Effect of bleaching agents on mechanical properties of bulk fill resin and conventional composites

Efeito do clareamento dental nas propriedades mecânicas de resinas bulk fill e em compósitos convencionais

Efecto del blanqueamiento dental en las propiedades mecánicas de las resinas bulk fill y en los compósitos convencionales

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Hellen Caroliny de Carvalho
ORCID: https://orcid.org/0000-0002-1998-5482
University of North Parana, Brazil
E-mail: hellencaroliny11@gmail.com

Omar Geha
ORCID: https://orcid.org/0000-0001-8804-8954
University of North Parana, Brazil
E-mail: omargeha@yahoo.com.br

Gabriela Torres Zanin
ORCID: https://orcid.org/0000-0002-7802-7014
University of North Parana Brazil
E-mail: gabrielatorreszanin@gmail.com

Maria Fernanda Braga Reis
ORCID: https://orcid.org/0000-0001-8516-4845
University of North Parana, Brazil
E-mail: mbragareis@gmail.com

Ricardo Danil Guiraldo
ORCID: https://orcid.org/0000-0002-1625-3120
University of North Parana, Brazil
E-mail: rdguiraldo@gmail.com

Arlison Nunes Ferreira de Matos
ORCID: https://orcid.org/0000-0002-0695-402X
University of Cuiabá, Brazil
E-mail: drarilsonmatos@yahoo.com.br
Abstract
This study evaluated the effect of tooth whitening on resin composites (RC) on roughness, microhardness and flexural strength. There were three RC: one Bulk Fill type resin (Filtek Bulk Fill) and two conventional, Filtek Z350 (Z350) and Z250 (Z250). Twenty-four bar-shaped specimens were made for each RC. First, the initial roughness and microhardness were evaluated; thus, the RC were subdivided into 3 other groups, according to treatments: control group (C), this did not receive bleaching treatment; home bleaching with 10% carbamide peroxide (10%CP) (Opalescence PF), 8h/14 days; home-office bleaching with 40% hydrogen peroxide (40%HP) (Opalescence Boost PF) 3 × 15 minutes for 3 days at 48 hour intervals. After treatments, roughness and microhardness were again evaluated and then the samples submitted to the three-point flexural test. Data were tabulated, normality assessed, then submitted to ANOVA followed Tukey’s test (p <0.05). The results showed that the bleaching change the roughness of RC, but 10%CP had a significant increase. The gel concentration does not influence microhardness and flexural strength. After bleaching, Z350 has a lower elastic modulus. Based in the results obtained, could be concluded that the different bleachings did not promote significant changes on the RC studied.

Keywords: Composite resins; Tooth bleaching; Flexural strength.
clareador; clareamento caseiro com peróxido de carbamida 10% (10% PC) (Opalescence PF), 8h / 14 dias; clareamento caseiro com peróxido de hidrogênio 40% (40% PH) (Opalescence Boost PF) 3 x 15 minutos durante 3 dias com intervalos de 48 horas. Após os tratamentos, a rugosidade e a microdureza foram novamente avaliadas e as amostras submetidas ao ensaio de flexão em três pontos. Os dados foram tabulados, a normalidade avaliada e, em seguida, submetidas à ANOVA com teste de Tukey (p <0,05). Os resultados mostraram que o clareamento alterou a rugosidade da RC, mas 10% do PC teve um aumento significativo. A concentração do gel não influenciou a microdureza e a resistência à flexão. Após o clareamento, a Z350 apresentou um módulo de elasticidade inferior. Com base nos resultados obtidos, pode-se concluir que os diferentes clareadores não promoveram alterações significativas nas RC estudadas.

**Palavras-chave:** Resinas compuestas; Clareamento dental; Resistência à flexão.

**Resumen**

Este estudio evaluó el efecto del blanqueamiento dental en las resinas compuestas (RC) en cuanto a rugosidad, microdureza y resistencia a la flexión. Fueron comparadas tres RC: una resina tipo Bulk Fill (Filtek Bulk Fill) y dos convencionales, Filtek Z350 (Z350) y Filtek Z250 (Z250). Veinticuatro muestras en forma de barra fueron confeccionadas para cada RC. Primeramente, fueron evaluadas la rugosidad y microdureza iniciales; así, las muestras fueron subdivididas en otros tres grupos, de acuerdo con el producto utilizado: un grupo control (C), éste no recibió ningún tratamiento blanqueador; blanqueamiento casero con peróxido de carbamida 10% (10% PC) (Opalescence PF), 8h/14 días; blanqueamiento casero con peróxido de hidrógeno 40% (40% PH) (Opalescence Boost PF) 3 x 15 minutos durante 3 días con intervalos de 48 horas. Luego de los tratamientos, la rugosidad y la microdureza fueron nuevamente evaluadas y las muestras fueron sometidas a la prueba de flexión en tres puntos. Los datos fueron tabulados, se evaluó la normalidad y luego sometidos a ANOVA con la prueba de Tukey (p<0,05). Los resultados mostraron que el blanqueamiento alteró la rugosidad de las 3 RC, pero 10% PC tuvo un aumento significativo. La concentración del gel no tiene influencia sobre la microdureza y la resistencia a la flexión. Después del blanqueamiento, la Z350 presentó un módulo de elasticidad inferior. En base a los resultados obtenidos, se concluye que los diferentes blanqueadores no promovieron alteraciones significativas en las RC estudiadas.

**Palabras clave:** Resinas compuestas; Blanqueamiento de dientes; Resistencia flexional.
1. Introduction

The tooth bleaching technique is considered the most traditional one to change the color of the tooth element (Yu, et al., 2011) and (Souza, et al., 2020), however, the substances used, carbamide and hydrogen peroxides, may damage the tooth structure, as well as in restorative materials (Polydorou, et al., 2007) and (Berger, et al., 2013). The materials used by dentists in direct restoration are the resin composites, because of the advantages they provide compared to dental amalgam, such as conservative preparation, more lasting work, esthetics (Jacob & Kumar, 2007), and resistance to dental abrasion (Da Veiga, et al., 2016) and (El-Safty, Silikas & Watts, 2012).

The composites are formed by a dimethacrylate-based polymer matrix, that are inorganic particles of radiopaque glass, a silane union agent which allows the union among the matrixes; these composites are metal oxide pigments, and modulator agents of the polymerization reaction (indicators and inhibitors) (Petrovic, et al., 2013). The predominant monomer used on composite resin is the Bis-GMA (bisphenol A glycidylmethacrylate), which is mixed with other dimethacrylates because of its high viscosity, as the TEGDMA (triethylene glycol dimethacrylate), or other monomers (Petrovic, et al., 2013) and (Ilie, Bucuta & Draenert, 2013).

The composites are distinguished by the characteristics of their charge and by the size of the particles. Conventional composites (macroparticles) had inorganic quartz charge particles, strontium glass particles or barium; they also had medium-sized particles from five to 12 µm and sometimes 100µm, a radioactivity minor than the dentine spite of the great durability. The microparticles have fumed silica or 0.04-µm-sized colloidal silica, which is 300 times smaller than quartz; nevertheless, they have mechanical and physical properties inferior to traditional composites. On the other hand, the hybrid and microhybrid composites are a mixture of microparticles with macroparticles, presenting both characteristics. The difference between the hybrid and the microhybrid is on the proportion of particles used, in which there is a greater quantity of smaller particles on the microhybrid composite. In order to supply the necessity of a universal restorative material, so as to provide mechanical consistency and also the advantage of high polymer, nanohybrid (0.04 to 3.0 µm) and nanoparticle composites (5 to 70 nanometers) have been introduced on the market, enabling the advance of dental material radiopacity due to the use of nanotechnology, which enhanced the diagnose of secondary cavity and control of tooth restorative interface (Ilie, Bucuta & Draenert, 2013).
The degree of conversion is an important factor known to affect polymerization stress development through its influence on volumetric shrinkage (Marovic, et al., 2014). Incremental filling techniques have often been indicated to decrease the effects of shrinkage and stress generated at the adhesive interface (Versluis, et al., 2004). However, a singular increment composite (Bulk-Fill) has been created in order to optimize dentists’ timework. This kind of composite can be polymerized into 4-5mm layers, in which there is an increase in translucency, allowing higher light absorption (Attin, et al., 2004). There is also the use of new methacrylate-based monomers, as well as new photoinitiator systems that assure their polymerization and efficient mechanical properties (Petrovic, et al., 2013).

When bleaching agents are used in composite restored teeth, they may change the organic and inorganic structures of this material (Polydorou, et al., 2007). Studies show that the carbamide and hydrogen peroxides might degrade the composites (Polydorou, et al., 2007), (Janda, Roulet & Latta, 2006) and (American dental association council on scientific affairs, 2009). These changes may be perceived through the composite flexural strength, because it is a crucial parameter to the restoration occlusal charge (Kwon & Wertz, 2015), as well as its roughness, since there is a correlation between the aspect of the restorative surface and the accumulation of plaque (Seghi & Denry, 1992). The bleaching agents may cause changes on the composite surface smoothness, making the adherence of bacteria easier, causing several problems, such as restoration bleaching, lesion on secondary cavity, and gum irritation (Hess & Kirk-O, 1995).

Due to the introduction of Bulk Fill composites and the lack of research on the effect of bleaching treatments on these materials, this study aimed to evaluate the flexural strength, roughness and microhardness of different kinds of composites after home and in-office bleaching treatments. Thus, the hypothesis tested is that the composites change their properties when they are submitted to bleaching treatments.

2. Materials and Methods

Experimental design

This study used as experimental unit three kinds of composite resins: microhybrid, nanoparticle, and singular increment nanoparticle. These resins were submitted to two levels of treatments, 10% carbamide peroxide (home bleaching) and 40% hydrogen peroxide (in-
office bleaching), subject to 4 variable replies: roughness, microhardness, flexural strength, and elastic modulus.

**Samples preparation**

Three composites were evaluated on this study: Microhybrid (Filtek Z250 – 3M ESPE), Nanoparticulate (Filtek Z350 – 3M ESPE) and singular increment nanoparticle composite (Bulk Fill – 3M ESPE), each composite composition is described in Table 1.

**Table 1 – Composites used on the study and composition.**

| Material          | Type            | Organic matrix                                  | Filler                                              | Filler % weight/Vol |
|-------------------|-----------------|-------------------------------------------------|-----------------------------------------------------|---------------------|
| Filtek Z250 (Z250)| Microhybrid     | UDMA (urethane dimethacrylate) and bisEMA (6)   | Silica and zirconia                                 | 78/60               |
|                   |                 | (Bisphenol A - polyethylene glycol dieter dimethacrylate) |                                                     |                     |
| Filtek Bulk Fill (BF) | Nanoparticulate | Dimethacrylate urethane aromatic UDMA, DDDMA, pentanedioic acid, 2.2-dimethyl-4-methylene, reagent with glycidyl methacrylate, EDMAB, BENZOTRIAZOL, Water | Ceramic treated with Silane, Ybf3, Silica treated with Silane, titanium dioxide | 64.5/42.5            |
| Filtek Z350 XT (Z350) | Nanoparticulate | Bis-GMA, Bis-EMA, UDMA, TEGDMA                 | Nano particles of silica and zirconia, agglomerate zirconia-silica nanoclusters | 82/60               |

Source: Authors.

Twenty-four samples for each composite were manufactured using a matrix to obtain the bar shape (25 mm in length x 2 mm in thickness x 2 in width) in color A2.
The composite was inserted into the matrix and light cured with a polyester strip (K–Dent, Quimidrol, Joinville, SC, BR) pressured to a glass plate to certify the sample had a polished surface. Each sample was light cured for 40 seconds in four parts of the surface in order to ensure total polymerization with the light cure Radii-Cal – SDI (Radii – call, Radii Plus – Plastic Protection, SDI Limited, São Paulo, SP, BR) (1200 mW/cm²). After the composite bars have been manufactured, the samples were submitted to the cleaning in ultrasonic bath with distilled water for 2 minutes to remove any debris. Every specimen has been stored in distilled water at 37 °C for 24 hours. Each composite was subdivided into three different groups, as shown in Table 2.

Table 2 – Experimental group description based on the treatments.

| Treatment               | Description                                                                 |
|-------------------------|-----------------------------------------------------------------------------|
| Control (C)             | Immersed in distilled water, with no bleaching treatment.                   |
| 10% Carbamide peroxide  | Home bleaching treated with 10% Carbamide Peroxide (Opalescence PF 10%*) for 8 hours, during 14 days. |
| 40% Hydrogen peroxide   | In-office bleaching treated with 40% Hydrogen Peroxide (Opalescence boost PF 40%*) in three sessions, for 45 minutes each session. |

* Opalescence, Ultradent, South Jordan, UT, USA.  
Source: Authors.

Initial roughness

After manufacturing the samples, they were submitted to the reading of the initial superficial roughness, in three different directions, with the aid of a rugosimeter (SJ-410, Mitutoyo, Japan), which analyzed the surface at 0.25 mm in length and input the average of the superficial roughness in Ra (µm), which represents the arithmetic mean of the peaks and valleys found during the superficial scan.
Initial microhardness

The surface microhardness was made using a microdurometer (HMV-G 21S, Shimadzu Corporation, Japan). A pyramidal diamond indenter, Knoop-type was used, with static load of 25g, applied for 15 seconds. Three indentations were done in each sample, 100 μm away from each one. The indentations were made at the heart of each sample, where two adjusted lines determined the length of the bigger diagonal; thus, the values in Knoop were obtained automatically by the software. The average of the values was calculated after obtaining the three indentations.

Bleaching treatment

Home bleaching with 10% carbamide peroxide (10%CP) was made for eight hours daily for 14 days, simulating the nightly bleaching treatment, according to the manufacturer instructions (Opalescence PF, Ultradent, South Jordan, UT, USA). During the treatment interval the samples were stored in deionized water (Polydorou, et al., 2007) at 37 °C.

In-office tooth bleaching treatment with 40% hydrogen peroxide (40% HP) was performed in three sessions with three applications of 15 minutes each session, corresponding to a total of 45 minutes for the exhibition session, with intervals of 48 hours for each session, according to the manufacturer’s instructions. During the treatment intervals, the samples were in distilled water in a stove, at 37°C.

The 10%CP or 40%HP were applied on each RC (Table 1). After the treatments described above the samples were washed with running water and then cleaned by the ultrasonic cleaner (USC-1400, Unique Indústria e Comércio de Produtos Eletrônicos, Indaiatuba, SP, Brazil) for 5 minutes. Subsequently, the samples were stored in distilled water for 24 hours in a stove, at 37 °C to evaluate the roughness, microhardness, and flexural test.

Final roughness and microhardness

After the bleaching treatment, the samples were submitted again to the roughness and microhardness test, as described before. The initial roughness and microhardness data were compared with the final data (Berger, et al. 2019).
Flexural test

The three-point flexural test was performed according to ISO 4049: 1988 in a universal testing machine (DL2000, EMIC), at a speed of 0.5 mm/min. Each sample was put on a device 20 mm away from two metallic holders. Flexural strength (FS) was calculated by the formula:

\[ FS = \frac{3P_fL}{2WH^2} \]

in which \( P_f \) is the necessary maximum load to break the specimen (N), \( L \) is the distance between the holders (20 mm), \( W \) is the specimen width (mm), and \( H \) is the specimen thickness (mm).

The flexural test was monitored by a software in a computer connected to the trail mechanical machine, creating automatically a ‘tension x deformation’ chart during the test. The elastic modulus \( (E) \), for each specimen, was calculated from the ‘tension x deformation’ curve linear portion, which corresponds to the elastic deformation of the material, using the formula:

\[ E = \frac{(\Delta F / \Delta y)}{L^3 / 4WH^3} \]

in which \( \Delta F / \Delta y \) is the strength alteration \( (\Delta F) \) for the alteration unit of deflexion of the specimen heart \( (\Delta y) \), \( L \) is the distance between the holders (5 mm), \( W \) is the specimen width (mm), and \( H \) is the specimen thickness (mm).

Data analysis

Data were tabulated and evaluated, normality assessed using the Kolmogorov–Smirnov test, as presented a normal distribution, the data were submitted to ANOVA followed by Tukey’s test (\( p < 0.05 \)).

3. Results and Discussion

Comparing the roughness of each RC separately, before and after the treatments, it was possible to observe that Z350 was the only one which presented differences between the initial and final roughness values. However, when the composites were analyzed, all of them presented different results before the bleaching and did not present differences after the bleaching treatment. Table 3 shows the roughness average (standard deviation) according to the RC and evaluation time.
Table 3 – Average (standard deviation) of the Roughness values (Ra) according to the composites and evaluation time.

| Resin          | Time       |
|---------------|------------|
|               | Before     | After     |
| Filtek Bulk Fill | 0.058Aa   | 0.059aA  |
| Filtek Z350   | 0.053Aab  | 0.058aA  |
| Filtek Z250   | 0.050Ab   | 0.063bA  |

Measurements followed by different lowercase letters, in line, differ statistically from Tukey test (p < 0.05).
Measurements followed by different uppercase letters, in column, differ statistically from Tukey test (p < 0.05).
Source: Authors.

When the RC roughness was compared about the treatment used, was possible to observe that 40% HP presented similar results to the control group, whereas 10% CP presented roughness statistically superior to the others (Table 4).

Table 4 – Average (standard deviation) of the roughness values (Ra) according to the concentration used, independently of the composite and evaluation time.

| Concentration | Roughness |
|---------------|-----------|
| CONTROL       | 0.050B    |
| 10% CP        | 0.071A    |
| 40% HP        | 0.050B    |

Measurements followed by different uppercase letters, differ statistically from Tukey test (p < 0.05).
Source: Authors.

In Table 5 can observed that, when the treatments were evaluated in a general view, it was possible to observe that the roughness had increased significantly, independently of the treatment.
Table 5 – Average (standard deviation) of the Roughness values (Ra) according to the concentration used, independently of the composite and evaluation time.

| Time  | Roughness |
|-------|-----------|
| Before| 0.054A    |
| Final | 0.060B    |

Measurements followed by different uppercase letters, differ statistically from Tukey test (p < 0.05). Source: Authors.

When the microhardness of each composite was analyzed according to treatment, there were not statistical difference was observed (Table 6).

Table 6 – Average (standard deviation) of the Microhardness values according to the composites and treatment.

| Composites      | Concentration |
|-----------------|---------------|
|                 | Control       | 10% CP | 40% HP |
| Filtek Bulk Fill| 52.83aB       | 50.49aC| 53.22aB|
| Filtek Z350     | 64.27aA       | 60.77aB| 63.20aA|
| Filtek Z250     | 67.13aA       | 68.86aA| 65.53aA|

Measurements followed by different lowercase letters, in line, differ statistically from Tukey test (p < 0.05). Measurements followed by different uppercase letters, in column, differ statistically from Tukey test (p < 0.05). Source: Authors.

However, to RC microhardness comparison independently of the bleaching, the resin Bulk Fill presented a lower value in microhardness, followed by Z350 and Z250 (Table 7).
Table 7 – Average (standard deviation) of the microhardness values according to the composite used, independently of the concentration and evaluation time.

| Resin    | Microhardness |
|----------|---------------|
| Z250     | 67.17A        |
| Z350     | 62.75B        |
| Bulk Fill| 52.18C        |

Measurements followed by different uppercase letters, differ statistically from Tukey test (p < 0.05). Source: Authors.

All the composites present higher microhardness after the bleaching treatment (Table 8).

Table 8 – Average (standard deviation) of the microhardness values according to time, independently of the treatment and concentration.

| Time   | Microhardness |
|--------|---------------|
| Before | 59.73B        |
| Final  | 61.67A        |

Measurements followed by different uppercase letters, differ statistically from Tukey test (p < 0.05). Source: Authors.

When the flexural strength was evaluated, the composite Bulk Fill presented higher values when analyzed independently of the bleaching treatment, followed by Z250, which presented statistically similar results to Z350 (Table 9).
Table 9 – Average (standard deviation) of the flexural strength values according to the composite used, independently of the concentration and evaluation time.

| Resin     | Flexural strength |
|-----------|-------------------|
| Z250      | 148.22AB          |
| Z350      | 142.29B           |
| Bulk Fill | 163.36A           |

Measurements followed by different uppercase letters, differ statistically from Tukey test (p < 0.05).

Source: Authors.

When the elastic modulus of the RC was compared to control, the resin Bulk Fill presented a lower value; however, in both bleachings, the Z350 there is a lower elastic modulus (Figure 1).

Figure 1 - Average (standard deviation) of the elastic modulus values according to the composite and treatment.

Source: Authors.
This study evaluated different composite resins submitted to the treatment with 10% Carbamide Peroxide and with 40% Hydrogen Peroxide, testing the roughness, microhardness and flexural strength of the nanoparticle composites (Bulk Fill and Z350) and microhybrids (Z250). The hypothesis that the resins would suffer changes on their properties (Moraes, et al., 2006) was partly refused. There were not significant alterations in relation to the microhardness and to the flexural strength of the composites treated (Tables 6 and 9). It was only possible to verify, in a general way, changes on the superficial smoothness of the composite resins, in which all of them presented a roughness increase after the bleaching treatments (Table 3). Other studies also found alterations on the roughness of the composite resins when they were submitted to the bleaching treatment, as in Rodrigues, et al. (2011), using carbamide peroxide (10%) and hydrogen peroxide (10% and 35%). Wongpraparatana, et al. (2018), who used carbamide peroxide (10%) and hydrogen peroxide (40%), certified that all the tested composites had an increase on their superficial roughness (Wongpraparatana, et al., 2018). Mendes, et al. (2012) also found significant alterations on the roughness of the nanoparticle and nanohybrid composite resins submitted to 10% and 35% hydrogen peroxide. In spite of this, they used the sample polymer to observe whether it was possible to reverse the values; however, this was not observed by the authors mentioned above. On the other hand, Zuryati, Qian & Dasmawati (2013) contradict these findings, because they do not point any adverse effect on the superficial smoothness when they examined the nanoparticle composite roughness (Zuryati, Qian & Dasmawati, 2013). This divergence of results may be related to the composition differences of the materials used, as well as to the polymerization time. Thus, the authors used a nanocomposite designed to the previous restoration, obtaining an inferior charge (29% in volume) (Zuryati, Qian & Dasmawati, 2013), when compared to the composites of this study, as they also made the bleaching right after the composite polymerization, without waiting for the post-polymerization period, in which the polymerization process continues occurring for a period of time (Mohamad, et al., 2007).

Besides, it was possible to observe, through this study, that the resin composite submitted to the bleaching treatment with 10% carbamide peroxide increased significantly their roughness (Table 4). It was expected the higher bleaching gel concentration the higher roughness values would be. Nevertheless, it is relevant to consider that the total exposure time of the composite in relation to low concentration bleaching agents was higher than that of high concentration gels (Bahari, et al., 2016) and (Monaghan, Lim & Lautenschlager, 1992). Although the concentration is lower (10%) when compared to hydrogen peroxide (40%), the
carbamide peroxide treated group (10%) kept a treatment time of eight hours for 14 days, whereas hydrogen peroxide (40%) exposed a 45 minutes period over three days at 48 hours’ intervals. However, Wongpraparatana, et al. (2018) used the same bleaching concentration of this study, and yet, the data differed from this study, as no significant difference was found between the bleaching agent groups for each analyzed composite (Wongpraparatana, et al., 2018). The different results may be due to the use of different kinds of composite resins and also due to measurement methods of the superficial roughness.

Despite the fact that the influence of the bleaching treatment has not been found in the analysis of the tested composite microhardness (Table 6), Bulk Fill showed microhardness inferior when compared to the other composites, independently of the bleaching treatment (Table 7). The same effect was observed by Kelic, et al. (2016), who verified that Bulk Fill had inferior microhardness to conventional composites, because they concluded that the composite resin of singular increment, due to its low microhardness, would need an additional cover layer, because its microhardness is not high enough to resist to masticatory strengths (Kelic, et al., 2016). However, Tomaz, et al. (2016) showed different results when analyzing the superficial and deep microhardness of Bulk Fill composites, which presented higher roughness values than the conventional composite resin.

In this study, the bleaching treatment had no statistically significant influence on flexural strength (Table 9), as previous studies (Feiz, et al., 2016) and (Yu, et al., 2010). Firoozmand, et al. (2009) contradicted these results, as they found statistical differences on the flexural strength after the bleaching treatment of the materials studied. Thus, it is assumed again that the different results are related to the different bleaching techniques, to analysis, and to the composites involved.

When the composites were analyzed independently of the bleaching treatment used, Z350 nanoparticle resin showed lower strength value, obtaining results similar to the study of Hatanaka, et al. (2013), in which the flexural strength of Filtek Z350 nanoparticle composite was inferior to the hybrid and microhybrid composites. This may be the result of a possible negative effect of zirconia and silica nanotechnology material, which made possible the proliferation of fissures (Yu, et al., 2010) and (Hatanaka, et al., 2013).

Another property evaluated was the elastic modulus. The results show that this was similar to all composites. Besides, the bleaching treatment did not have influence on the values obtained (Fig. 1). This effect was also observed in other previous studies (Scribante, et al., 2019) and (Rizzante, et al., 2019).
Although this study had used the temperature of 37 °C in a stove, simulating the oral environment (Haywood & Heymann, 1991), there are limitation in terms of bacteria development and other living elements in the oral cavity, such as the presence of saliva that has an important function in the definition of the composition and oral microbiota activity (Marsh, et al., 2016), which could influence directly on tooth bleaching. However, controlled clinical trials would be necessary to determine a clinical implication (Zuryati, Qian & Dasmawati, 2013) of the impact this environment in the behavior of resin composite, because may be the results obtained by in vitro researches are clinically insignificant.

4. Conclusions

Based on the results of this study, it was concluded that the tested bleaching treatments did not produce significant alterations on the mechanical properties of the composite resins studied.

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**Percentage of contribution of each author in the manuscript**

- Hellen Caroliny de Carvalho – 25%
- Omar Geha – 5%
- Gabriela Torres Zanin – 20%
- Maria Fernanda Braga Reis – 5%
- Ricardo Danil Guiraldo – 10%
- Arilson Nunes Ferreira de Matos – 5%
- Andreza Maria Fábio Aranha – 5%
- Sandrine Bittencourt Berger – 25%