% Ethanol olution (BMCiclo Álcool Hidratado, Ciclofarma, Serrana, SP, Brazil)

ADDRESS FOR CORRESPONDENCE

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