Research Article

Effect of Light Curing Tip Distance and Immersion Media on the Degree of Conversion, Sorption and Solubility of Methacylate and Silorane-Based Composites

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Abstract

The purpose of this study in vitro was evaluate the degree of conversion (DC%), sorption (Sp) and solubility (Sl) of a methacrylate composite (Filtek™ Z250) and silorane-based composite (Filtek™ P90) cured in three curing tip distances (0 mm, 3 mm and 6 mm) with LED of second generation and immersed in artificial saliva (AS), Plax FeshMint (PFM), Plax Alcohol-Free (PAF) and Plax Whitening (PW). The methodology was based on the ISO 4049/2009 standard and five repetitions (n=5) were made for each group. Specimens (6mm in diameter and 1mm thick) were prepared and initially the degree of conversion (DC%) of discs was obtained by using an FT-IR spectrometer. After, the specimens were placed in a desiccator at 37°C and to be weighed until a constant mass (m_1) was obtained. The discs were immersed separately into the 4 media for 30 days. Twice a day, for two minutes, the samples were immersed in 2 ml of three mouthrinses. A control group was maintained in artificial saliva. After the period the discs was reweighed (m_2). The reconditioning in the desiccator was done until a constant mass (m_3) was obtained. The dates of DC% were analyzed by analysis of variance (ANOVA) in split plots and methacrylate-based composite showed significant higher DC than silorane-based composite and there is no statistical difference in DC between top and bottom surfaces. The sorption data were submitted to three-way ANOVA and Tukey's Test at a 5% level of significance and the rate of sorption-based composite silorane (P90) was significantly lower than the methacrylate-based composite (Z250). Moreover, the artificial saliva did not affect the increase in rate of sorption in any of the photoactivation distances. In 3 mm and 6 mm light distance, the mouthwash Plax Whitening caused the increase of sorption for the two composites. The solubility date were analyzed by Kruskal-Wallis's Test (p=0,05) and Dunn's Test, since a non-normal date distribution was observed. The values were negative, which means that there was mass gain. It may be concluded that the curing tip distance affect the degree of conversion and sorption only when cured to 6mm, however not influenced by the type of mouthwash. The solubility test demonstrated negative data, masking the real solubility..

INTRODUCTION

Today, resin-based composites are widely used as restorative material. Most of the available composites contain a polymeric matrix of dimethacrylate monomers, inorganic filler particles coated with a methyl methacrylate-functional silane coupling agent to bond the filler to the organic matrix, and a photoinitiator system to permit photoactivation by light units [1,2] [1Bis-GMA is the monomer most used in dental composites, however due to its high molecular weight, high viscosity, and low mobility others monomers with lower viscosity and/or higher mobility, as TEGDMA and UDMA, are used to increase the degree of conversion (DC) and crosslinking of the polymer resultant [3].

During the polymerization reaction, resin-based composites are transformed from a viscous to a rigid state by a free radical polymerization of the dimethacrylate monomers present in their polymeric matrixes. This reaction involves the rupture of the C=C aliphatic bonds, the reduction of intermolecular distances of 0.3–0.4 nm between polymer chains, maintained by Van der Waals attraction forces, and the establishment of primary C-C covalent bonds with lengths of about 0.15 nm [4,5].

Methacrylate restorative materials exhibit volumetric polymerization shrinkage[5] and a significant proportion of unreacted monomer due the incomplete C=C bond conversion [6,7]. Thus, the study of others physical properties together with

Cite this article: de Azevedo Miranda D, Pini NP, Bovi Ambrosano GM, Baggio Aguiar FH, Leite Lima DAN, et al. (2013) Effect of Light Curing Tip Distance and Immersion Media on the Degree of Conversion, Sorption and Solubility of Methacylate and Silorane-Based Composites. JSM Dent 1(2): 1013.

DC measurement are better to knowledge of the performance of dental materials [8,9]. However, there are many factors that affect the amount of light energy received at the top and bottom surfaces of a resin composite restoration, resulting in ineffective polymerization. These include: the design and size of the light guide; distance of the light guide tip from the dental composite; power density; exposure duration; shade [8] and opacity of the dental composite; increment thickness and material composition [10-12].

In the attempt to reduce these problems some techniques were proposed to decrease the shrinkage stress effects such as different incremental composite placement [13], light-curing protocols [14], and intermediate layer with hybrid glass ionomer and flowable composite [15] and materials with a low shrinkage monomer [16]. The silorene-based composites showed molecules presents a siloxane core with four oxirane rings attached that open upon polymerization to bond to other monomers [17]. The oxirane ring opening causes a volumetric expansion that partially compensates the shrinkage resultant from molecular bonding. Literature data confirmed that a silorane-based commercial composite presented less than 1.0% of total volumetric shrinkage, compared to 2.0–3.5% for Bis-GMA-based composites [17,18], causing less tooth deflection [13] and microleakage [19,20]. Its mechanical properties are comparable to those of dimethacrylate-based materials [21,22].

In deep cavities the reduction of the light intensity reaches the material due the distance between guide tip and resinous material surface is one factor that cannot be controlled by dentist during the restorative procedure. Thus, the restoration weakening can occur by lower DC and/or formation of more linear polymers, presenting inferior physical properties, such as reduced hardness, increased sorption, solubility [23], and discoloration [8]. The increase of the curing time and proximity of the tip of the device, increase the irradiance and consequently the energy density available for the monomer conversion can improve the physical properties of resin-based materials [7,24], and thus increase the long-term durability of adhesive restorations [25].

The DC is an important physical property that may have some impact on the restoration longevity, but this property alone is not enough to characterize the 3-dimensional dental composite structure, whereas different C=C bond concentrations coexist in the same polymer [26]. The same DC% value may result in different linear polymer content, which is more susceptible of softening than a more cross-linked polymer [6,27].

Miranda et al. [23] have investigated the sorption and solubility phenomena in nanofilled composites when immersed in mouthwashes contains alcohol, hydrogen peroxide and alcohol-free however due to changes in monomer composition becomes important to evaluate the effect of these solvents on matrices of microhybrid composites. The main purpose of this study was to evaluate the effect of light curing tip distance on the DC, sorption, and solubility of methacrylate and silorane-based composite resins after immersion in different solutions. The hypotheses tested were that: (1) there would be no difference between the materials and (2) the greatest distance from the tip distance of surface radiated decreases the physical properties

tested. (3) There would a difference among solvents tested in the sorption and solubility.

METHODS AND MATERIALS

Two resins composites with different monomer compositions were analyzed: methacrylate-based composite (Filtek Z250, 3M ESPE, St Paul, MN, USA) and silorane-based composite (Filtek P90, 3M ESPE, St Paul, MN, USA). The composition of the composites is described in (Table 1). Three mouth rinses were chosen: Plax Alcohol Free (Colgate-Palmolive), Plax Fresh Mint (Colgate-Palmolive) and Plax Whitening (Colgate-Palmolive). Artificial saliva (Proderma) was used as the control. The composition and characteristics of the solutions are described in (Table 2).

PREPARATION OF SPECIMENS

Cylindrical specimens were prepared in teflon ring molds (6.0 mm Ø and 1 mm height). A polyester strip was placed on a glass slab (Dentsply, Petropólis, RJ, Brazil) and teflon matrix. After filling the mold to excess, the material surface was covered with another polyester strip and a glass slide, and compressed with a device (500 g) for 20 s to accommodate the composite better and remove the excess material. All the composite specimens were light activated with an LED set (Flash Lite 1401, Discus Dental) for 20 s. Three curing tip distances were used in this study: 0 mm,

Table 1: Type, manufacture and composition the composites analyzed in this study.

Composite	Manufacture	Composition*
Filtek Z250 shade A2	3M ESPE(St Paul, MN, USA)	Filler: 60 vol%, aluminum oxide, silica, and zirconium oxide (0.01-3.5 μm). Resin: bisphenol-A glycidyl dimethacrylate (Bis-GMA), ethoxylated bisphenol A dimetacrylate (Bis-EMA), and urethane dimethacrylate (UDMA).
Filtek P90 shade A2	3M ESPE(St Paul, MN, USA)	Filler: 55 vol%, silica, and yttrium trifluoride (0.04-1.7 μm). Resin: Bis-3,4-Epoxycyclohexylethyl-Phenyl-Methylsilane and 3,4-Epoxycyclohexylcyclopolymethylsiloxane.

*According to manufacturers.

Table 2: Solution, manufacture, pH and composition of the media immersions analyzed in this study.

Solution	Manufacture	pH	Composition*
Artificial Saliva (AS)	Proderma	6,9	KCl, NaCl, MgCl, CaCl, nipacin, carboxymethyl, cellulose, sorbitol and deionized water
Plax Fresh Mint (PFM)	Colgate-Palmolive	6,6	Water, Sorbitol, Alcohol, Glycerin, Sodium, Lauryl Sulfate, Sodium Methyl Cocoyl Taurate, PVM/MA Copolymer, Aroma, Disodium Phosphate, Sodium Fluoride, Sodium Hydroxide, Triclosan, Sodium Saccharin, Cl 47005, Cl 42090
Plax Alcohol Free (PAF)	Colgate-Palmolive	4,9	Water, Glycerin, Propylene Glycol, Sorbitol, PEG-40 Hydrogenated Castor Oil, Sodium Benzoate, Aroma, Phosforic Acid, Sodium Fluoride, Cetylpyridinium Chloride, Sodium Saccharin.
Plax Whitening (PW)	Colgate-Palmolive	3,8	Water, Sorbitol, Ethylic Alcohol (8%), Hydrogen peroxide 1,5%, Polaxamere 338, Polissorbate 20, Methyl saliciltate, Menthol, Sodium saccharine, Cl 42090

*According to manufacturers.

To measure the pH, 20 mL of each substance was added to a beaker, and the pH was obtained with a glass pH electrode 1.5 cm in diameter (PROCYON model AS 720 (Procyon Instrumental Científica) and each value is described in Table 2.

3 mm and 6 mm (Figure 1). The polymerization was performed with the light tip positioned in a device at a distance from the top surface of the sample that was controlled by an electronic digital caliper (Mitutoyo Sul Americana, Suzano, SP, Brazil). The optical power (mW) delivered by the device was measured with a power meter (Ophir Optronics, Har-Hotzvim, Jerusalem, Israel). The tip diameter was measured with digital caliper (Mitutoyo Sul Americana, Suzano, SP, Brazil) to determine tip area (cm²). Irradiance (mW/cm²) was calculated dividing optical power by tip area. The irradiance in each group were cured was determined by the ratio between power and area of the tip of the device, expressed in mW/cm² (Figure 2). The measured values of irradiance and energy density are described in (Table 3).

DEGREE OF CONVERSION (DC%)

After polymerization, the specimens were removed from the matrices, dry stored in light proof containers at 37º C for 24 h, and polished with 1200-grit silicon carbide (SiC) grinding paper (CarbiMet 2 Abrasive Discs, Buehler, Lake Bluff, IL, USA). The degree of conversion assessments was recorded in the absorbance mode using a Fourier transform infrared (FTIR) spectrometer model (Spectrum 100 FTIR/ UATR, Perkin Elmer) equipped with an attenuated total reflectance crystal (Figure 3a). Standard increments of each resin composite were compressed between two polyethylene strips and two glass slides to produce
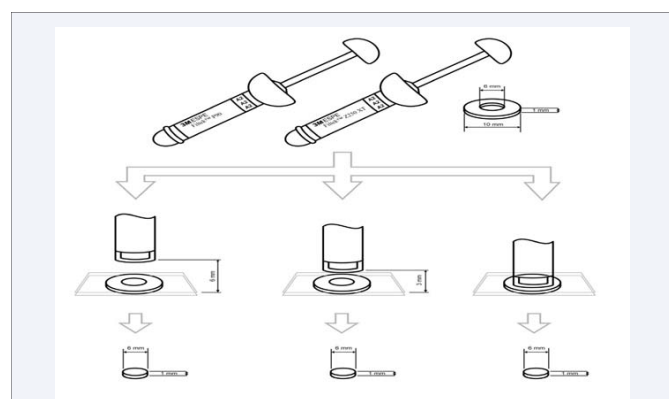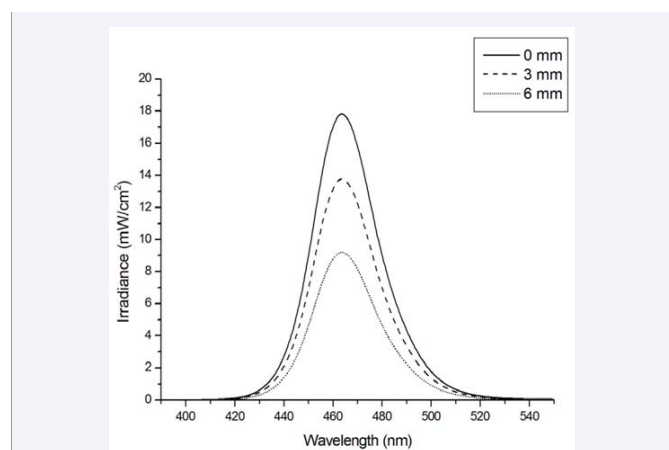

Figure 1 Preparation of specimens.

Figure 2 The irradiance for each distance of photoactivaton.

Table 3: Power, irradiance and energy density of the LED Flash Lite 1401.

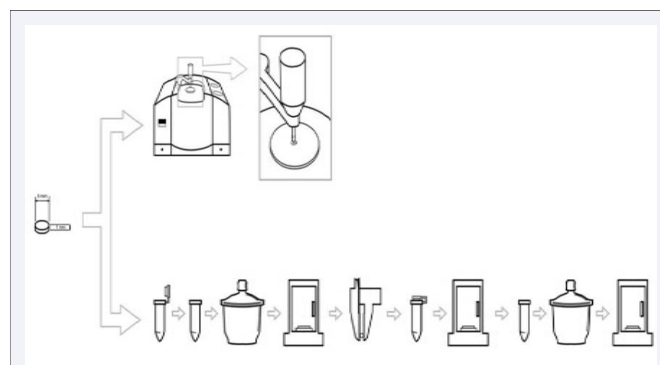| Tip of Distance	Power (mW)	Area (cm²)	Irradiance (mW/ cm²)	Energy Density (J)
0mm	310	0,5024	618	12,36
3mm	240	0,5024	478	9,56
6mm	160	0,5024	319	6,38

Figure 3 (a) - Spectra of the unpolymerized and polymerized specimens were recorded with an FTIR spectrometer model Spectrum 100 FTIR/UATR / 3 (b) - The discs were placed in a desiccator containing freshly dried silica gel and transferred to an oven at 37°C. After 24 hours, the discs were repeatedly weighed on an analytical balance, until a constant mass (m1) was attained. After final drying, the thickness and diameter of the discs were measured at four points with a digital and the volume (V) was calculated in mm3. The discs were then individually placed in plastic vials and immersed in 1 ml of artificial saliva at 37°C. Twice a day, the discs were immersed in 2 ml of each mouthwash for two minutes (12 hour intervals) and agitation in orbital rotational table and immersion in the respective mouthwashes, the discs were immersed in artificial saliva. After thirty days, the discs were removed from the vials, washed in distilled water and weighed (m2). The discs were then placed in a desiccator and weighed daily until the mass variation was less than 0.1 mg (m3).

a thin film. Five specimens were prepared for each distance. Absorption spectra of the cured and uncured composites were obtained in the top and bottom surfaces with 16 scans at 4 cm⁻¹ resolution in the region between 1000-2000 and 600-2000 cm⁻¹ for methacrylate and silorane-based resins, respectively. To calculate the DC the ratio (R) between the peak heights of the C=C aliphatic (1638 cm⁻¹) and aromatic (1608 cm⁻¹) for methacrylate, and oxirane (C-O-C) - 770 cm⁻¹- and siloxane (C-H) - 1038 cm⁻¹- for silorane band absorptions for cured and uncured composite was used. The formula: DC (%) = [1 – (R polymer/R monomer)] x 100 was used to calculate the DC.

SORPTION (SP) AND SOLUBILITY (SL)

The specimens were stored in desiccator at 37 ºC containing silica gel and daily weighted in an analytical balance (Tel Marke, Bel Quimis, São Paulo, SP, Brazil) accurate to 0.001 mg, constituting a weighing cycle every 24 h. The complete cycle was repeated until a constant mass (m1) was obtained until that the mass loss of each specimen was not more than 0.1 mg per 24 h cycle. Thickness (4 measurements at four equally points on the circumference) and diameter (2 measurements at the right angles) of each specimen were measured using a digital electronic caliper. Mean values were used to calculate the volume (v) of each specimen (mm³). The discs were then individually

placed in plastic vials and immersed in 1 ml of artificial saliva at 37°C. Twice a day, the discs were immersed in 2 ml of each mouthrinse for two minutes (12 hr intervals) and agitation in orbital rotational table (MA 140 CFT, Marconi, Piracicaba). After immersion in the respective mouthrinses, the discs were immersed in artificial saliva. A control group was kept in artificial saliva at 37°C throughout the entire experiment. After 30 days, the discs were removed from the vials, washed in distilled water and weighed (m_2). The discs were then placed in a desiccator and weighed daily until the mass variation was less than ± 0.1 mg (m_3) (Figure 3b). The sorption (Sp) and solubility (Sl) were obtained using the following formulas:

$$Sl = m_1 - m_3/V \quad Sp = m_2 - m_3/V$$

where m_1 is the conditioned mass prior to immersion in the solvent in µg; m_2 is the mass of the specimen after immersion in the solvent in µg; m_3 is the reconditioned mass in µg; V is the volume of the specimen in mm³. The units used in the formula were (µg/mm³).

After exploratory data analysis was applied to analysis of variance (ANOVA) in a split plot design to degree of conversion and in factorial for sorption, multiple comparisons were performed by Tukey's test. The solubility data did not meet the assumptions of ANOVA and were analyzed by the non-parametric Kruskal Wallis and Dunn's test. In all analyzes was considered the significance level of 5%.

RESULTS

Degree of Conversion (DC%)

(Table 4) shows the means and standard deviations of DC%. The ANOVA in the split plot showed an interaction between the surfaces and light curing tip distances (P = 0.03). The results showed that, regardless of the surface and the distance light activation, methacrylate showed significant higher DC than silorane-based composite. Furthermore, for two types of composites tested, there was only a difference in DC (top and bottom) when the samples were photoactivated for distance of 6mm. There was no statistical difference in DC between top and bottom surfaces.

Sorption

(Table 5) shows the means and standard deviations of sorption. For the sorption was found statistical difference between the variables: resin, distance of photoactivation and mouthrinses (p<0,05). The data showed that, regardless of the distance photoactivation and the immersion medium, the rate of sorption-based composite silorane (P90) was significantly lower than the methacrylate-based composite (Z250). Moreover, the artificial saliva did not affect the increase in rate of sorption in any of the photoactivation distances. In 3mm and 6mm light distance, the mouthwash Plax Whitening caused the increase of sorption for the two composites. For the composite Z250, the Plax Fresh Mint demonstrated the influence of sorption values only when the polymerization was carried out to 6mm; differently for composite P90, the same mouthwash already changed the rate of sorption when the polymerization was carried out to 3mm. When photoactivated to 6mm, all mouthwashes tested caused a

Tabela 4: Means and standard deviations of the degree of conversion in top and bottom surfaces.

Surface	Distance (mm)	Composite	
		Z250	P90
Bottom	0	58.88 (2.19) Aa	49,68 (2,61) Ba
	3	58.92 (3.08) Aa	49.83 (3.74) Ba
	6	55.70 (3.90) Ab	46.62 (3.00) Bb
Top	0	58.35 (1.64) Aa	51.11 (2.78) Ba
	3	58.93 (0.88) Aa	50.65 (4.78) Ba
	6	54.21 (1.42) Ab	47.96 (1.85) Bb

Means followed by distinct capital letters in the same row and distinct small letters in the same column are significantly different at P < 0.05.

Table 5: Means and standard deviations of solvent sorption in µg/mm³.

Composite	Solution	Distance (mm)		
		0	3	6
P90	AS	*9,60 (1,59) Aa	*10.23 (3.63) Aa	*10.83 (2.61) Aa
	PFM	*10.41 (1.54) Aa	*11.72 (3.02) Ab	*12.79 (1.32) Bb
	PAF	*10.33 (1.23) Aa	*10.71 (3.16) Aa	*12.16 (2.56) Ab
	PW	*13.97 (1.91) Ab	*14.05 (1.79) Bb	*16.46 (3.85) Cc
Z250	AS	20.24 (0.67) Aa	20.56 (1.36) Aa	20.53 (1.44) Aa
	PFM	21.56 (1.29) Aa	21.89 (1.30) Aa	23.53 (1.21) Bb
	PAF	20.72 (0.64) Aa	20.90 (1.38) Aa	23.44 (1.35) Bb
	PW	22.43 (1.38) Aa	23.85 (0.64) Bb	27.71 (1.34) Cc

Means followed by distinct capital letters in the same row and distinct small letters in the same column are significantly different at P < 0.05. * Differs the resin Z250 in the same solution and distance (p ≤ 0.05).

significant increase in the rate of sorption both in the artificial saliva.

Solubility

The data of (Table 6) showed median and maximum and minimum values of each solvent solubility. Although it is noticed statistical differences among the factors, negative values attributed indicate an inconclusive result.

DISCUSSION

The clinical performance of dental composites is to a great extent dependent on their mechanical properties and resistance to chemical degradation by acids and other organic substances found in the oral cavity [28,29]. A satisfactory degree of conversion of the composite may be associated with the polymer network, with different amounts of cross linking [30], which can be influenced by the distance between the tip end of the curing light and the composite surface [8,23].

The distance between the light source tip end and composite surface is another important factor to be considered. Clinical variations have showed that this distance directly affects light intensity that reaches the material [23,31] and thus interferes in the polymerization depth and degree of conversion[23]. Unlike previous studies [23], this study did not observe a statistical difference between the top surface and base for any of the composites tested. Furthermore, it can be noted that regardless of the composite when the polymerization was performed at 6

Table 6: Median and maximum and minimum values of solvent solubility in μg/mm^3.

Composite	Solution	Distance		
		0 mm	3 mm	6 mm
P90	AS	-4,73 (-7,95; -2,28) Aa	-5.32 (-9.90; -2.52) Aa	-4.49 (-7.90; -2.12) Ab
	PFM	-5.41 (-9.55; -2.60) Aa	-5.53 (-11.55; -2.42) Aa	-4.86 (-7.70; -2.53) Ab
	PAF	-5.03 (-7.88; -2.38) Aa	-2.63 (-10.05; 0.00) Aa	-2.64 (-4.98; 0.00) Aab
	PW	-2.30 (-2.72; 0.00) Aa	-6.09 (-8.98; -4.04) Ba	-2.40 (-2,46; 0.00;) Aa
Z250	AS	-5.52 (-8.16; -2.79) Aa	-5.47 (-9.03; 0.00) Aab	-5.23 (-9.22; -2.77) Aa
	PFM	-6.24 (-9.87; -2.59) Aa	-7.69 (-9.36; -5.57) Aab	-7.25 (-14.12; -2.79)Aa
	PAF	-5.13 (-8.39; 0.00) Aa	-2.96 (-6.35; -2.40) Aa	-2.49 (-11.57; 2.77) Aa
	PW	-2.78 (-8.72; 0.00) Aa	-9.25 (-14.78; -5.61) Ab	-4.60 (-12.09; -2.67)Aa

Means followed by distinct capital letters in the same row and distinct small letters in the same column are significantly different at P < 0.05.

mm, the DC was significantly lower (top and bottom). Although studies demonstrate that polymerization is a phenomenon not uniform [23,32], it is believed that the results obtained in this study are justified by the thickness of the specimen made (1mm). The literature has shown that larger increments than 2mm could favor differences in DC's top and bottom restoration.

Studies [23]. have shown that photoactivation at 6 mm from the tip end of the light curing unit to the composite surface led to a decrease of 50% in the amount of irradiance reaching the material's surface. Coincidentally, for the 6 mm distance, the irradiance observed was 319 mW/cm^2, which corresponds to about half of the irradiance for the 0 mm distance, which was 618 mW/cm^2. This light dispersion yielded to a loss of energy dose and probably promoted a lower camphorquinone excitation [32,33] and a polymer chain formation with lower crosslinks [30]. Thus, there will be more space for solvent molecules to diffuse inside the polymer network, making the polymer more susceptible to the plasticization effect of solvent [34].

Thus, the first hypothesis that both materials showed similar performance was rejected. According to results of this current investigation, higher DC was found for methacrylate compared to silorane composite [26]. Probably, the UDMA monomer contained in the Filtek Z250 composite gives more mobility and has been related to increase DC^3; and differences in the mechanism of the polymerization reaction can explain these results. Methacrylate is cured by radical intermediates and cycloaliphatic oxirane polymerizes via cationic intermediates [6]. Moreover, the onset of cationic ring-opening polymerization of the silorane is slower due to the necessary formation of sufficient cations to initiate the polymerization, thus more time of light-curing is required compared to radical cure of methacrylate monomers molecules into polymer network [5,13].

Another plausible explanation for the lower degree of conversion of the silorane-based composite that would the light-curing time recommended for silorane composite resin using quartz-tungsten-halogen (QTH) with irradiance between 500-1400 mW/cm^2 is 40 s, as well as LEDs with output between 500-1000 mW/cm^2. For LEDs with irradiance between 1000-1500 mW/cm^2 is indicated an exposure light time of 20 s. In this study, to maintain a standardized with the methacrylate-based composite was standardized time of 20 seconds using a second generation LED for each distance photoactivation tested. It

may have happened due to the short time of light curing for all specimens (20 s), which possibly resulted in a low energy dose and contributing to form a less established polymer network [32,35].

The data showed that, regardless of the distance photoactivation and the immersion medium, the rate of sorption-based composite silorane (P90) was significantly lower than the methacrylate-based composite (Z250) . This can be explained by the composition of the material and not by the DC. Moreover, this result showed that the polymeric structure formation of the silorane based composite may be different of the methacrylate one and it should be more elucidated in future studies. The monomers present in the polymeric matrices of the methacrylate-based composite (Filtek Z250) (Bis-GMA, Bis-EMA, TEGDMA, UDMA) have hydrophilic groups in their backbones, i.e., -OH-, >C=O, -O-, -NH-[36,37], which probably make them more prone to salivary sorption. In addition to the polymeric matrix, the filler particle system may also influence the sorption phenomenon in resin composites [2]. Besides that, silorane represents a merger of siloxane and oxirane species and reveals good biocompatibility even in uncured condition and high hydrophobicity and oxirane exhibits low shrinkage and high reactivity [22,38]. Thus, this more hydrophobic nature of the silorane may contribute for lower sorption comparable to conventional methacrylate resin-based.

Regardless of the type of composite, saliva did not influence the rate of sorption in no distance photoactivation. Thus, it is believed that, even with the possibility of different configurations crosslinking, the difference of the observed sorption 3 mm to 6 mm and PW is justified by its composition and pH. That is, the presence of hydrogen peroxide at low concentration (1.5%) decrease in pH favors the challenge solution (3,8%). , Regarding the effects of hydrogen peroxide in the composite matrix, it is known that acid solutions can change the composition of organic resinous material [39], due to the high oxidative power of hydrogen peroxide. Miranda et al. [23] showed that even in low concentrations, it is capable of causing harm to the links polymer, yielding this material to be more susceptible to softening and degradation.

Another important observation is that the 6mm, the UFW had similar behavior to PAF. Notably, (Table 2) shows that the pH of PAF is less than the PFM. Miranda et al. [23] verified the presence in Plax Alcohol Free of phosphoric acid (0.174 g / l), which, even

in small quantities, could reduce the pH of the solution and change the sorption phenomena and hygroscopic expansion, due to the presence of free hydrogen groups [40].

Almeida et al. [28] showed that low pH mouthrinses may have a role in the polymer composite resin through the catalysis of ester groups from dimethacrylate monomers present in their compositions (Bis-GMA, Bis- Ema, UDMA and TEGDMA) .The hydrolysis of these ester groups can form alcohol and carboxylic acid molecules, which can accelerate the degradation of the composite resin, due to the low pH inside their matrixes [28]. On the other hand, the low pH cause softening of the matrix surface by removing the monomer structure of the polymer [30], or can promote an opening in the structure of the polymer, facilitating saliva diffusion [28] and decreasing its physical properties [41,42] , accelerating the detachment or release of ions from its surface [34,43] .

The susceptibility of the more linear or less crosslink polymer to softening [26] in solvents may be explained by the solvent-polymer interaction and, consequently, by Hoy's solubility parameter for polar forces [44]. The solubility of the composite is strongly influenced by monomer conversion [45], and high hydrophobicity of the siloxane species may decrease the solubility of the silorane [26]. However, although it can be observed statistical differences in solubility values, negative values observed for all factors examined (composite, distance and solution) lead to a more rational conclusion that the phenomenon occurred; the composites tested were more susceptible to water sorption leading to mass gain [44], which could mask the real solubility [46]. It does not mean that no solubility occurred, but rather that the water sorption was greater than the solubility [47], because the final mass was greater than the original. Supposedly, some molecules of the solvent, the polymer chains are joined by hydrogen bonds, as occurs with water, and remained firmly adhered to the polar sites along the polymer network [46] preventing the removal of all the solvent during the dissection [48].

Although this study attempted to simulate cycles alternating between saliva and mouthwash, the chemical degradation of the restoration surface in vivo is the result of complex reactions with different chemicals components. Clinically, the mouthwashes' effects on composite resins may be different according to some factors, such as acquired film, food inhabits, beverages, and oral care products, which cannot be reproduced in vitro. Those factors, acting together or in isolation, may interfere with the physical and mechanical properties of the materials, influencing the durability of the restorative treatment [23,29].

CONCLUSIONS

Under the conditions of this in vitro study, it may be concluded that: (1) the composite metacrylate-based showed higher DC compared to the silorano-based composite; no statistical difference in DC from top and bottom. Only when the polymerization was carried out to 6mm, the DC was significantly lower for both composites. (2) the methacrylate-based composite had higher rates of sorption than the base composite silorano; saliva did not affect sorption, regardless of the distance and of the composite. The PW showed significantly change the sorption,

regardless of the type of composite; (3) the solubility test demonstrated negative data, masking the real solubility.

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