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Color stability of silorane-based composites submitted to accelerated artificial ageing—An *in situ* study

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ABSTRACT

Objectives: To assess the *in situ* color stability, surface and the tooth/restoration interface degradation of a silorane-based composite (P90, 3M ESPE) after accelerated artificial ageing (AAA), in comparison with other dimethacrylate monomer-based composites (Z250/Z350, 3M ESPE and Esthet-X, Dentsply).

Methods: Class V cavities (25 mm² × 2 mm deep) were prepared in 48 bovine incisors, which were randomly allocated into 4 groups of 12 specimens each, according to the type of restorative material used. After polishing, 10 specimens were submitted to initial color readings (Easyshade, Vita) and 2 to analysis by scanning electronic microscopy (SEM). Afterwards, the teeth were submitted to AAA for 384 h, which corresponds to 1 year of clinical use, after which new color readings and microscopic images were obtained. The values obtained for the color analysis were submitted to statistical analysis (1-way ANOVA, Tukey, $p < 0.05$).

Results: With regard to color stability, it was verified that all the composites showed color alteration above the clinically acceptable levels ($\Delta E \geq 3.3$), and that the silorane-based composite showed higher ΔE (18.6), with a statistically significant difference in comparison with the other composites ($p < 0.05$). The SEM images showed small alterations for the dimethacrylate-based composites after AAA and extensive degradation for the silorane-based composite with a rupture at the interface between the matrix/particle.

Conclusion: It may be concluded that the silorane-based composite underwent greater alteration with regard to color stability and greater surface and tooth/restoration interface degradation after AAA.

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1. Introduction

Since their appearance, composites are obtained from the mixture of several types of dimethacrylate monomers (Bis-GMA, Bis-EMA and UDMA) and different types and sizes of load particles, which are incorporated to increase their physico-

mechanical properties. With the purpose of improving the clinical performance of these materials, new types of monomers, such as silorane, have been developed and introduced onto the market with the objective of presenting less polymerization shrinkage and therefore, better properties.^{1–4} Silorane-based composites (SBC) are obtained from the reaction of oxirane and siloxane molecules that provide these

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materials with two advantages: low polymerization shrinkage due to the oxirane ring-opening polymerization reaction^{4,5} and increase of hydrophobicity due to the presence of siloxane promoting the insolubility of the material in the presence of oral fluids.⁶

The polymerization reaction of SBC occurs from the cationic epoxy ring opening reaction, which is a form of addition polymerization in which the terminal of the polymer acts as a reactive centre, whilst other cyclic monomers bond to form a larger polymer chain through ionic propagation.¹ The polymerization process is initiated with an electron donation helping the oxi-reduction mechanism and degrading the onium salt of an acid cation. After adding an oxirane monomer, the epoxy ring is opened to form a chain. This process is responsible for the lower polymerization shrinkage of the composite,¹ which may result in better adaptation of the restoration in the cavity preparation than is observed in methacrylate-based composites (MBC).⁷ Whilst the MBC show volumetric shrinkage values ranging from 2.3 to 3.0%,⁷ the SBC show a mean shrinkage of 0.9%, causing less stress on the cavity preparation walls and less cusp deformations caused by the distribution of residual stress during mastication.⁸ Preliminary studies demonstrated the mechanical efficiency of SBC, however, little is known about their optical behaviour, especially in relation to the longevity of restorations.^{9–11}

Considering that lower polymerization shrinkage of composites provides lower formation of tooth/restoration marginal gaps, a factor directly related to restoration staining,¹² the aim of this study was to compare color stability and surface and tooth/restoration interface degradation of SBC and MBC after accelerated artificial ageing (AAA). The hypothesis tested was that SBC would present better aesthetic and surface properties than MBC.

2. Materials and methods

Forty-eight healthy bovine incisors were selected and Class V cavity preparations were made on the vestibular face using diamond burs 1091 (KG Sorensen, São Paulo, SP, Brazil), at high speed (Silent—MRS 400, Dabi Atlante, Ribeirão Preto, SP, Brazil). To standardize the cavity dimensions, a 25 mm² black plastic guide was used, which was adapted on the centre of the vestibular tooth surface. To standardize the preparation depth, a small resin composite stop was made and placed at a distance of 2.0 mm from the cutter extremity. All margins of the cavity preparation were made on enamel and were not bevelled.

After the preparations, the teeth were randomly separated into 4 groups of 12 specimens, according to the material used for the restoration (Table 1); ten were submitted to color analysis ($n=10$) and two were submitted to scanning electronic microscopy (SEM) analysis of the material surface and the tooth/restoration interface ($n=2$), before and after accelerated artificial ageing (AAA).

Before restoration, the teeth to be restored with MBC were submitted to acid etching with 37% phosphoric acid (Condicionador Gel Dental, Dentsply, Petrópolis, RJ, Brazil) for 15 s, washing, removal of the excess humidity with absorbent paper, application of the adhesive system (Single Bond 2 3M

ESPE Dental Products, St Paul, MN, USA) and light activation (FlashLite 1401, Discus Dental, Culver City, CA, USA—light intensity ≥ 1100 mW/cm², wavelength between 460 and 480 nm) for 20 s. The teeth restored with SBC were submitted to the application of a self-etching primer recommended by the manufacturer, under agitation on the surface for 15 s. The active application of the primer on the dentine surface may be useful to remove the smear layer and increase solvent evaporation, allowing greater monomer penetration.^{13,14} After the primer was light activated (FlashLite 1401) for 10 s, the adhesive was applied and also light activated (FlashLite 1401) for 10 s.

The teeth were restored according to the incremental technique and the material was light activated for 40 s. The restorations were polished with abrasive disks in a decreasing order of abrasiveness (Sof-Lex, 3M do Brasil, Sumaré, SP, Brazil) with intermittent movements interspersed with surface wetting in order to prevent the occurrence of overheating and consequent alteration of the surface. Next, the teeth were stored in artificial saliva at 37 °C for 24 h and submitted to the first color readings (Easysshade, VITA Zahnfabrik, Bad Säckingen, Germany), according to the CIE Lab scale,¹⁵ with the specular component excluded, which simulates a measurement 45/0, standard illuminant D₆₅ and observer pattern of 2°. The specular component excluded is related to the color measurement on the sample surface to prevent the interference of surface brightness.^{15,16} Three color readings were made for each sample in the centre of the restoration.

Afterwards, the teeth were submitted to AAA (C-UV, Comexim Matérias Primas Ltda, São Paulo, SP, Brazil) that simulates intemperate conditions, and is capable of predicting the relative durability of the material, which is exposed to eight ultraviolet fluorescent light sources with emission concentrated in the UV-B region with irradiation concentrated at 280/320 nm, as in nature, and to steam condensation at a controlled temperature. The working program was set to 4 h of exposure to UV-B at 50 °C and 4 h of water condensation at 50 °C and the maximum ageing time was 384 h, which corresponds to 1 year of clinical use.¹⁷ Next, new color readings were made.

The color alteration was determined by the difference (ΔE) between the coordinates of the samples obtained before and after AAA, calculated by the formula $\Delta E^* = [(\Delta L^*)^2 + (-\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Values of $\Delta E \geq 3.3$ were considered clinically unacceptable.¹⁸

The Kolmogorov–Smirnov test was applied to test the normal distribution of data and the values of ΔE were submitted to statistical analysis (1-way ANOVA, Tukey, level of significance of 95%).

To assess the integrity of the composite surface and the tooth/restoration interface, impressions of the sections of the surfaces between the tooth and the restoration were obtained using addition silicone (Express, 3M ESPE, St. Paul, MN, USA). After polymerization of the material, replicas obtained with the epoxy resin (Buehler, Lake Bluff, IL, USA), prepared in accordance with the manufacturer's recommendations, were mounted on stubs and placed in silica gel desiccant for 1 week. Next, they were sputter coated with gold (300–500 Å) under high vacuum (Denton Vacuum, model Desk 11, Moorestown, NJ, USA) for 120 s. After, the samples were examined at 150×

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