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Análise de propriedades químicas e mecânicas de diferentes resinas compostas quando mantidas em temperatura de refrigerador e ambiente

Governador Valadares
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Dissertação apresentada ao Programa de Pós-graduação em Ciências Aplicadas à Saúde, da Universidade Federal de Juiz de Fora, Campus Governador Valadares, como requisito parcial à obtenção do título de Mestre em Ciências Aplicadas à Saúde, área de concentração Biociências.

Orientador: Prof. Dr. Hugo Lemes Carlo

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Dedico este trabalho, com todo meu amor, à minha família,
que é motivo e razão de todas minhas lutas e esforços.
Sem vocês, nenhuma de minhas conquistas seriam possíveis.

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“Whether you think you can or think you can't,
you're right.” – **Henry Ford**.

RESUMO

As resinas compostas são amplamente utilizadas na prática odontológica para restaurar a forma, a função e a estética dos dentes. Elas podem ser manipuladas em diferentes temperaturas e devem resistir às condições da cavidade bucal. Este estudo “in vitro” avaliou o grau de conversão (FTIR/ATR), sorção e solubilidade (ISO 4049: 2000) e dureza superficial (Koop) de sete resinas compostas (Filtek Z100, Z250, 350, One Bulk Fill, Bulk Fill Flow, Universal e P60) polimerizadas em duas diferentes condições: 1 – temperatura: ambiente (25°C) ou refrigerada (5°C); e 2 – tempo de armazenamento: 7 dias e 30 dias. Para a obtenção das amostras foi utilizado um molde de silicone cilíndrico com dimensões internas de 6mm de diâmetro e 1mm de profundidade. As amostras foram polimerizadas por 20s utilizando-se um equipamento LED (VALO Cordless LED). O grau de conversão foi analisado por ANOVA on ranks e teste Student-Newman-Keuls. Os demais resultados foram analisados por ANOVA dois fatores para medidas repetidas e Teste de Tukey. A associação entre as características das resinas compostas e o potencial benéfico do uso dos materiais a 25°C ou a 5°C foi realizada por meio do teste Qui-quadrado. Os resultados foram correlacionados pelo teste de Correlação de Pearson. O nível de significância, para todas as análises, foi de 5%. Todos os materiais apresentaram maior grau de conversão a 25°C. Maior sorção e solubilidade foram observadas a 25°C e após 30 dias. Maiores resultados de dureza foram observados a 25°C e após 7 dias. Considerando a indicação clínica, os materiais indicados para a região posterior apresentaram maior potencial para aumentar a dureza quando armazenados em temperatura ambiente (p=0,047), enquanto os materiais de uso universal apresentaram maior potencial para redução da sorção e solubilidade à temperatura ambiente (p=0,047) Os materiais do tipo microhíbridos apresentaram maior potencial de redução da solubilidade quando comparados aos materiais nanoparticulados (p=0,047). As resinas com maior quantidade de partículas demonstraram maior capacidade de reduzir a solubilidade (p=0,008). Por fim, observou-se que quanto maior o grau de conversão, maior a possibilidade de se observar maiores valores de dureza superficial.

Palavras-chave: Resina composta; Temperatura de polimerização; Grau de conversão; dureza; Sorção; Solubilidade; Armazenamento

ABSTRACT

Resin composites are widely used in dental practice to restore contour, function, and esthetics. They can be handled at different temperatures and must support the conditions of the oral cavity. This in vitro study evaluated the degree of conversion (FTIR/ATR), sorption and solubility (ISO 4049: 2000) and surface hardness (Koop) of seven composite resins (Filtek Z100, Z250, 350, One Bulk Fill, Bulk Fill Flow, Universal and P60) polymerized under two different conditions: 1 – temperature: ambient (25°C) or refrigerated (5°C); and 2 – storage: 7 days and 30 days. The samples were obtained using a cylindrical silicone mold with internal dimensions of 6mm in diameter and 1mm in depth and were polymerized for 20s using a LED device (VALO Cordless LED). The degree of conversion was analyzed by ANOVA on ranks and Student-Newman-Keuls test. The other results were analyzed by two-way ANOVA for repeated measures and Tukey's test. The association between the characteristics of resin composites and the beneficial potential of using the materials at 25°C or 5°C was performed using the Chi-square test. The results were correlated using the Pearson correlation test. The level of significance for all analyses was 5%. All materials showed higher degree of conversion at 25°C. Greater sorption and solubility were observed at 25°C and after 30 days. Greater hardness results were observed at 25°C and after 7 days. Considering the clinical indication, the materials indicated for the posterior region had greater potential to increase hardness when stored at room temperature (p=0.047), while materials for universal use preented greater potential to reduce sorption and solubility at room temperature (p=0.047) Microhybrid materials showed greater potential for reducing solubility when compared to nanoparticulate materials (p=0.047). Resins with greater amount of particles showed higher ability to reduce solubility (p=0.008). Finally, it was observed that the greater the degree of conversion achieved by the material, a higher hardness should be expected.

Keywords: Resin composite; Curing temperature; Degree of conversion; microhardness; Sorption; Solubility; Storage.

SUMÁRIO

1 INTRODUÇÃO

As resinas compostas são amplamente utilizadas em práticas odontológicas para restabelecer a forma, função e estética dos dentes e, portanto, se tornaram parte integrante da odontologia estética moderna (Puspitasari et al., 2019), sendo consideradas uma substituição bem-sucedida de restaurações de amálgama nos dentes posteriores (Almozainy, 2018). Melhorias contínuas nas propriedades das resinas compostas levaram à aceitação clínica generalizada desses materiais (Prasanna et al., 2007). No entanto, as resinas compostas ainda representam um desafio na prática odontológica (Darabi et al., 2019; Silva et al., 2017) devido às alterações químico-mecânicas que pode sofrer decorrentes de seu armazenamento, manipulação, técnica utilizada e exposição à umidade da cavidade oral.

As resinas compostas devem ser capazes de permanecerem na cavidade bucal por longos períodos sem o comprometimento de suas propriedades (Chaves et al., 2015). Profissionais possuem o hábito de armazenarem as resinas compostas em refrigerador para prolongar sua vida útil, geralmente em temperaturas que variam de 2°C a 5°C (Tauböck et al., 2015). Entretanto, os monômeros necessitam de certa mobilidade para serem adequadamente convertidos em polímeros (Trujillo et al., 2004). Uma vez que a refrigeração pode aumentar a viscosidade do material, diminuindo assim a mobilidade dos monômeros, uma alteração das propriedades da resina composta polimerizada pode ocorrer.

A polimerização é definida como uma reação química que converte monômeros em uma estrutura de cadeia polimérica (Galvão et al., 2013). Uma polimerização completa pode maximizar os benefícios químicos, mecânicos e de biocompatibilidade das resinas compostas (Dionysopoulos et al., 2016; Almozainy, 2018). No entanto, nem todos os monômeros são convertidos em polímeros durante a polimerização, o que resulta em monômeros livres insaturados no produto final (Puspitasari et al., 2019). Estes monômeros não reagidos podem ser

lixiviados pela saliva atuando como um plastificante e reduzindo as propriedades mecânicas do material. Além disso, podem desencadear reações alérgicas e favorecer o crescimento bacteriano em torno das restaurações (Daronch et al., 2005). Portanto, um adequado grau de conversão de monômeros em polímeros é de extrema importância para o bom desempenho clínico das restaurações de resinas compostas, melhorando suas propriedades de maneira geral (Almozainy, 2018).

Uma propriedade mecânica importante deste material é a dureza de superfície, definida como a resistência do material à penetração ou indentação permanente (Puspitasari et al., 2019). Bouschlicher et al. (2004) demostraram uma correlação linear entre dureza e grau de conversão ao investigar as diferenças entre o tipo de resina composta e a profundidade de polimerização do espécime. Quanto às propriedades químicas das resinas, a absorção/adsorção de moléculas de água por monômeros hidrófilos do material resinoso exposto ao ambiente úmido da boca representa um dos vários mecanismos de deterioração e pode resultar em degradação hidrolítica e ruptura da união das partículas de carga da matriz resinosa, além de lixiviação de monômeros não reagidos e de outros componentes da matriz polimérica (Santerre et al., 2001). Portanto, a baixa sorção e baixa solubilidade em meio aquoso são características desejáveis em materiais restauradores, como as resinas compostas (Carvalho et al., 2012).

Embora existam estudos analisando a influência das baixas temperaturas na polimerização das resinas compostas, tal efeito ainda necessita ser analisado, tornando oportuno a realização de mais estudos que busquem seus efeitos deletérios para a prática clínica. Assim, este estudo foi realizado com o intuito de avaliar a influência da temperatura (5 e 25ºC) e do tempo de armazenamento (7 e 30 dias) de sete diferentes resinas compostas, produzidas por um mesmo fabricante, com diferentes indicações de manipulação e clínicas. As propriedades analisadas foram o grau de conversão, sorção, solubilidade e dureza de superfície.

2 DESENVOLVIMENTO

2.1. Objetivos

2.1.1. Objetivo Geral

Avaliar a influência da temperatura e do tempo de armazenamento nas propriedades químicas e mecânicas de diferentes resinas compostas.

2.1.2. Objetivo Específico

Capítulo 1 –

O objetivo deste estudo foi avaliar sete resinas compostas diferentes, manipuladas em duas temperaturas (5 ° C e 25 ° C) e armazenados por 7 e 30 dias.

2.2. Capítulo 1

Chemical and mechanical properties of dental resin composites: effect of temperature and time of storage

O presente artigo científico será submetido para publicação no periódico *Journal of the Mechanical Behavior of Biomedical Materials,* Qualis CAPES Interdisciplinar A2. O trabalho é apresentado nesta seção com a formatação exigida pelo referido periódico (ANEXO A).

CHEMICAL AND MECHANICAL PROPERTIES OF DENTAL RESIN COMPOSITES: EFFECT OF TEMPERATURE AND TIME OF STORAGE

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ABSTRACT

Resin composites are produced to present different clinical indications, insertion techniques, monomer compositions, viscosities, filler sizes, and filler contents. They can be manipulated at different temperatures and must last for longer periods at the oral cavity. This study evaluated seven different resin composites (Filtek Z100, Z250, 350, One Bulk Fill, Bulk Fill Flow, Universal and P60), manipulated at two different temperatures (5°C and 25°C), and stored for 7 and 30 days. The materials were tested for degree of conversion (FTIR/ATR), sorption/solubility (ISO 4049:2000) and surface microhardness (Knoop). The degree of conversion was analyzed by ANOVA on ranks and the Student-Newman-Keuls test (α=5%). The other results were analyzed by two-way ANOVA for repeated measures and Tukey's Test (α=5%). The association between the characteristics of the resin composites and the potential benefit of using the materials at 25°C or at 5°C was performed using the Chi-square test (α=5%). The results were correlated using the Pearson Correlation test (α=5%). A higher degree of conversion was observed at 25°C. Higher sorption and solubility were observed 25°C and after 30 days. All materials showed higher surface hardness at 25ºC and after 7 days. It was not observed an association between the variables and degree of conversion, but it was observed for sorption and solubility and surface hardness. None of the correlations resulted in statistical significance. Precooling and a long-stored period influenced on the properties of the resin composites.

Keywords: Resin composite; Curing temperature; Degree of conversion; microhardness; Sorption; Solubility; Storage.

INTRODUCTION

Resin composites have changed the Restorative Dentistry in terms of aesthetics, performance, and practicality[1]. It is one of the most commonly used dental materials due to its ability to bond to enamel and dentin, similarity to tooth structure in terms of color and mechanical properties, facility of clinical application and comparatively low cost to ceramic restorations[2]. Although in recent years the quality of resin composites restorations has improved, this material still represents a huge challenge in dental practice due to the physical and chemical changes it can undergo as a result of its storage, handling, technical procedure and exposure in the oral cavity[3,4].

It is composed of methacrylate monomers (organic phase), inorganic filler particles (inorganic phase), photoinitiator system and other minor additions, including stabilizers and color pigments[5,6]. The organic phase is constituted predominantly by the monomer Bisphenol A Diglycidyl Ether Dimethacrylate (BISGMA) in combination with other dimethacrylates[6]. BISGMA has high intrinsic reactivity, but usually does not achieve high conversion due to its high initial viscosity (η=1,200Pa)[7]. For this reason, and also to improve handling characteristics and to allow the incorporation of higher inorganic filler contents, it is usually combined with the low viscosity monomers[8].

The material realized considerable evolution regarding optical, chemical, and mechanical properties, resulting in better aesthetic and clinical longevity and consequently it has been recommended for restoring all kinds of cavities in anterior and posterior teeth. The filler technology has improved allowing materials with new composition, size, shape, distribution, and content of filler particles. New color pigments provide translucency and opalescence to these materials,

mimetizing enamel and dentin optical properties[9]. Even the challenge for a better insertion and polymerization technique has changed. New resin composite materials class has been introduced relying upon bulk-fill technique[10]. All this evolution brought to a moment in which the same manufacturer may produce different resin composites with different organic and inorganic phases, different handling properties but the same clinical indication.

The cooling of methacrylate-based resin materials is important to be investigated, as the cold storage of resin composites is a common practice among dentists, performed to increase the useful life of the material and is even suggested by some manufacturers[11,12]. Its effect was already analyzed and it was observed no polymerization reaction at 0°C [13], lower heat of polymerization at 15°C [13], higher values of sorption and solubility at 10°C [1], lower hardness values at top e bottom (2mm) surfaces at 4°C after 7 days[14] and at 5°C immediately after polymerization[15] and lower degree of conversion, maximum conversion rate (Rpmax), time to achieve Rpmax, and degree of conversion at Rpmax at the top and bottom (2mm) surfaces at 3° and 10°C [11,16].

Despite the determination of some influence of low temperatures on the properties of the polymerized resins, it is still of significance to evaluate the refrigeration effect on the polymerization efficacy and longevity of the so-called new resin composites materials. A longer time since the composite was polymerized needs to be investigated too. So, the aim of this study was to evaluate the influence of the storage temperature (5º and 25ºC) and the time after the composite has been polymerized (7 or 30 days) of seven different resin composites, from the same manufacturer, with different clinical and handling indications. The properties analyzed were sorption and solubility, Knoop

microhardness and degree of conversion. The null hypothesis tested was that the storage temperature and the time would not influence the tested properties independently of the resin composite.

MATERIALS AND METHODS

Seven different commercial brands of resin composites, with different clinical and handling indications, produced by the same manufacturer were used (3M/ESPE, Saint Paul, USA). The materials had different insertion technique, clinical indication, monomer composition, viscosity, filler size, and filler content and are described in table 1.

The materials were kept in a refrigerator (5ºC) or at room temperature (25ºC) prior to sample preparation. In case of samples prepared at cold temperature (5ºC), the resin tube was returned to the refrigerator for 5min before performing a new sample. The samples were storage for seven and thirty days.

Attenuated Total Reflection – Fourier Transform Infrared (ATR-FTIR) Spectroscopy Analysis

A cylinder-shaped silicone mold with internal dimensions of 6mm-diameter and 1mm-depth was used to obtain the samples. The samples were cured for 20s by a LED light curing device (VALO Cordless LED Curing Light – Ultradent, Salt LakeCity, Utah, USA). The device's irradiance was 1.000 mW/cm² and its tip diameter was 9.75mm.

Portions of uncured material and the upper surfaces of the light-cured samples were analyzed by FTIR-ATR spectroscopy (Perkin Elmer, Norwalk, California, USA) at 4000-650nm (n=3). Three scans of each sample were performed with a resolution of $2cm^{-1}$. Peak heights at $1637cm^{-1}$ (aliphatic carbon-

carbon bonds) and at 1608cm^{-1} (aromatic carbon-carbon bonds) were used. Measurements were made using the normalized baseline method[17] and the values of percentage of conversion degree (%DC) of monomers were determined by the following equation:

$$\%DC = \left[1 - \left(\frac{\frac{1637cm^{-1}}{1608cm^{-1}} Peakheightcured}{\frac{1637cm^{-1}}{1608cm^{-1}} Peakheightuncured}\right)\right] x100$$

Sorption/Solubility

The same cylinder-shaped silicone mold (6mm x 1mm) was used to obtain the samples. Water sorption and solubility were determined according to the ISO specification n.4049 (ISO 4049:2000)[18], except for specimens' dimensions and period of water immersion that was extended up to 30 days as performed by Malacarne et al (2006)[19]. A polyester strip was placed on a glass plate to receive the silicone mold. A 12cm long orthodontic wire was placed between the matrix and the polyester strip. The samples (n=10) were cured for 20s as described previously and were finished and polished with silicon carbide sandpaper (#1.200 – 3M, Sumaré, SP, Brazil) to remove excess resin.

Five samples from each group were placed inside Falcon tubes on perpendicular position and with a minimum separation of 3mm between them. The tubes were placed in a desiccator maintained at 37±1°C. After 22h, they were removed and stored in a second desiccator kept at 23±1°C for 2h and subsequently weighed using a precision balance (AX200 – Shimadzu, Kyoto, Japan) with an accuracy of ±0.1mg. This cycle was repeated until obtaining a

constant mass (M1), that is, until the mass loss of each sample was not greater than 0.1mg in any period within 24h. After the final drying, two diameter measurements were taken perpendicularly to each other, and the average diameter was calculated. The thickness of the sample was measured at its center and at four equally spaced points on the circumference. The area was calculated in square millimeters from the average diameter and then, using the average thickness, the volume (V) was calculated in cubic millimeters.

The samples were immersed in distilled water at 37±1°C for 7 days. The volume of water for immersion of the samples was 10ml per sample (5 samples per tube/50 ml). After 7 days, they were removed, washed with water, dried until free of visible moisture, agitated in the air for 15s and weighed 1min after removing the water. This mass was recorded as M2. After this weighing, the samples were reconditioned in desiccators to obtain the constant mass (M3) using the same cycle described to obtain M1.

The samples were immersed in distilled water at 37±1°C again, this time for 23 days, following the same method to obtain M2, and after weighing, M4 was obtained. After this weighing, the samples were dried again in a desiccator and weighed to obtain a third constant mass (M5).

Sorption and solubility data, after seven and thirty days, were calculated using the following formula:

Sorption after seven days: M2-M3/V

Solubility after seven days: M1-M3/V

Sorption after thirty days: M4-M5/V

Solubility after thirty days: M1-M5/V

Knoop Microhardness

The same cylinder-shaped silicone mold (6mm x 1mm) was used to obtain the samples. The samples (n=10) were cured for 20s as described previously and were finished and polished with silicon carbide sandpaper (#1.200 – 3M, Sumaré, SP, Brazil) to remove excess resin. Knoop microhardness analysis was obtained in a microhardness tester (HMV-G – Shimadzu, Kyoto, Japan) after 7 and 30 days of storage. A force of 50g was applied to the upper surface of the specimens for 15s. The resulting impression was evaluated at 10x magnification lenses. The longest diagonal was measured, and the data was determined by automatic calculation. Three measurements were performed for each sample and the average value was calculated.

Statistical Analysis

Data related to continuous variables (hardness, sorption, solubility and degree of conversion) were statistically analyzed using SigmaPlot software v.12 (Systat Software Inc., San Jose, CA, USA). All data were assessed for normality distribution using the Shapiro-Wilk test. The degree of conversion was analyzed by ANOVA on ranks Test and the Student-Newman-Keuls Test. The analysis considered two variation factors: the resin brand and the storage temperature. To verify the effect of the storage time factor data were analyzed with two-way ANOVA for repeated measures Test followed by the Tukey's Test. The significance level was α=5% for all analyses.

The JASP software (version 0.8.1.1 – JASP Team, University of Amsterdam, Amsterdam, NH, The Netherlands) was used to verify the association between the characteristics of each composite resin and the potential benefit of using the material at room temperature (25°C) when compared to its use at refrigerated

temperature (5°C). First, the data relating to the temperature of 25°C were compared to those obtained at 5°C, thus obtaining the percentage variation between these two groups. The percentage variation values were dichotomized into “greater” or “smaller” increase/reduction of the following dependent variables: hardness, sorption, solubility and degree of conversion. The following filters were considered to determine the greater or smaller increase/reduction of the variables: 10% hardness increase; 45% sorption reduction; 40% solubility reduction; and 15% degree of conversion increase. Each filter was decided taking into account the distribution of values obtained for each resin group. Each dependent variable was analyzed separately with six possible independent variables: clinical indication (universal × posterior), insertion technique (conventional × bulk-fill), monomer composition (BISGMA/TEGDMA only x BISGMA/TEGDMA and other monomers), viscosity (regular × flow), filler size (microhybrid x nanoparticle) and filler content by weight (%) (high (>60) x low (≤60)). The association of the variables described was performed using the chi-square test. Continuous data of percentage variation of the variables hardness, sorption and solubility were correlated with each other using the Pearson correlation test. The significance level was α=5% for all analyses.

RESULTS

The results of the degree of conversion are shown in table 2. Variation factors were significant ($p \leq 0.001$), although their interaction was not significant ($p=0.316$). At the temperature of 5°C, Filtek Z100, Z250, Z350, Universal and P60 showed a degree of conversion similar to each other and higher than that of Filtek One Bulk Fill and Bulk Fill Flow ($p \leq 0.044$). The only exception was for resin Z250,

which demonstrated a similar degree of conversion to bulk-fill resins ($p \geq 0.054$). At the use temperature of 25°C, Filtek Z100 resulted in the highest degree of conversion in the study, similar to Universal ($p=0.063$). The latter showed a similar degree of conversion to Filtek Z250, Z350, Bulk Flow and P60 ($p \geq 0.410$), which did not differ from each other nor as to Filtek One Bulk Fill ($p>0.05$). All resins maintained at room temperature showed a higher degree of conversion than resins at refrigerated temperature ($p<0.001$).

Water sorption results are shown in table 3. At 5°C the variation factors were significant ($p \leq 0.001$), although their interaction was not significant ($p=0.113$). The same was observed at 25°C. The factors were not significant ($p>0.05$), although their interaction was significant ($p=0.004$). Regardless of the temperature, all resins had their sorption values significantly increased after storage in a humid environment for 30 days ($p<0.001$). Filtek Bulk Fill Flow showed the highest sorption values, while Filtek P60 and Z250 presented the lowest values at 5°C and 25°C respectively.

The solubility in water data is presented in table 4. For both temperatures (5°C and 25°C) the variation factors were not significant ($p>0.05$), although their interaction was significant ($p \leq 0.012$). All composites increased their solubility after storage for 30 days regardless of the temperature ($p<0.001$). Filtek Bulk Fill Flow resin presented the highest solubility. The lowest solubility was registered for the conventional resins.

The Knoop hardness results are shown in Table 5. For the data obtained at 5°C the variation factors were not significant ($p>0.05$), although their interaction was significant ($p=0.007$). At 25°C the variation factors were significant ($p \leq 0.001$), although their interaction was not significant ($p=0.116$). Regardless of the

temperature, all resins had their hardness values significantly reduced after storage in a humid environment for 30 days ($p<0.001$). The composite Filtek Z100 showed the highest data, while Filtek Bulk Fill Flow showed the lowest ($p<0.001$).

The associations between the variables investigated are shown in Table 6. Six independent variables were analyzed and three of them did not show any association with the dependent variables: insertion technique ($p\geq0.053$), monomer composition ($p \geq0.053$) and viscosity of the resin composite ($p\geq0.155$). The increase in the degree of conversion was the only dependent variable that did not show an association with any of the independent variables tested ($p\geq0.147$). Considering the clinical indication, the materials indicated for the posterior region showed greater potential to increase hardness when storage at room temperature ($p=0.047$), whereas the materials for universal use presented greater potential to reduce sorption and solubility at room temperature ($p=0.047$). Considering the filler size, it was observed that the microhybrid materials showed greater potential to reduce solubility when compared to the nanoparticle materials at room temperature ($p=0.047$). Relative to filler content by weight, resins with high filler content demonstrated a greater ability to reduce solubility when compared to resins with low filler content at room temperature ($p=0.008$).

The dependent variables were correlated with each other and the results are shown in Table 7. None of the correlations resulted in statistical significance ($p\geq0.056$). The only almost significant correlation was demonstrated between the hardness increase and the degree of conversion increase that showed a strong positive correlation between the variables ($r^2=0.743$; $p=0.056$), indicating that the greater the degree of conversion achieved by the material, a higher hardness should be expected.

DISCUSSION

The present study investigated the influence of the storage of seven different resin composites from the same manufacturer at refrigerator (5°C) and room (25°C) temperature on their polymer degree of conversion, sorption/solubility in water and microhardness. The materials presented different clinical indications, insertion technique, monomer composition, viscosity, filler size, and filler content. The samples were stored for 7 and 30 days. The null hypothesis was rejected, as both temperatures and storage periods affected the composites properties.

Refrigeration is a common practice in dental offices and sometimes recommended by manufacturers. However, when refrigerated, the composites may suffer an increase in viscosity [14,20] resulting in a reduction in the mobility of the monomers, which may difficult their conversion into polymers[14,21,22] and may change the properties of the polymerized resins[14,20]. In the presented study the resin composites showed superior chemical-mechanical behavior at room temperature, corroborating that they were negatively affected by refrigeration[1,13,15]. Furthermore, the increase in viscosity may have a direct impact on the handling properties of the materials, affecting the ease of handling of the more commonly viscous materials at room temperature[23,24].

A crucial point to be reached during restorative procedures with resin composites is to obtain satisfactory restorations with an appropriate light activation technique[25]. The polymerization process takes place in the organic matrix through monomer-polymer conversion due to an activation mechanism that requires sufficient light energy intensity and an adequate wavelength in order to activate a photoinitiator that will react with a reducer agent to form free radicals and initiate the polymerization process[26]. A low conversion rate of the monomers

reduces the mechanical strength of the restoration and its color stability due to oxidation of the unsaturated monomers as well as allergic reactions[27,28]. All composites tested presented a higher degree of conversion when polymerized at 25°C as it was observed by Daronch et al. (2005)[11] but it was not observed an association between the classification of the composites and the degree of conversion. Filtek Z100, Z250, Univeral and P60 showed the highest conversion results at 5°C (43.5%). At 25°C only Filtek Z100 presented the highest conversion results. These results showed how unpredictable the conversion of these materials is. This probably occurred due to changes in the extent of the different constituents by the manufacturer, being able to completely change a certain characteristic of the material even when presenting the same clinical indication and/or insertion technique.

The composite, as well the temperature and the period of storage affected the sorption and solubility. It was observed significant difference for sorption and solubility results at 5°C and 25°C and after 7 and 30 days of storage for all resin composites. Filtek Bulk Fill Flow showed the highest sorption and solubility results at 5ºC and 25ºC at both short and long periods of storage. Materials for universal use (anterior/posterior) showed greater potential to reduce sorption and solubility (Chi-square Test - p=0.047), the microhybrid filler sized materials demonstrated greater potential to reduce solubility (Chi-square Test - p=0.047) and resins with higher filler content presented greater ability to reduce solubility (Chi-square Test - p=0.008). Low conversion degree values may lead to increased sorption and solubility[1,5,29]. Higher degree of conversion allows the formation of higher cross-linking chains that lead to lower chemical affinity of the polymer with the solvent and less space available for penetration of the solvent between the polymer

chains[1]. Other important factors are the hydrophilicity of the polymer, the filler size and its content by weight[1,30]. When the content by weight of the fillers increases, the polymeric matrix decreases with consequent decrease of water sorption and solubility since it is a phenomenon mainly associated with the polymeric phase[30]. The water sorption and solubility values of all resins were higher after 30 days of storage than those at 7 days indicating the increase in sorption and solubility after a longer period of storage. These results are in accordance with Alshali et al. (2015)[30]. Although it was not determined by statistical analysis, it is possible to verify that the results of sorption and solubility at 5°C after seven days seems to be similar to the results at 25°C after 30 days, indicating that a longer period of storage must be carried out to verify its effect on these resins properties.

It was observed significant difference for the surface hardness results at 5°C and 25°C and after 7 and 30 days of storage. All materials showed higher Knoop hardness when polymerized at 25ºC and after 7 days of storage. Filtek Bulk Fill Flow showed the lowest hardness results at 5ºC and 25ºC at the short and long periods of storage. Filtek Z100 showed the highest results at 5ºC and 25ºC with no significant difference to Filtek P60 at 25ºC. Considering the clinical indication, the materials designated to the posterior region showed greater potential to increase hardness when stored at room temperature (Chi-square Test – p=0.047). Hardness tests data for a specific material provide information about its wear, polish ability and abrasive effect on antagonist teeth[31]. The surface hardness of a composite resin is influenced by resin matrix, filler particle shape, distribution, size and density. In addition to these, other factors are known to affect hardness, such as light intensity, curing time, storage, monomers

compositions, and photoinitiators used[32]. An increase in the degree of conversion also improves surface hardness[28]. The correlation analysis identified an almost significant result between hardness and degree of conversion increase with a positive correlation between the variables (r^2=0.743; p=0.056), indicating that, although not statistically significant, an increase in the degree of conversion is expected to generate a corresponding increase in hardness. All materials showed lower surface hardness after 30 days of storage indicating that a longer period of storage must be carried out to verify its effect on the properties of these resins. Unfortunately, it was not possible to identify how each one of the factors analyzed contributed alone to the results obtained in this study.

Seven brands of resin composites were tested. They were classified according to clinical indication, handling technique and composition. They have presented different compositions both in the organic phase and in the inorganic phase. The same manufacturer offers many brands so that professionals may have a wide range to choose from. The same material can be indicated for different clinical situations. Just as another material can be indicated exclusively for a single clinical situation. It was found that the handling temperature (5ºC and 25ºC) influenced the final characteristics of the polymer. As well as the effect of the interaction of a longer storage period. It was not possible to determine an association between all the variables and it was not observed any correlation with statistical significance. But it could be observed that materials with smaller amounts of particles, that is, with larger amounts of monomers, seems to be more sensitive to storage and temperature variations. In addition, it seems reasonable to indicate the use of refrigerated materials after allowing them to reach room temperature. Analyzes must be performed to verify the effect of temperature on

other material properties. Longer storage times must also be analyzed. Finally, it is recommended that when chosing a restorative resin composite to do that based on a habitual restorative technique and achieving the best degree of conversion as possible.

CONCLUSION

The resin composites tested were significantly affected by lower temperatures and higher periods of storage. It was only possible to determine an association between the variables with sorption, solubility and surface hardness. None of the correlations resulted in statistical significance.

REFERENCES

1. Castro FLA, Campos BB, Bruno KF, Reges RV. Temperature and curing time affect composite sorption and solubility. **J. Appl. Oral Sci**. 2013; 21(2): 157-162.
2. Bolat M, Stoleriu S, Iovan VV, Pancu G, Sandu AV, Taraboanta I. Comparative Study of Color Stability of Three Composite Materials, Treated by Finishing and Coated Sealing, After Immersion in Different Wholesale. **Rev. Chim.** 2019; 70(5): 1681-1684.
3. Darabi F, Seyed-Monir A, Mihandoust S, Maleki D. The effect of preheating of composite resin on its color stability after immersion in tea and coffee solutions: An *in-vitro* study. **J Clin Exp Dent**. 2019; 11(12):e1151-e1156.
4. Silva TMD, Sales ALLS, Pucci CR, Borges AB, Torres CRG. The combined effect of food-simulating solutions, brushing and staining on color stability of composite resins. **Acta Biomater Odontol Scand**. 2017; 3(1):1-7.
5. Sideridou I, Tserki V, Papanastasiou G. Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins. **Biomaterials**. 2002; 23:1819–1829.
6. Shobha HK, Sankarapandian M, Kalachandra S, Taylor DF, McGrath JE. Structure property relationship among novel dental composite matrix resins. **J Mater Sci Mater Med**. 1997 Jun;8(6):385-9.
7. Peutzfeldt A. Resin composites in dentistry: the monomer systems. **Eur. J. Oral Sci**. 1997; 105(2):97–116.
8. Baroudi K, Mahmoud S. Improving Composite Resin Performance Through Decreasing its Viscosity by Different Methods. **Open Dent J**. 2015; 26(9):235-42

9. Almeida CM, Piva E, Duarte CG, Vieira HT, Isolan CP, Valente LL, Munchow EA. Physico-mechanical characterization and fracture reliability of dental resin composites for enamel restoration. **J Braz. Soc**. Mech. Sci. Eng**.** 2019; 41:398.
10. Martins LC, Oliveira LRS, Braga SSL, Soares CJ, Versluis A, Borges GA, Veríssimo C. Effect of Composite Resin and Restorative Technique on Polymerization Shrinkage Stress, Cuspal Strain and Fracture Load of Weakened Premolars. **J Adhes Dent**. 2020; 22(5):503-514.
11. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. **J Dent Res**. 2005; 84:663-667.
12. Borges GA, Spohr AM, Oliveira WJ, Correr-Sobrinho L, Correr AB, Borges LH. Effect of refrigeration on bond strength of self-etching adhesive systems. **Braz Dent J**. 2006; 17: 186-190.
13. Jafarzadeh-Kashi TS, Mirzaii M, Erfan M, Fazel A, Eskandarion S, Rakhshan V. Polymerization behavior and thermal characteristics of two new composites at five temperatures: refrigeration to preheating. **J Adv Prosthodont**. 2011; 3(4):216-20.
14. Osternack FH, Caldas DB, Rached RN, Vieira S, Platt JA, Almeida JB. Impact of refrigeration on the surface hardness of hybrid and microfilled composite resins. **Braz Dent J**. 2009; 20(1):42-7.
15. Awliya WY. The Influence of Temperature on the Efficacy of Polymerization of Composite Resin. **J Contemp Dent Pract**. 2007; 8(6):009-016.
16. Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. **J Dent Res**. 2006; 85(1):38-43.

17. Rueggeberg FA, Hashinger DT, Fairhurst CW. Calibration of FTIR conversion analysis of contemporary dental resin composites. **Dent Mater**. 1990; 6(4):241-9.
18. International Organization for Standardization, Technical Committee. ISO/TC 106/SC 1. Dentistry-polymer-based restorative materials (ISO 4049). **3rd ed. Geneva:** ISO; 2000.
19. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, Yiu CK, Carrilho MRO. Water sorption/solubility of dental adhesive resins. **Dent Mat.** 2006; 22(10):973-80.
20. Charton C, Colon P, Pla F. Shrinkage stress in light-cured composite resins: influence of material and photoactivation mode. **Dent Mat**. 2007; 23(8):911-20.
21. Trujillo M, Newmann SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. **Dent Mat**. 2004; 20:766-777.
22. D'Alpino PHP, Vismara MVG, Mello LMM, Hipólito V, González AHM, Graeff CFO. Resin composite characterizations following a simplified protocol of accelerated aging as a function of the expiration date. **J Mech Behav Biomed Mater**. 2014; 35:59-69.
23. Lee IB, Son HH, Um CM. Rheological properties of flowable, conventional hybrid, and condensable composite resins. **Dent Mat**. 2003; 19(4):298–307.
24. Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. **Dent Mat**. 2006; 22(6):515–26.

25. Porto IC, Soares LE, Martin AA, Cavalli V, Liporoni PC. Influence of the photoinitiator system and light photoactivation units on the degree of conversion of dental composites. **Braz Oral Res**. 2010; 24(4):475-81.
26. Carvalho AA, Moreira FCL, Fonseca RB, Soares CJ, Franco EB, Souza JB, Lopes LG. Effect of light sources and curing mode techniques on sorption, solubility and biaxial flexural strength of a composite resin. **J. Appl. Oral Sci**. 2012; 20 (2): 246-252.
27. Caughman WF, Rueggeberg FA, Curtis JW Jr. Clinical guidelines for photocuring restorative resins. **J Am Dent Assoc**. 1995; 126 (9): 1281-6.
28. Prasanna N, Pallavi Reddy Y, Kavitha S, Lakshmi Narayanan L. Degree of conversion and residual stress of preheated and roomtemperature composites. **Indian J Dent Res**. 2007; 18(4):173-176.
29. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. **Dent Mater**. 2006; 22(3):211-22.
30. Alshali RZ, Salim NA, Satterthwaite JD, Silikas N. Long-term sorption and solubility of bulk-fill and conventional resin-composites in water and artificial saliva. **J Dent**. 2015; 43(12):1511-8.
31. Almozainy M. Influence of storage temperature on vickers microhardness of resin composite. **Annals and Essences of Dentistry**. 2018; 10(1):1-11.
32. Galvao MR, Caldas SGFR, Bagnato VS, de Souza Rastelli AN, de Andrade MF. Evaluation of degree of conversion and hardness of dental composites photo-activated with different light guide tips. **Eur J Dent**. 2013; 7(1): 86-93.

Tables

Table 1 – Materials tested.

Resin Composite (Batch Number)	Composition*	Classification#
Filtek Z100 (2026900495)	Silane treated ceramic (aprox. 84% by Wt). BISGMA. TEGDMA.	Conventional Universal BISGMA/TEGDMA Regular Microhybrid High
Filtek Z250 (1921400294)	Silane Treated Ceramic (aprox. 75% by Wt). BISGMA. BISEMA. UDMA. TEGDMA	Conventional Universal Others Regular Microhybrid High
Filtek Z350 (2004200294)	Silane Treated Ceramic (aprox. 72% by Wt). BISGMA. BISEMA. UDMA. PEGDMA. TEGDMA.	Conventional Universal Others Regular Nanoparticle High
Filtek Bulk Fill Flow (980594)	Silane Treated Ceramic (aprox. 60% by Wt). UDMA. Substituted Dimethacrylate (Procrylat). BISGMA. BISEMA. TEGDMA.	Bulk Fill Posterior Others Low Nanoparticle Low
Filtek One Bulk Fill (2028800619)	Silane Treated Ceramic (aprox. 69% by Wt). AUDMA. DDDMA. UDMA.	Bulk Fill Posterior Others Regular Nanoparticle Low
Filtek Universal (2007300338)	Silane Treated Ceramic (aprox. 68% by Wt). AUDMA. DDDMA. UDMA.	Bulk Fill Posterior Others Regular Nanoparticle High
Filtek P60 (10002070084)	Silane treated ceramic (aprox. 83% net wt.). BISGMA. BISEMA. UDMA. TEGDMA.	Conventional Posterior Others Regular Microhybrid High

*Reported by the manufacturer in the Material Safety Data Sheet (MSDS).

#Autohors' Classification: Insertion Technique – Conventional/Bulk; Clinical Indication – Universal (anterior/posterior)/Posterior (posterior); Monomer Composition – BISGMA/TEGDMA (BISGMA/TEGDMA only)/Others (BISGMA/TEGDMA and other monomers); Viscosity: Regular/Low; Filler Size: Microhybrid/Nanoparticle; Filler Content by weight (%): High (>60)/Low (≤60).

Table 2 – Mean and standard deviation (SD) of conversion degree (%) of resin composites tested varying the temperature of storage.

Resin Composite	Temperature of storage	
	5°C	25°C
Filtek Z100	43.5 (1.0)^{A,b}	53.5 (1.4)^{A,a}
Filtek Z250	40.1 (1.6)^{AB,b}	49.1 (3.4)^{BC,a}
Filtek Z350	41.7 (1.0)^{A,b}	48.8 (1.5)^{BC,a}
Filtek One Bulk Fill	37.1 (1.3)^{B,b}	46.0 (1.1)^{C,a}
Filtek Bulk Fill Flow	36.6 (1.0)^{B,b}	48.1 (0.6)^{BC,a}
Filtek Universal	43.8 (1.9)^{A,b}	50.7 (1.6)^{AB,a}
Filtek P60	40.8 (2.0)^{A,b}	49.6 (2.9)^{BC,a}

Uppercase superscript letters indicate significant differences ($p<0.05$) between resin composite (same column) and lowercase superscript letters on the same line represent significant differences between the temperatures tested ($p<0.05$).

Table 3 – Mean and standard deviation (SD) of water sorption of the resin composites tested varying the temperature of storage and period of storage after polymerization.

Resin Composite	5°C		25°C	
	7 days	30 days	7 days	30 days
Filtek Z100	7.1 (0.8)^{BC,b}	10.6 (1.3)^{C,a}	3.6 (0.8)^{B,b}	7.2 (0.9)^{B,a}
Filtek Z250	6.5 (0.9)^{BC,b}	10.5 (1.8)^{C,a}	3.4 (0.4)^{B,b}	5.0 (1.0)^{D,a}
Filtek Z350	7.1 (1.1)^{BC,b}	10.5 (1.2)^{C,a}	3.6 (0.6)^{B,b}	6.2 (0.6)^{C,a}
Filtek One Bulk Fill	7.7 (1.2)^{B,b}	13.0 (1.9)^{B,a}	4.1 (0.8)^{B,b}	7.3 (1.4)^{B,a}
Filtek Bulk Fill Flow	9.6 (0.3)^{A,b}	14.4 (1.3)^{A,a}	5.5 (0.4)^{A,b}	8.8 (0.8)^{A,a}
Filtek Universal	6.9 (0.8)^{BC,b}	10.5 (0.9)^{C,a}	4.0 (0.7)^{B,b}	6.2 (0.5)^{C,a}
Filtek P60	6.2 (0.7)^{C,b}	10.3 (1.2)^{C,a}	3.5 (0.8)^{B,b}	6.1 (0.6)^{C,a}

Uppercase superscript letters indicate significant differences ($p<0.05$) between resin composites (same column) and lowercase superscript letters in the same line represent significant differences between the investigated periods ($p<0.05$) for each temperature tested.

Table 4 – Mean and standard deviation (SD) of water solubility of the resin composites tested varying the temperature of storage and period of storage after polymerization.

Resin Composite	5°C		25°C	
	7 days	30 days	7 dias	30 days
Filtek Z100	4.7 (0.8)^{B,b}	7.7 (1.1)^{BC,a}	2.4 (0.7)^{C,b}	4.1 (0.4)^{D,a}
Filtek Z250	5.1 (0.8)^{B,b}	7.8 (1.2)^{BC,a}	2.4 (0.6)^{C,b}	4.1 (0.5)^{D,a}
Filtek Z350	4.2 (0.7)^{B,b}	7.3 (1.2)^{C,a}	2.3 (0.8)^{C,b}	5.0 (0.8)^{C,a}
Filtek One Bulk Fill	4.9 (1.0)^{B,b}	8.5 (1.6)^{B,a}	3.4 (1.0)^{B,b}	7.1 (1.0)^{B,a}
Filtek Bulk Fill Flow	6.5 (0.5)^{A,b}	1.0 (0.9)^{A,a}	4.9 (0.6)^{A,b}	8.1 (0.7)^{A,a}
Filtek Universal	4.1 (0.5)^{B,b}	7.2 (0.7)^{C,a}	2.5 (0.5)^{C,b}	4.7 (0.6)^{CD,a}
Filtek P60	4.5 (0.7)^{B,b}	6.8 (0.5)^{C,a}	2.5 (0.6)^{C,b}	4.8 (0.6)^{CD,a}

Uppercase superscript letters indicate significant differences ($p<0.05$) between resin composites (same column) and lowercase superscript letters in the same line represent significant differences between the investigated periods ($p<0.05$) for each temperature tested.

Table 5 – Mean and standard deviation (SD) of Knoop hardness of the resin composites tested varying the temperature of storage and period of storage after polymerization.

Resin Composite	**5°C**		**25°C**	
	7 days	**30 days**	**7 dias**	**30 days**
Filtek Z100	106,1 (4,7)^{A,a}	93,1 (4,1)^{A,b}	114,2 (2,6)^{A,a}	100,6 (2,4)^{A,b}
Filtek Z250	97,8 (2,6)^{B,a}	84,5 (2,1)^{C,b}	108,1 (3,7)^{B,a}	94,8 (2,8)^{B,b}
Filtek Z350	93,4 (2,3)^{C,a}	84,5 (2,7)^{C,b}	103,2 (2,8)^{C,a}	91,2 (2,7)^{C,b}
Filtek One Bulk Fill	78,9 (2,6)^{E,a}	68,2 (2,2)^{E,b}	90,1 (2,8)^{D,a}	75,4 (3,4)^{D,b}
Filtek Bulk Fill Flow	51,0 (1,7)^{F,a}	40,4 (1,4)^{F,b}	60,0 (2,7)^{E,a}	48,6 (1,7)^{E,b}
Filtek Universal	83,2 (1,5)^{D,a}	71,9 (0,9)^{D,b}	88,5 (1,4)^{D,a}	75,4 (2,0)^{D,b}
Filtek P60	98,1 (3,1)^{B,a}	88,2 (2,0)^{B,b}	113,3 (1,2)^{A,a}	100,6 (2,5)^{A,b}

Uppercase superscript letters indicate significant differences ($p<0.05$) between resin composites (same column) and lowercase superscript letters in the same line represent significant differences between the investigated periods ($p<0.05$) for each temperature tested.

Table 6 – Association between dependent and independent variables investigated regarding the potential benefit of applying the resin at 25°C compared to 5°C. Chi-square Test.

Independent Variables	Hardness Increase	Sorption Reduction	Solubility Reduction	Degree of conversion Increase
Clinical Indication	**p=0.047**	**p=0.047**	**p=0.047**	p=0.809
Insertion Technique	p=0.053	p=0.809	p=0.053	p=0.290
Monomer Composition	p=0.809	p=0.809	p=0.053	p=0.427
Viscosity	p=0.155	p=0.155	p=0.350	p=0.571
Filler Size	p=0.659	p=0.659	**p=0.047**	p=0.147
Filler Content By Weight	p=0.270	p=0.270	**p=0.008**	p=0.809

Statistically significant p values are shown in **bold**.

Table 7. Correlation index between the dependent variables and their statistical significance (p value). Pearson correlation ($p<0.05$).

Hardness Increase × Sorption Reduction	**Hardness Increase × Solubility Reduction**	**Hardness Increase × DC Increase**	**Sorption Reduction × Solubility Reduction**	**Sorption Reduction × DC Increase**	**Solubility Reduction × DC Increase**
r^2=-0.254 p=0.583	r^2=-0.558 p=0.193	r^2=0.743 p=0.056	r^2=0.534 p=0.217	r^2=-0.120 p=0.798	r^2=-0.530 p=0.222

3 CONCLUSÃO

As resinas compostas avaliadas apresentaram, em ambos os períodos de armazenamento (7 e 30 dias), comportamento químico-mecânico superior ($p<0,05$) quando utilizadas à temperatura ambiente (25°C) quando comparadas ao mesmo compósito refrigerado (5°C), apresentando resultados inferiores em 30 dias. A refrigeração das resinas compostas pode afetar negativamente seu potencial de polimerização e, consequentemente, as propriedades do material.

REFERÊNCIAS

1 Puspitasari D, Prasetyo A, Rahman MD, Diana S, Ichrom Nahzi MY. Storage Temperature Effect on Degree of Polymerization and Surface Hardness of Bulk-Fill Composite Resin. **Journal of International Dental & Medical Research**. 2019; 12(2):405-410.

2 Almozainy M. Influence of storage temperature on vickers microhardness of resin composite. **Annals and Essences of Dentistry**. 2018; 10(1):1-11.

3 Prasanna N, Pallavi Reddy Y, Kavitha S, Lakshmi Narayanan L. Degree of conversion and residual stress of preheated and roomtemperature composites. **Indian J Dent Res**. 2007; 18(4):173-176.

4 Darabi F, Seyed-Monir A, Mihandoust S, Maleki D. The effect of preheating of composite resin on its color stability after immersion in tea and coffee solutions: An in-vitro study. **J Clin Exp Dent**. 2019; 11(12):e1151-e1156.

5 Silva TMD, Sales ALLS, Pucci CR, Borges AB, Torres CRG. The combined effect of food-simulating solutions, brushing and staining on color stability of composite resins. **Acta Biomater Odontol Scand**. 2017; 3(1):1-7.

6 Chaves FO, Farias NC, Medeiros LC, Alonso RC, Di Hipòlito V, D'Alpino PH. Mechanical Properties of Composites as Functions of The Syringe Storage Temperature and Energy Dose. **J Appl Oral Sci**. 2015; 23(2):120

7 Tauböck TT, Tarle Z, Marovic D, Attin T. Preheating of high-viscosity bulk-fill resin composites: Effects on shrinkage force and monomer conversion. **Journal of Dentistry**. 2015; 43(11):1358-64.

8 Trujillo M, Newmann SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. **Dent Mat**. 2004; 20:766-777.

9 Galvao MR, Caldas SGFR, Bagnato VS, de Souza Rastelli AN, de Andrade MF. Evaluation of degree of conversion and hardness of dental composites photo-activated with different light guide tips. **Eur J Dent**. 2013; 7(1):86-93.

10 Dionysopoulos D, Tolidis K, Gerasimou P. The Effect of Composition Temperature and Post-Irradiation Curing of Bulk Fill Resin Composites on Polymerization Efficiency. **Mat Res**. 2016; 19(2):466-73.

11 Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. **J Dent Res**. 2005; 84:663-667.

12 Bouschlicher MA, Rueggeberg FA, Wilson BM. Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. **Oper Dent.** 2004; 29:698-704.

13 Santerre JP, Shajii L, Leung BW. Relation of dental composite formulations to their degradation and the release of hydrolyzed polymeric-resin-derived products. **Crit Rev Oral Biol Med**. 2001; 12:136-51.

14 Carvalho AA, Moreira FCL, Fonseca RB, Soares CJ, Franco EB, Souza JB et al . Effect of light sources and curing mode techniques on sorption, solubility and biaxial flexural strength of a composite resin. **J. Appl. Oral Sci.** 2012; 20(2):246-252.

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