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Evaluation of Sorption, Solubility and Staining of Universal and Silorane Resin-Based Composites

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Abstract - *Resin-based composite staining is a multifactorial phenomenon and can be caused by intrinsic and extrinsic factors. The purpose of this study was to compare staining, sorption and solubility of silorane resin-based and universal resin-based composites. Five different resin-based composites (4 Seasons, Charisma, Filtek Silorane, Filtek Supreme and Grandio) were tested. Twenty five specimens were prepared (10 mm diameter and 1.5 mm thick). To staining test, the specimens were divided into 3 groups (n=5): distilled water (control), coffee and red wine. The specimens were immersed in one of the solutions at 37ºC for 7 days. Using the values of L*, a*, b*, color variation (CIEDE2000) was determined. For sorption and solubility test, the specimens were divided into 2 groups (n=5): with previous desiccation (Group 1) and with no previous desiccation (Group 2). The methodology used for sorption and solubility test was based on ISO 4049:2000. The results presented no significant difference in staining between composites. In sorption and solubility test, Filtek Silorane presented the smallest values, followed by Grandio. Under tested experimental conditions, it is not possible to assert the dependence of staining in sorption that composites are undergone. There was no significant correlation between colour change and sorption values.*

KEYWORDS: resin-based composite, sorption, discoloration

INTRODUCTION

Resin-based composite is extensively used in dentistry as an aesthetic restorative material. The success of an aesthetic restoration depends on the correct choice of shade and the color stability of the material. Colour stability is an important factor for aesthetic restoration longevity. Resin-based composite staining is a multifactorial phenomenon and can be caused by intrinsic and extrinsic factors.

Internal colour changes depend on the composite photoinitiator system[1-3]. Intrinsic discoloration is permanent and is related to polymer quality, type and quantity of inorganic filler and type of accelerator added to the photoinitiator system[4] .

Extrinsic factors include staining through the adsorption and/or absorption of colorants, as a result of external sources of contamination[2]. Extrinsic staining depends on the individual's diet, hygiene and the chemical properties of the composite. Composition and volume of organic matrix, type and volume of filler particles, type of filler-matrix silanization and polishing are relevant factors in the composite susceptibility to staining[5, 6].

Currently, replacement of restorations is the main reason for performing direct restorations. Discoloration of the material is one of the main reasons for replacing aesthetic restorations[7-12]. Thus, researches about the properties of the composite related to staining is important to understand the process of color change and for the improvement of the restorative material.

Resin-based composite restorations are not stable after polymerization and constantly interact with the environment [13]. In the oral environment, composites absorb water and chemical substances and release components into the environment [14, 15]. Sorption in resin-based composite is a diffusion-controlled process that causes chemical degradation due to monomer release and filler-polymeric matrix debonding[16, 17]. The phenomena of sorption and solubility may serve as precursors to various physical and chemical properties that can produce deleterious effects on the structure and function of the polymeric material[18, 19] .

Various factors related to chemistry and structure of the polymeric networks are important in determining the extent to which the material will be affected by the aqueous environment. Chemical characteristics include hydrophilicity of the polymer and differences between the solubility parameters of the polymer and the solvent. Important structural parameters include the cross-linking density and porosity of the polymer network [19, 20]. There is a hypothesis that staining is related to water sorption[21, 22].

The most recent commercial composite are silorane resin-based. Some authors[23, 24] relate that this material is stable in aqueous environment. The experimental hypothesis was that if silorane resin-based composite presents low water sorption, it should be more color stable. The purpose of this study was to compare staining, sorption and solubility of silorane resin-based and universal resin-based composites.

MATERIALS AND METHODS

Specimen Preparation

In this study 5 different resin-based composites were tested (table 1). Twenty five specimens were prepared in a Teflon mold, 10 mm in diameter and 1.5 mm thick. A glass micro-

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Table 1. *Tested materials*

Composite	*Manufacturer*	*Photoactivation time*
4 seasons	Ivoclar Vivadent	20 s
Charisma	Heraeus Kulzer	20 s
Filtek Silorane	3M ESPE	40 s
Filtek Supreme	3M ESPE	20 s
Grandio	Voco	20 s

scope slide was placed on a flat surface with a polyester strip over it, and the Teflon mould was set on it. Resin-based composite was inserted in one increment and another polyester strip and glass microscope slide were set on it and pressed over the mould to obtain a flat surface. The composite was light polymerized with LED light-curing-unit (Elipar Freelight – 3M ESPE) for the time recommended from manufacturer (table 1). The light intensity was 650 mW/cm² and was verified with a radiometer before each specimen was made. Light activation was performed with the unit tip touching the glass microscope slide (1 mm thick) to standardize the light activation distance.

Staining and Colour Measurement

The specimens were immersed in distilled water at 37°C for 24 h. This storage is important to leach unreacted components in composites[25]. After this initial storage period, the specimens were polished under irrigation (Ecomet – Buehler – USA) on both surfaces, with #600 grit silicon carbide paper. The specimens were polished with a device that allows the abrasion to occur parallel to the surface. After polishing, the colour of the specimens was measured (M_1) with a spectrophotometer Cinta 10 UV (Visible Spectrometer, GBC, Australia) against a white background, illuminant D65 and by a 2° observer using CIELab colour space. Reflection values were recorded in the visible spectra range (380 – 780 nm) in increments of 10 nm. Three color measurements of each specimen were made and a mean value obtained.

The specimens were divided into 3 groups (n=5): distilled water (control), coffee (Nescafé Original, Nestlé, SP, Brazil) and red wine (13% vol alcohol - Santa Carolina, Reservado, Cabernet Sauvignon, D.O. Valle Central, Chile, 2009). The coffee was prepared with 3 g of powder in 100 ml of distilled water, according to the manufacturer's instructions. The specimens were immersed in one of the solutions at 37°C for 7 days[2, 26]. Ertas et al.[27] related that a 24-hour storage time simulates about one month of coffee consumption. After the staining period, the specimens were dried with tissue paper and were taken to the spectrophotometer for colour measurement (M_2). Using the values of L*, a*, b*, color variation (CIEDE2000) between 7-day immersion and baseline measurements was determined using the following equation:

$$\Delta E_{00} = \left\{\left[\frac{\Delta L'}{K_L S_L}\right]^2 + \left[\frac{\Delta C'}{K_C S_C}\right]^2 + \left[\frac{\Delta H'}{K_H S_H}\right]^2 + R^T \left[\frac{\Delta C'}{K_C S_C}\right] x \left[\frac{\Delta H'}{K_C S_C}\right]\right\}^{\frac{1}{2}}$$

where, $\Delta L'$, $\Delta C'$ and $\Delta H'$ are the differences in lightness, chroma and hue between two specimens being compared. SL, SC, and SH are the weighing functions for the lightness, chroma, and hue components, respectively. KL, KC, and KH are the parametric factors to be adjusted according to different viewing parameters. In this study, KL, KC, and KH were set to 1.[28]

Sorption and Solubility

The methodology used for sorption and solubility test was based on ISO 4049:2000 [29]. The specimens were divided into 2 groups (n=5): with previous desiccation (Group 1) and with no previous desiccation (Group 2). ISO 4049:2000 [29], recommends to desiccate the specimens to achieve a constant mass before submitting the discs to sorption. However, in clinical situation it's not a real condition. That's the reason the authors preferred to make sorption test on both ways.

In Group 1, after the specimens were made, they were transferred to a desiccator containing silicone gel and kept at 37° C. After 24 h, the specimens were repeatedly weighed in an analytical balance (Mettler-Toledo AB204) until a constant mass (m_1) was attained, i.e., specimen mass variation was less than ±0.1 mg.

After the specimens dried, two measurements were made: diameter and thickness. For the diameter, two measurements were made and a mean obtained. For the diameter, two measurements were made and a mean obtained. After this the area was calculated in square millimeters, from the mean diameter measurement, then using the mean thickness, the volume, V, was calculated in cubic millimeters.

Next, the specimens were separately immersed in distilled water at 37° C for 7 days. The volume of the solution was 10 ml per specimen. After the 7 days, the specimens were dried with absorbent paper and weighed. The value obtained was recorded as m_2.

After all the specimens were weighed, they were reconditioned at a constant mass in a desiccator by means of the previously described cycle. Thus, the mass obtained was recorded as m_3.

In Group 2, the specimens were weighed right after they were made and the volume of each specimen was calculated. The initial mass was recorded as m_1. All the sequence method was the same as Group 1.

Water Sorption

To calculate water sorption, W_{sp} (µg/mm³), the following expression was used for each specimen:

$$Wsp = \frac{m_2 - m_3}{V}$$

where:

m_2 is the mass of the specimen (µg) after immersion in distilled water

m_3 is the reconditioned mass of the specimen (µg)

V is the volume of the specimen (mm³)

Solubility

To calculate the solubility, W_{sl} (µg/mm³) the following expression was used for each specimen:

$$Wsl = \frac{m_1 - m_3}{V}$$

where:

m_1 is the initial mass (µg) before immersion in distilled water

m_3 is the reconditioned mass of the specimen (µg)

V is the volume of the specimen (mm^3)

Statistical analysis

The means and standard deviations of colour change, sorption and solubility were calculated and submitted to statistical analysis. Data of staining test were submitted to one-way ANOVA and followed by Tukey test. For sorption and solubility test, for both groups, it was applied two-way ANOVA followed by Tukey test. Pearson correlation test was applied for the values obtained from staining, sorption and solubility tests.

Table 2. *Means of colour difference (ΔE_{00}) in staining test*

Composite	*N*	*Subset 1*
Grandio	15	5.3169
4 seasons	15	5.3807
Filtek supreme	15	5.5006
Filtek silorane	15	5.7360
Charisma	15	6.1770
Sig.		.137

Table 3. *Means of composites colour difference (ΔE_{00}) in different media. Values in distinct subsets are significantly different (p = 0.05).*

Media	*N*	*Subset*		
		1	*2*	*3*
Control	25	2.7987		
Red Wine	25		6.0344	
Coffee	25			8.0336

RESULTS

One-way ANOVA revealed no significant difference in staining test between the materials evaluated (table 2). Among tested drinks, coffee showed higher color change than red wine (table 3). The authors compared L* and a*b* values, separately, and the results demonstrate Filtek Supreme had higher color stability in both comparisons.

For sorption and solubility tests, Groups 1 and 2 were evaluated separately. Comparing both groups, the specimens in Group 2 presented less sorption than Group 1, in all tested resin-based composites. Comparing the materials, Filtek Silorane presented significantly less water sorption, followed by Grandio. The other three composites (Filtek Supreme, 4 Seasons and Charisma) have not presented significant difference between them and showed higher sorption. Solubility of the materials presented similar behaviour, with the exception that there was no significant difference between Grandio and Filtek Supreme (table 4).

Pearson correlation test revealed strong correlation (r = 0.872) between sorption and solubility. Comparing Groups 1 and 2, there was correlation (r = 0.813) of values of sorption test. In the comparison of discoloration and sorption, there was no significant correlation (r = -0.004).

DISCUSSION

Colour stability is an important factor for the lasting of aesthetic restorations. To be considered as clinically acceptable, the composites must not only provide an initial shade match, but also maintain an aesthetic appearance over the years in the restored tooth. Therefore, stain ability may be considered as a significant criterion in the selection of a material for use in an aesthetically critical area[30]. The replacement of anterior restorations is caused mainly from discoloration of composites[31, 32]. Factors related to composite staining must be studied to search alternatives for material improvement.

Discoloration can be caused by intrinsic or extrinsic factors. Extrinsic factors are directly related to the patient's diet[33, 34]. The impact of a beverage on composite properties can be related to quantity and frequency of ingesting it[33-35].

Table 4. *Means of composites in sorption and solubility test (µg/mm3). Values in distinct subsets are significantly different (p = 0.05).*

Composite	*N*	*Subset*					
		1		*2*		*3*	
		Sorption	*Solubility*	*Sorption*	*Solubility*	*Sorption*	*Solubility*
Filtek Silorane	10	0.8110	-0.2605				
Grandio	10			1.3117	0.5507		
Filtek Supreme	10				0.7810	2.0083	
4 Seasons	10					2.0450	1.1999
Charisma	10					2.2620	0.504

In this study, the authors tested coffee and red wine because they are frequently consumed beverages.

In this investigation it was tested universal resin-based composites and a silorane resin-based composite. Filtek Silorane is based on a new monomer silorane synthesized from hydrophobic siloxane and low shrinkage ring-opening oxirane[36]. The results of sorption and solubility test of Filtek Silorane are in accordance with literature[23], showing the minor values in comparison with other tested materials.

The present study addressed the problem of color change susceptibility of resin-based composites. Some studies[21, 22, 37] relate the correlation between sorption and color stability. However, in this investigation, there was no significant correlation. Filtek Silorane presented significantly less sorption than the other tested materials. However, there was no significant difference in staining between them. Thus, the experimental hypothesis was rejected. Um & Ruyter[2] discussed about the relationship between sorption and discoloration. Nevertheless, sorption may not be the main factor related to composite staining.

CONCLUSIONS

Under tested experimental conditions, it is not possible to assert the dependence of staining in sorption that composites are undergone. There was no significant correlation between color change and sorption values. Filtek Silorane presented the smallest values of sorption and solubility, however color change values was not significantly different from other tested materials. More studies should be undertaken to improve understanding of composite discoloration phenomenon.

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