

**UNIVERSIDADE FEDERAL DE PELOTAS**  
**Faculdade de Odontologia**  
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**Tese**

**Avaliação laboratorial de materiais e técnicas utilizados em Ortodontia**

**Adauê Siegert de Oliveira**

**Pelotas, 2015**

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Tese apresentada ao Programa de Pós-Graduação em Odontologia da Faculdade de Odontologia da Universidade Federal de Pelotas, como requisito parcial para obtenção do título de Doutor em Odontologia, área de concentração Materiais Odontológicos.

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## **Aduauê Siegert de Oliveira**

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Tese aprovada, como requisito parcial, para obtenção do grau de Doutor em Odontologia, Programa de Pós-Graduação em Odontologia, Faculdade de Odontologia, Universidade Federal de Pelotas.

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**Dedico este trabalho aos meus sobrinhos, Tiago  
e Júlia, fontes de esperança.**

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## **Resumo**

OLIVEIRA, Adauê Siegert de. **Avaliação laboratorial de materiais e técnicas utilizadas em Ortodontia.** 2015. 57f. Tese (Doutorado) - Programa de Pós-Graduação em Odontologia. Universidade Federal de Pelotas, Pelotas.

A evolução dos materiais ortodônticos facilitou a prática clínica, difundiu a especialidade e popularizou os tratamentos. Propiciar tratamentos mais rápidos, sem prejuízo estético, confortáveis ao paciente e minimizando efeitos colaterais são os maiores objetivos das pesquisas nessa especialidade. Diversos fatores clínicos motivam, constantemente, novos estudos para desenvolvimento e evolução dos materiais associados à técnica. A exigência estética direciona o aprimoramento dos braquetes cerâmicos bem como das ligaduras elásticas transparentes. Por outro lado, a forte associação em Ortodontia e Odontopediatria exige que procedimentos ortodônticos em crianças sejam rápidos e eficientes devido ao tempo clínico reduzido. Assim, este estudo buscou contribuir para a evolução desses materiais por meio de estudos *in vitro*. Os objetivos foram: avaliar *in vitro* o efeito do tipo de dentífrico utilizado (regular ou clareador) na estabilidade de cor de ligaduras elásticas translúcidas, onde a variável-resposta foi alteração cromática ( $\Delta E$ ) de acordo com os parâmetros CIEL $^*a^*b^*$  e foi aferida através de espectrofotômetro; analisar as implicações na adesão de braquetes ao esmalte relacionadas a alterações de posicionamento dos braquetes e remoção do excesso de cimento durante a colagem, para isto, foi analisada a resistência de união (MPa) de braquetes metálicos unidos ao esmalte bovino utilizando diferentes protocolos de colagem, tendo como variável-resposta secundária o modo de falha da união; avaliar o efeito da utilização de duas fontes de luz simultâneas durante a fotoativação do cimento ortodôntico na resistência de união de braquetes ao esmalte e avaliar a adição de sal de iodônio a compósitos adesivos fotoativados para reduzir o tempo de fotoativação necessário para colagem de braquetes ortodônticos ao esmalte dentário.

Palavras-chave: Adesão. Braquetes ortodônticos. Cor. Dentífrico. Elásticos ortodônticos. Pigmentação.

## **Abstract**

OLIVEIRA, Adauê Siegert de. **Avaliação laboratorial de materiais e técnicas utilizados em Ortodontia.** 2015. 57f. Thesis (PhD in Dentistry) - Programa de Pós-Graduação em Odontologia. Universidade Federal de Pelotas, Pelotas, 2015.

The evolution of orthodontic materials facilitated clinical practice, spread the specialty and popularized treatments. Providing treatments faster, without prejudice aesthetic, comfortable for the patient and minimizing side effects are the major objectives of research in this specialty. Several clinical factors motivate constantly new studies for development and evolution of materials associated with the technique. The aesthetic requirement directs the improvement of ceramic brackets and the transparent elastic bandages. On the other hand, the strong association in Orthodontics and Pediatric Dentistry requires orthodontic procedures in children are fast and efficient due to reduced clinical time. The objectives are to evaluate in vitro effect of the type of toothpaste used (regular or bleaching) on color stability of elastic bandages translucent, where the response variable is color change ( $\Delta E$ ) according to the parameters CIEL \* a \* b \* and will be measured by a spectrophotometer; analyze the implications for the accession of brackets to enamel related to changes in positioning of the brackets and removing excess cement during bonding, for this, we will analyze the bond strength (MPa) of metal brackets attached to bovine enamel using protocols collage. With the response variable secondary mode joint failure; assess the effect of using two sources of simultaneous light for the photoactivation of orthodontic cement in bracket bond strength to enamel and evaluate the addition of iodonium salt to photoactivated adhesive composite to reduce the time of curing required for bonding of orthodontic brackets to tooth enamel.

**Keywords:** Adhesion. Brackets, orthodontic. Color. Dentifrice. Elastic, orthodontic. Staining.

## **Sumário**

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## **Notas preliminares**

A presente tese foi redigida segundo o Manual de Normas para trabalhos acadêmicos da UFPel, adotando o nível de descrição em capítulos não convencionais. Disponível no endereço eletrônico:

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O projeto de pesquisa que originou esta tese foi apresentado em 29 de novembro de 2012 e aprovado pela Banca Examinadora composta pelos Professores Doutores Marcos Britto Corrêa, Miguel Roberto Simões Régio e Rafael Ratto de Moraes.

## **1 Introdução**

O estudo *in vitro* de materiais odontológicos é um importante recurso para simular e prever o comportamento desses materiais em condições clínicas. Estudos laboratoriais colaboram para o desenvolvimento e controle de biomateriais, principalmente quando buscam resolver dúvidas oriundas da clínica odontológica e permitem estabelecer protocolos práticos que não podem ser testados clinicamente antes de evidências preliminares de sua aplicabilidade serem obtidas. Nas mais diversas áreas da odontologia, a boa correlação entre investigações laboratoriais e clínicas é fundamental para que o bom desempenho de novos materiais e técnicas seja alcançado.

Em ortodontia, diversos fatores clínicos motivam, constantemente, novos estudos para desenvolvimento e evolução dos materiais associados à técnica. A busca da força ótima para movimentação dentária, por exemplo, impulsiona a qualificação dos elásticos ortodônticos; por outro lado, a exigência estética direciona o aprimoramento dos braquetes cerâmicos bem como das ligaduras elásticas translúcidas. Contribuir para a evolução desses materiais por meio de estudos *in vitro* é um desafio.

A demanda por estética em dispositivos utilizados em tratamentos ortodônticos é cada vez maior, especialmente porque é crescente a procura de adultos por esse tipo de tratamento. Braquetes cerâmicos tornaram-se populares e, atualmente, são amplamente utilizados como alternativa estética a braquetes de aço inoxidável. Além dos já populares braquetes cerâmicos opacos, braquetes cerâmicos translúcidos também estão disponíveis comercialmente como alternativas a braquetes metálicos. No mesmo passo, enquanto ligaduras coloridas ou metálicas são utilizadas associadas à braquetes de aço inoxidável, ligaduras elastoméricas translúcidas (em geral de poliuretano) são utilizadas para braquetes estéticos, de forma a não interferir na estética destes dispositivos.

Apesar dos braquetes serem teoricamente resistentes ao manchamento, as ligaduras podem pigmentar devido ao contato com pigmentos oriundos de alimentos

ou bebidas (ARDESHNA; VAIDYANATHAN, 2009; KIM; LEE, 2009; LEW, 1990). Isso pode resultar em um problema, visto que o conjunto aparelho-ligadura torna-se menos estético conforme os elásticos mudam sua coloração (isto é, tornam-se menos translúcidos). Dessa forma, o determinante do intervalo entre as consultas pode deixar de ser o protocolo de ativação do aparelho ortodôntico e passar a ser a limitação estética da ligadura elástica. Nesse contexto, o profissional despende muito tempo clínico com a troca de ligaduras e o paciente necessita visitar o profissional frequentemente para a substituição destas.

Vários estudos acerca dos efeitos da pigmentação de bebidas, como café, chá e vinho, que são comumente associados à pigmentação de estruturas dentárias na cavidade bucal, foram realizados nos últimos anos (VALENTINI et al., 2011; FONTES et al., 2009; MEIRELES et al., 2009). Entretanto, pouco se sabe acerca da resistência ao manchamento de ligaduras ortodônticas. A pigmentação de elásticos pode ser dependente do tipo ou marca de ligadura utilizada, além do tipo de agente corante e, dessa forma, exposições que se assemelham às que ocorrem na cavidade bucal são preferíveis. Ademais, o tipo de dentífricio utilizado pelo paciente pode ter efeito na estabilidade de cor de materiais no meio bucal (SOLDATI et al., 2013; KIM, LEE, 2009).

Além da importância cada vez maior da estética relacionada a tratamentos ortodônticos, a possibilidade de adesão direta de braquetes à superfície dental e a evolução no desenho dos dispositivos ortodônticos não só facilitaram a prática clínica mas também permitiram a evolução da técnica ortodôntica. Andrews, em 1970, introduziu o primeiro aparelho ortodôntico pré-ajustado, com torque e angulação pré-determinados e inseridos no braquete. Assim, eliminou a necessidade de dobras, o que caracteriza a difundida técnica *straight-wire* (ANDREWS, 1976). Técnicas que utilizam braquetes pré-ajustados, tais como *straight-wire*, *tip-edge* e MBT, oferecem grande simplificação na prática ortodôntica (DELLINGER, 1978; MCLAUGHLIN; BENNETT, 1995). Essas técnicas, entretanto, exigem precisão durante a colagem dos braquetes, pois pequenos erros podem levar ao inadequado posicionamento dentário (MAGNESS, 1978).

A posição do braquete na coroa tem relação direta com a posição final do dente, influenciando diretamente no torque, altura, inclinação e rotação do dente (BALUT et al., 1992). O braquete mal posicionado no eixo horizontal pode levar à rotação dentária, enquanto a inadequada posição no sentido vertical leva ao

desnívelamento ou alteração de torque, possivelmente interferindo na inclinação buco-lingual. Assim, o mal posicionamento de braquetes exige recolagem ou ajuste no arco, o que pode aumentar o tempo do tratamento (VAN LOENEN et al., 2005; GERMANE et al., 1990) e interferir com a estética, por exemplo. Buscando a ótima posição do braquete, pequenos deslocamentos e giros do dispositivo na superfície do dente normalmente são necessários durante o processo de colagem. A movimentação do braquete durante o processo pode, pelo menos teoricamente, deslocar o cimento adesivo e interferir na união ao esmalte dentário condicionado. Entretanto, não há evidências na literatura que indiquem alterações na resistência de união de braquetes devido à movimentação durante a colagem. Além disso, outras variáveis, como o momento da remoção do excesso de cimento ortodôntico, antes ou depois da movimentação do braquete, também podem interferir na qualidade da adesão.

A utilização de materiais resinosos fotopolimerizados facilita a fixação de dispositivos ortodônticos, porém também está relacionada ao aumento do tempo clínico gerado pela necessidade de múltiplas exposições do cimento ortodôntico à luz polimerizadora. O procedimento de múltiplas exposições à luz pode ser inconveniente ao profissional, desconfortável aos pacientes e, muitas vezes, torna a ortodontia impraticável em crianças. De maneira geral, cada dispositivo ortodôntico necessita de 20 a 40 segundos de fotoativação para adequada polimerização (OESTERLE et al., 1995; STAUDT et al., 2006). A redução deste tempo pode ocasionar perda considerável na resistência de união do braquete ao esmalte dentário e consequentes problemas clínicos como a desunião prematura do dispositivo. O desenvolvimento de novos materiais e/ou protocolos de fotoativação que permitam reduzir o tempo de exposição à luz sem interferir na resistência de união têm sido, assim, objetivo de investigação.

A evolução dos materiais ortodônticos facilitou a prática clínica, difundiu a especialidade e popularizou os tratamentos. Propiciar tratamentos mais rápidos, sem prejuízo estético, confortáveis ao paciente e minimizando efeitos colaterais são os maiores objetivos das pesquisas na área da ortodontia. Dessa forma, este trabalho, dividido em 4 artigos, teve por objetivos:

- avaliar in vitro o efeito do tipo de dentífrico utilizado (regular ou clareador) na estabilidade de cor de ligaduras elásticas translúcidas;

- analisar as implicações na adesão de braquetes ao esmalte relacionadas a alterações de posicionamento dos braquetes e remoção do excesso de cimento durante a colagem;
- avaliar o efeito da utilização de duas fontes de luz simultâneas durante a fotoativação do cimento ortodôntico na resistência de união de braquetes ao esmalte;
- avaliar a adição de sal de iodônio a compósitos adesivos fotoativados para reduzir o tempo de fotoativação necessário para colagem de braquetes ortodônticos ao esmalte dentário.

## **2 Capítulo 1**

Influence of whitening and regular dentifrices on orthodontic clear ligature color stability<sup>1</sup>

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<sup>1</sup>Artigo publicado no periódico *Journal of Esthetic and Restorative Dentistry* (DOI: 10.1111/jerd.12138), sendo aqui apresentado com estrutura conforme normas do referido periódico.

## 2.1 Abstract

**Objectives:** This study evaluated the effect of brushing orthodontic clear ligatures with a whitening dentifrice containing a blue pigment (Close Up White Now) on their color stability, when exposed to a staining agent.

**Materials and Methods:** Ligatures from 3M Unitek and Morelli were tested. Baseline color measurements were performed and non-stained groups (control) were stored in distilled water while test groups were exposed for 1 h daily to red wine. Specimens were brushed daily using regular or whitening dentifrice. Color measurements were repeated after 7, 14, 21, and 28 days using a spectrophotometer based on the CIEL\*a\*b\* system.

**Results:** Decreased luminosity (CIE L\*), increased red discoloration (CIE a\* axis), and increased yellow discoloration (CIE b\* axis) were generally observed for ligatures exposed to the staining agent. Color variation was generally lower in specimens brushed with regular dentifrice, but ligatures brushed with whitening dentifrice were generally less red and less yellow than regular dentifrice. The whitening dentifrice led to blue discoloration trend, with visually detectable differences particularly apparent according to storage condition and ligature brand.

**Conclusions:** The whitening dentifrice containing blue pigment did not improve the ligature color stability, but it decreased yellow discoloration and increased a blue coloration.

## 2.2 Introduction

Improved mechanical and optical properties allow the use of aesthetic brackets as an alternative to traditional stainless steel brackets in orthodontic treatments. Ceramic and polycarbonate brackets provide good color stability and are virtually resistant to visually perceptible dietary pigment staining. However aesthetic brackets are associated with translucent elastomeric ligatures, usually polyurethane-based, that generally do not provide adequate color stability. Elastic ligatures are vulnerable to pigment absorption from food and drink in the oral environment.<sup>1-4</sup> Ligature discoloration is a significant clinical concern because the bracket-ligature set may become less aesthetically appealing over time.

Color plays an important role in several aspects of dentistry, and much attention has been directed to the color stability of dental materials and devices

exposed to variably staining substances.<sup>5-13</sup> In orthodontics, the time interval between clinical appointments should be determined by the orthodontic device activation protocol. Premature discoloration of elastic ligatures compromises the device aesthetic properties, which increases patient dissatisfaction and decreases the between-visit interval. Pigmentation of orthodontic ligatures usually depends on the ligature type, commercial brand, and pigmenting agent.<sup>1, 2, 4</sup>

Tooth brushing frequency and dentifrice type may also affect the color stability of dental materials.<sup>14</sup> Some dentifrices contain bleaching or whitening chemicals and specific pigments that aim to improve the tooth aesthetic quality.<sup>15, 16</sup> The dentifrice type may also impact the color stability of elastic ligatures, but there is no evidence in the literature at present. The use of dentifrices with whitening characteristics could minimize aesthetic losses caused by cumulative ligature staining, thereby increasing patient satisfaction with orthodontic treatments, and reducing the costs of additional clinical visits.

The present study investigates the effect of brushing orthodontic clear ligatures with a whitening dentifrice containing a blue pigment on the color stability of ligatures exposed to a staining agent. The hypotheses tested were: (i) exposure to a staining agent increases ligature color variability; and (ii) use of a whitening dentifrice provides better ligature color stability.

## **2.3 Material and Methods**

### **2.3.1 Study design and materials tested**

This *in vitro* 2×2×2 factorial designed study (8 groups, n = 10 specimens) evaluated three factors: dentifrice type (brushing with either a regular dentifrice or blue pigmented whitening dentifrice); material (two elastic ligature brands); and ligature storage condition (exposure to a staining agent [red wine] or no exposure). Two clear elastic ligature brands, 3M Unitek (Monrovia, CA, USA) and Morelli (Sorocaba, SP, Brazil), and two dentifrices, Close Up Regular and Close Up White Now (Unilever, London, UK), were tested. This particular whitening dentifrice was chosen because it is widely available to the public, and the manufacturer claims that it helps remove stains and progressively whitens teeth. The same ligature was analysed under variable storage times: baseline, 7, 14, 21, and 28 days (repeated measures approach). CIE *L\**, *a\**, and *b\** individual coordinates and color difference

( $\Delta E$ ) were response variables evaluated.

### 2.3.2 Storage conditions

One week before color evaluation, the ligatures were encircled on around brackets fixed to poly(methyl) methacrylate (PMMA) bars. The PMMA device was sealed with nail varnish, and 10 Edgewise brackets (Morelli) were bonded to each bar. A straight orthodontic wire was also positioned through the brackets to simulate *in vivo* ligature stretching. In total, 40 elastic ligatures of each ligature brand were positioned around brackets. Once baseline ligature color measurements were performed, the non-stained groups (control) for each elastic brand were stored in distilled water (pH = 6.6) at 37°C for the remainder of the experiment. Test groups for each elastic brand were exposed for 1 h daily to red wine (San Martín assemblage, Flores da Cunha, RS, Brazil) with a 10.4% v/v ethanol (pH = 3.3). Red wine was selected as the staining agent based on pilot studies and a previous investigation<sup>4</sup> that illustrated its ability to stain the ligatures. Distilled water and red wine pH measurements were performed in triplicate using a pH meter (An2000; Analion, Ribeirão Preto, SP, Brazil). After the daily immersion in red wine for 1 h at 37°C, the test group ligatures were washed in running tap water and stored in distilled water at 37°C for 23 h. The immersion media (red wine and distilled water) were renewed daily for all groups.

### 2.3.3 Brushing protocols

In each experimental group half of the specimens were brushed daily using regular dentifrice, and half were brushed daily using whitening dentifrice containing blue pigment. A manual, single soft-bristled toothbrush (Colgate-Palmolive, New York, NY, USA) was used by a single trained operator. A 1:3 dentifrice:distilled water slurry was used to brush each bracket-ligature specimen for 20 s at the top, bottom, and front bracket surfaces using a toothbrush (1 min total). One toothbrush was used for each 10 specimens in each group. The specimens were maintained in contact with the dentifrice slurry for an additional 1 min after brushing for improved contact of the dentifrice with the ligatures, washed in running tap water, and stored in distilled water at 37°C.

### 2.3.4 Color measurements

Color measurements were repeated after 7, 14, 21, and 28 days of storage. A 28-day exposure period represents the average time between orthodontic clinical appointments. A spectrophotometer (SP60; X-Rite, Grand Rapids, MI, USA) measured ligature color based on the CIEL\*a\*b\* parameters, as previously described.<sup>4</sup> The ligatures were removed from the brackets and the specimen were placed on a standard white background for color measurement (CIE  $L^*$  = 93.2,  $a^*$  = -0.3, and  $b^*$  = 1.6). Three readings per specimen were performed and the values were averaged. The CIEL\*a\*b\* color difference ( $\Delta E$ ) was calculated using the following formula:  $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ , where  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  are the mathematical differences between CIE  $L^*$ ,  $a^*$ , and  $b^*$  at the different storage periods and baseline measurements.

### 2.3.5 Statistical analysis

Data for CIE  $L^*$ ,  $a^*$ , and  $b^*$  and  $\Delta E$  were separately analysed using two-way repeated measures analysis of variance (dentifrice type  $\times$  storage time) separately according to staining agent exposure and ligature brand. All pairwise multiple comparison procedures were performed using the Student-Newman-Keuls' method ( $\alpha=0.05$ ). Statistical analyses were carried out using SigmaStat 3.5 software (Systat Software Inc., Chicago, IL, USA).

## 2.4 Results

Figure 1 shows the CIE  $L^*$  results. The Unitek ligatures exposed to the staining agent had a significantly decreased luminosity at 14 days for the regular dentifrice and 21 days for the whitening dentifrice. Unitek ligatures had similar luminosities between both dentifrice groups. The Morelli ligatures exposed to the staining agent had a continuous significant luminosity loss during immersion and significantly lower luminosity of specimens brushed with regular dentifrice. Ligature groups immersed only in distilled water had minor CIE  $L^*$  changes during immersion and significantly lower luminosity in groups brushed with the whitening dentifrice.

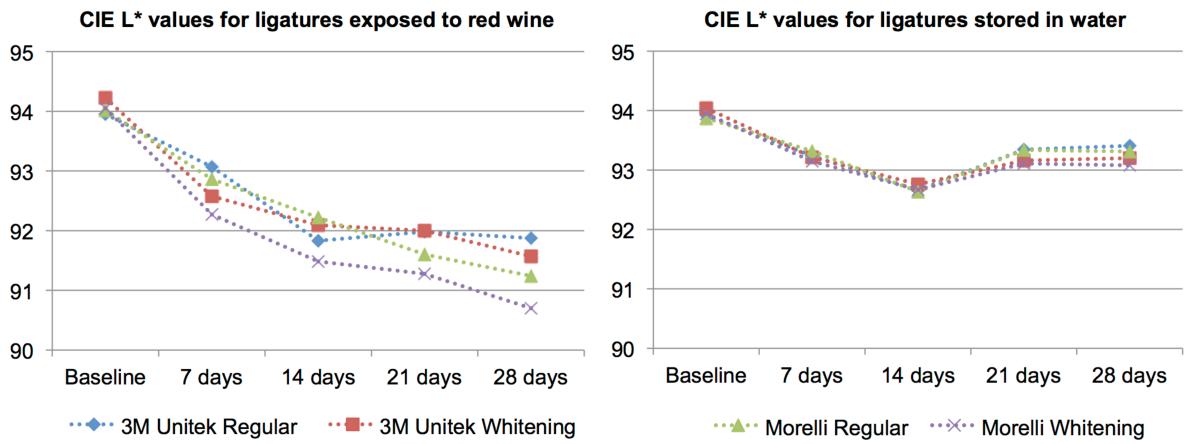


Figure 1. CIE  $L^*$  results under both storage conditions, dentifrice types, and clear ligature brands. Ligatures exposed to the staining agent had a continual significant luminosity loss over time. The whitening dentifrice decreased luminosity compared to the regular dentifrice.

The CIE  $a^*$  results (Figure 2) showed that exposure to the staining agent significantly increased CIE  $a^*$  values over time in both ligature groups, indicating a tendency towards red discoloration. Ligatures brushed with whitening dentifrice had significantly lower CIE  $a^*$  values (less red) than ligatures brushed with regular dentifrice. Control groups had decreased CIE  $a^*$  values with time, indicating a tendency towards green discoloration, particularly in specimens brushed with the whitening dentifrice. Groups exposed to the staining agent also showed increased CIE  $b^*$  over time, indicating an overall yellow discoloration (Figure 3). The increased CIE  $b^*$  was higher when the whitening dentifrice was used. The control groups, by contrast, showed a general decrease in CIE  $b^*$  over time, indicating blue discoloration. Blue discoloration was significantly higher in groups brushed with the whitening dentifrice, irrespective of the elastic ligature tested.

Color variability results are summarized in Table 1. The 3M Unitek ligatures exposed to the staining agent had a significantly increased  $\Delta E$  over time when the whitening dentifrice was used, but specimens brushed with the regular dentifrice had no significant color variation from 7 days onward. The color variation was significantly lower in specimens brushed with regular dentifrice compared to the whitening dentifrice. Morelli ligatures exposed to the staining agent had significantly increased color variation over time for both dentifrice types, with significantly higher color variation in specimens brushed with the whitening dentifrice. Distilled water

immersion only of both ligature brands minimally affected color variation; however, the whitening dentifrice always had higher color variation regardless of ligature type.

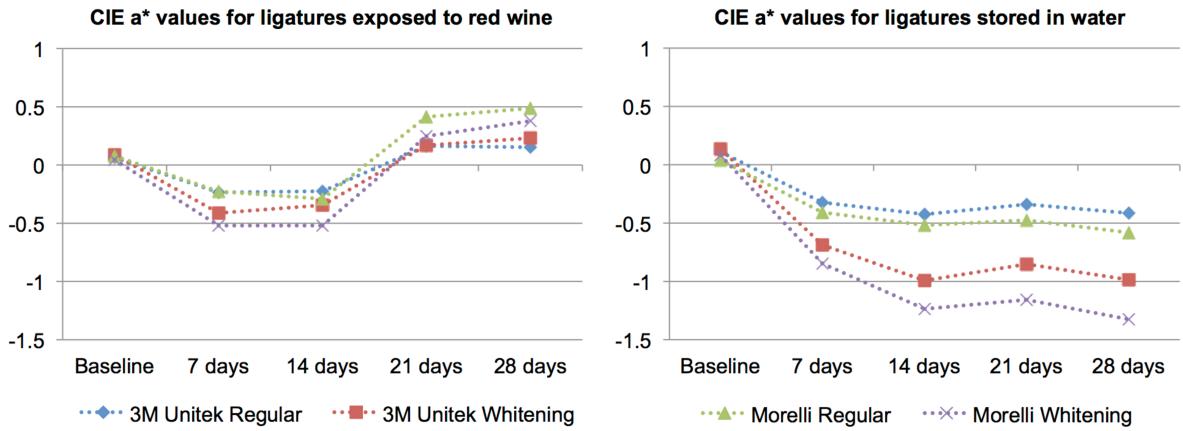


Figure 2. CIE  $a^*$  results under both storage conditions, dentifrice types, and clear ligature brands. Red discoloration was observed over time for exposure to staining agent. Ligatures brushed with whitening dentifrice had significantly lower  $a^*$  values (less red) than ligatures brushed with regular dentifrice.

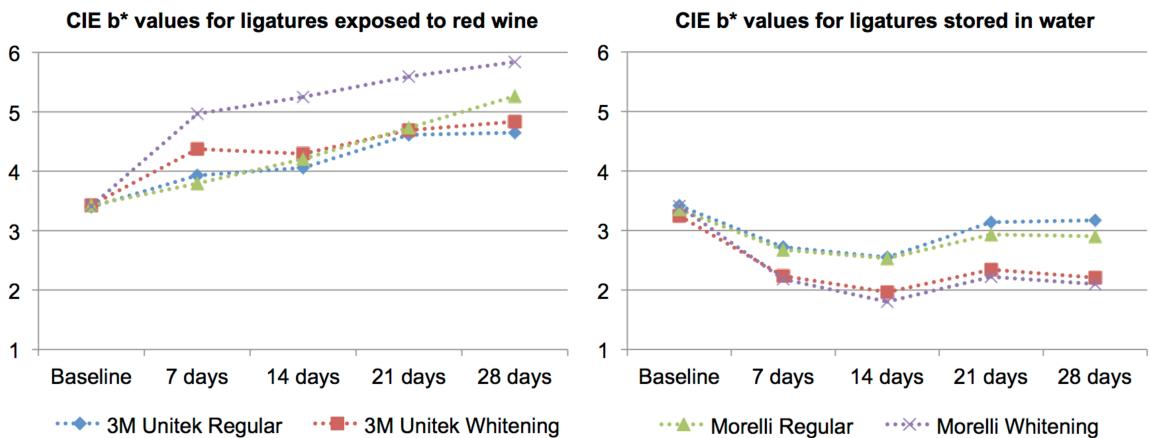


Figure 3. CIE  $b^*$  results under both storage conditions, dentifrice types, and clear ligature brands. Overall, yellow discoloration was observed for ligatures exposed to the staining agent. Ligatures stored in distilled water showed a general decrease in  $b^*$  values over time, indicating a blue discoloration. The blue discoloration was higher in the whitening dentifrice groups.

Table 1. Color variation ( $\Delta E$  means  $\pm$  standard deviations) for each storage time compared with baseline

Ligature brand	Storage condition/Time	Dentifrice type	
3M Unitek	Staining agent	Regular	Whitening
	7 days	1.58 $\pm$ 0.47 Bb	1.97 $\pm$ 0.23 Da
	14 days	2.24 $\pm$ 0.29 Aa	2.35 $\pm$ 0.32 Ca
	21 days	2.32 $\pm$ 0.11 Ab	2.56 $\pm$ 0.24 Ba
	28 days	2.43 $\pm$ 0.10 Ab	3.01 $\pm$ 0.25 Aa
Morelli	Distilled water		
	7 days	1.08 $\pm$ 0.05 Bb	1.55 $\pm$ 0.11 Ca
	14 days	1.65 $\pm$ 0.26 Ab	2.26 $\pm$ 0.23 Aa
	21 days	0.79 $\pm$ 0.13 Cb	1.60 $\pm$ 0.14 Ca
	28 days	0.78 $\pm$ 0.07 Cb	1.75 $\pm$ 0.26 Ba
Morelli	Staining agent		
	7 days	1.27 $\pm$ 0.29 Db	2.43 $\pm$ 0.15 Da
	14 days	1.99 $\pm$ 0.15 Cb	3.21 $\pm$ 0.15 Ca
	21 days	2.76 $\pm$ 0.25 Bb	3.54 $\pm$ 0.16 Ba
	28 days	3.35 $\pm$ 0.11 Ab	4.15 $\pm$ 0.34 Aa
Morelli	Distilled water		
	7 days	0.99 $\pm$ 0.05 Bb	1.74 $\pm$ 0.10 Da
	14 days	1.59 $\pm$ 0.25 Ab	2.45 $\pm$ 0.15 Aa
	21 days	0.86 $\pm$ 0.13 Bb	1.91 $\pm$ 0.17 Ca
	28 days	0.95 $\pm$ 0.11 Bb	2.11 $\pm$ 0.20 Ba

Capital letters in each storage condition column indicate differences between storage times. Lowercase letters in each row indicate differences between dentifrice types ( $\alpha=0.05$ ). Statistical comparisons between storage media or ligature brands were not carried out.

Figure 4 illustrates representative ligatures of each brand subjected to the different storage and brushing conditions; visual differences are particularly apparent based on storage conditions and ligature brand. All ligatures stored in distilled water and brushed with whitening dentifrice had a blue or green appearance; 3M Unitek ligatures stored in water and brushed with regular dentifrice had a yellow appearance. Exposure to red wine caused a red discoloration of both ligature brands.

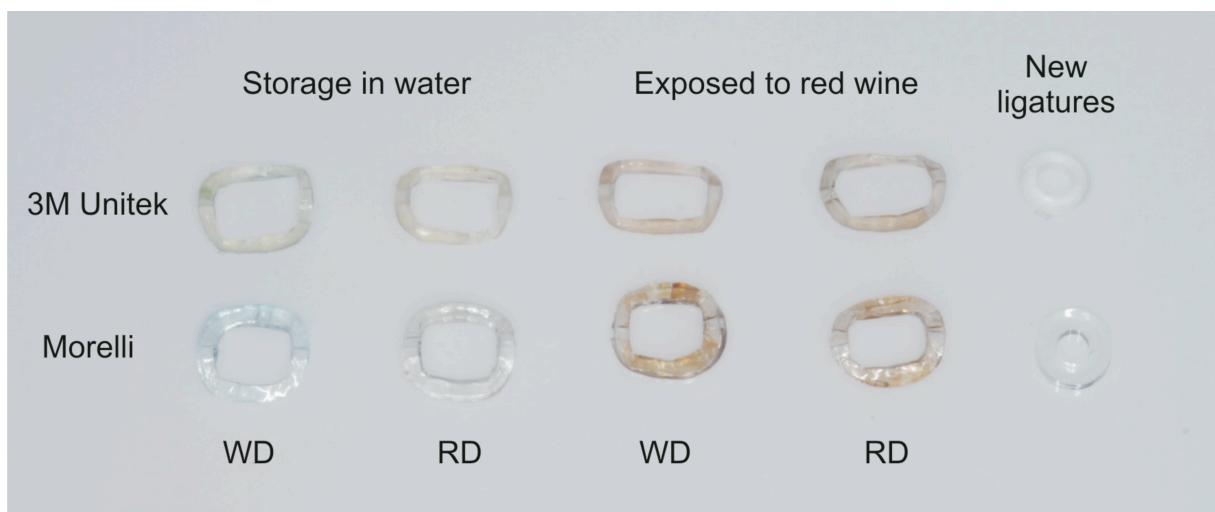


Figure 4. Representative ligatures of each brand subjected to the different storage and brushing conditions. Visually detectable differences are particularly apparent according to storage condition and ligature brand. Ligatures stored in distilled water and brushed with whitening dentifrice had a blue or green discoloration. Exposure to staining agent yielded a red discoloration of both ligature brands.

## 2.5 Discussion

Daily exposure to a red wine staining agent significantly increased the ligature color variability, while immersion in distilled water had a minimal effect. Thus, the first study hypothesis is accepted. Color variation due to red wine exposure results primarily from impregnation of the elastomeric material by dark pigments. The storage conditions also impact the staining susceptibility; it has been shown that chemical degradation and staining characterize early-stage ligature color variation, while simple staining prevails in the later stages.<sup>2</sup> In the present study, the ligatures were tested in a stretched state, i.e., encircling the orthodontic brackets to create a clinically-relevant setup. Staining is more likely to occur under stretching because of improved pigment absorption. In addition, prolonged water immersion of elastomeric materials may also increase staining across the bulk polymer structure. The low-pH of red wine and the presence of ethanol may additionally contribute to pigmentation because they impose chemical challenges to the ligature material and potentially make the ligature surface more prone to pigment absorption.<sup>17</sup>

In addition to decreasing luminosity, exposure to the staining agent caused red and yellow discoloration of the ligatures. These effects are visually perceptible in Figure 4. A previous study similarly indicated that clear elastic ligatures exposed to

dietary media experience primarily yellow color variation.<sup>1</sup> Color variation could be reduced by using a whitening dentifrice, which is theoretically more effective in removing surface stains than regular dentifrices. The present results, however, indicate that the whitening dentifrice increased the ligature color variation. Therefore, the second study hypothesis is rejected. This tendency is likely caused by the blue pigment in the whitening dentifrice used, known as blue covarine, which results in blue discoloration of the ligatures.

Although color variation was greater, red and yellow discoloration was lessened in ligatures brushed with whitening dentifrice than those brushed with regular dentifrice. Therefore, the yellow discoloration of clear ligatures was reduced.<sup>1</sup> Blue pigmented whitening dentifrices have been reported to mask tooth yellowing in a prior study,<sup>16</sup> but the current study is the first reporting that dentifrice type may affect the color stability of orthodontic clear ligatures. Traditional tooth whitening uses peroxide bleaching to alter the intrinsic tooth color or abrasive cleaning to alter extrinsic stain removal.<sup>18</sup> Blue pigmented whitening dentifrices were introduced to mask the tooth yellowing by depositing a thin blue pellicle on the enamel,<sup>16</sup> resulting in perceptibly whitened teeth. The present results indicate that the blue pigment in dentifrice may also impart a blue tint onto orthodontic ligatures. This finding is of clinical interest because a blue discolored ligature may be considered more aesthetic than a yellow tint, and the aesthetic decline in clear ligatures over time might be less severe.

As reported in a previous study, the staining of clear elastics depends not only on the exposure time and storage medium, but also on the material composition.<sup>4</sup> Elastic ligatures manufactured using injection mouldings were reportedly more resistant to color changes than those made by extrusion.<sup>1</sup> In the present study, no statistical comparisons were performed between ligature brands, but 3M Unitek ligatures presented less color variation after 28 days of staining agent exposure than the Morelli ligatures. Such a difference according to ligature material may reflect the differing manufacturing processes affecting porosity, surface texture, and overall material quality, which all affect pigment absorption.

A few studies have evaluated the color stability of clear orthodontic ligatures;<sup>1, 2, 4</sup> some expose the ligatures to severe staining protocols, such as methylene blue solution for up to five days<sup>2</sup> or dietary media for up to 72 h.<sup>1</sup> A 1-h daily exposure to red wine might also be considered a severe staining condition, as diet-mediated

pigmentation is caused by continuous low-level exposure to oral pigments. The blue tint imparted by the whitening dentifrice and the potentially improved ligature aesthetic could be even greater in clinical practice. Under clinical conditions, patients may consider blue discolored ligatures more aesthetically appealing than yellow colored, but this effect should be confirmed clinically. Maintaining a longer aesthetic interval may not only reduce the frequency of dental appointments and financial costs, but it may potentially reduce the negative social impact of anaesthetic orthodontic devices.

## **2.6 Conclusions**

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

- Exposure to a staining agent increased the color variation of orthodontic elastic ligatures;
- Use of a whitening dentifrice containing blue pigment did not improve ligature color stability, but it decreased yellow discoloration and increased a blue coloration.

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### **3 Capítulo 2**

Impact of bracket displacement or rotation during bonding and time of removal of excess adhesive on the bracket-enamel bond strength<sup>1</sup>

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### 3.1 Abstract

**Objective:** This study investigated the influence of bracket displacement or rotation during fixation and time of removal of excess adhesive from around the brackets on their bond strength to enamel. **Methods:** Stainless steel brackets were bonded to the buccal faces of bovine incisors using Transbond XT® adhesive resin. The teeth were divided into five groups ( $n = 20$ ). In the control group, no displacement or rotation of the bracket was carried out. In the Displac-A group, excess adhesive was removed after the bracket was displaced 2 mm incisally. In the B-Displac group, excess adhesive was removed before the bracket was displaced incisally. In the Rotat-A group, excess adhesive was removed after the bracket was rotated 45°. In the B-Rotat group, excess adhesive was removed before the bracket was rotated. Photoactivation was carried out on the lateral sides of the bracket. A shear test was conducted 10 min after fixation using a knife-edged chisel. Bond strength data were analyzed using ANOVA and Fisher's test (5%). The adhesive remnant index (ARI) was scored under magnification. ARI data were analyzed using the Kruskal-Wallis test (5%). **Results:** No significant differences were detected among the Control, Displac-A, Rotat-A, and B-Rotat groups. The B-Displac group showed lower bond strength than all of the other groups, except Displac-A. No significant differences were observed in ARI scores across groups. **Conclusions:** Displacements of the brackets during fixation did not seem to affect the enamel bond strength when excess adhesive is removed after the final positioning of the bracket.

**Keywords:** Bonding; displacement; excess adhesive; rotation; shear strength.

### 3.2 Introduction

The development of adhesion to tooth structures resulted in significant evolution in the methods used in the attachment of orthodontic appliances. Adhesion allows for the direct bonding of brackets to dental surfaces (Schnebel et al. 2012; BenGasseem et al. 2013), reducing the need for placing orthodontic bands. Concurrently, alterations in the design of brackets and improvements in orthodontic treatments took place in recent decades. In the late 1960s, the first pre-adjusted orthodontic appliance system was introduced, including brackets with pre-adjusted torque and angulation values that eliminated the need for bending wires to place

teeth in the proper orientation. It was characterized as the widespread Straight-Wire technique (Andrews, 1976a; Andrews, 1976b).

Systems that use pre-adjusted brackets, such as Straight-Wire® (Andrews, 1976a; Andrews, 1976b), Tip-Edge® (Parkhouse, 2003), and MBT® systems (McLaughlin et al. 2001) simplify orthodontic practice. These systems, however, require high accuracy when positioning the brackets for bonding, as even small placement errors may lead to inadequate tooth alignment (Andrews, 1976b; Dellinger, 1978). The position of the bracket on the crown affects the final tip, torque, height, and rotation of the tooth (Carlson at al. 2001). It is known that a poorly positioned bracket in the horizontal axis may lead to undesired tooth rotation (Magness 1978). In addition, inadequate positioning in the vertical axis may cause unevenness of the tooth crown or torque alterations, possibly interfering with the buccal-lingual inclination (McLaughlin et al. 1995; van Loenen et al. 2005). Poor positioning of brackets might ultimately result in the need for more archwire adjustments, increasing treatment time.

When clinicians attempt to find the optimal position at which to place and bond a bracket, displacing or rotating the bracket on the tooth surface is commonly necessary. Findings of a recent study indicate that the positioning of brackets may vary according to the type of bracket when using a direct bonding technique (Birdsall et al. 2012). Moving the bracket during bonding could displace the adhesive resin, which could interfere with the proper interlocking of the bonding agent with the acid-etched enamel. After a comprehensive literature review, no evidence could be found regarding the effect of moving the brackets during their fixation on the bond strength to enamel. In addition, another factor that could interfere with the bonding of brackets is the removal of excess adhesive agent from around the brackets before or after the bracket is positioned.

The aim of this study was to investigate the implications of bracket displacement or rotation during fixation as well as removing excess adhesive agent before or after moving the bracket on the early bond strength to enamel. The hypothesis tested was that bracket movement or the removal time of excess resin would not interfere with bond strengths.

### 3.3 Materials and Methods

This *in vitro* study involved a complete randomized and blinded design, considering the effect of different bonding conditions of brackets on their bond strength to enamel. The roots of 100 recently extracted bovine incisors were embedded in acrylic resin, leaving the outermost aspect of the buccal faces perpendicular to the horizontal plane. After prophylaxis with water-pumice slurry, the teeth were dried. Next, the buccal faces were etched for 30 s using 37% phosphoric acid gel. The etched surfaces were cleansed with air-water spray for 30 s and dried with compressed air. Stainless steel Edgewise brackets with a slightly curved base were used (Morelli, Sorocaba, SP, Brazil). A standard volume of the adhesive resin Transbond XT® (3M Unitek, Monrovia, CA, USA) was applied to the bracket base. The teeth were divided into five groups ( $n = 20$ ), according to the bonding protocol tested.

- *Control group*: The bracket was positioned and seated firmly in the center of the buccal face using a direct bond bracket tweezers. The archwire slot was perpendicular to the long axis of the tooth. Excess adhesive was removed from all bracket-enamel margins with a dental explorer. Photoactivation was carried out for 20 s on both lateral sides of the bracket using a light-emitting diode curing unit (Radii®; SDI, Bayswater, Victoria, Australia) with  $800 \text{ mW/cm}^2$  irradiance.
- *Displac-A group*: The bracket was positioned and seated firmly in the buccal face. It was then displaced 2 mm incisally, as measured with the aid of a marked periodontal probe. After the bracket was seated firmly again, excess adhesive was removed prior to photoactivation.
- *B-Displac group*: The bracket was positioned and seated firmly in the buccal face. Excess adhesive was removed before the bracket was displaced 2 mm incisally. The bracket was seated firmly again prior to photoactivation.
- *Rotat-A group*: The bracket was positioned and seated firmly in the buccal face with the archwire slot inclined at an angle of  $45^\circ$  to the long axis of the tooth. The bracket was rotated  $45^\circ$  to position the slot perpendicular to the long axis of the tooth. The slightly curved base did not affect the bracket rotation. After the bracket was seated firmly again, excess adhesive was removed prior to photoactivation.

- ***B-Rotat group:*** The bracket was positioned and seated firmly in the buccal face with the archwire slot inclined at an angle of 45° to the long axis of the tooth. Excess adhesive was removed before the bracket was rotated 45°. The bracket was seated firmly again prior to photoactivation.

The same operator carried out all bonding procedures. The shear bond strength test was conducted 10 min after fixation. In the meantime, the specimens were stored in distilled water at 37°C. The teeth were positioned in a mechanical testing machine (model DL500; EMIC, São José dos Pinhais, PR, Brazil). The archwire slot was parallel to the horizontal plane. A knife-edged chisel was placed in contact with the upper bracket-enamel interface parallel to the long axis of the tooth. The specimen was submitted to a compressive load at a crosshead speed of 0.5 mm/min until failure. Bond strength values were calculated in MPa. Data were statistically analyzed by Analysis of Variance. All *post hoc* pairwise multiple comparison procedures were carried out by Fisher's LSD method ( $p < 0.05$ ).

The debonded specimens were observed in a stereomicroscope under a 40× magnification to score the adhesive remnant index (ARI) as follows: 0 = no bonding resin left on the tooth, 1 = less than half of the bonding resin left on the tooth, 2 = more than half of the bonding resin left on the tooth, and 3 = all bonding resin left on the tooth, with distinct impression of the bracket mesh. ARI data were statistically analyzed by the non-parametric Kruskal-Wallis test ( $p < 0.05$ ).

### 3.4 Results

Results for shear bond strength to enamel are shown in Table 1. No significant differences were detected among the Control, Displac-A, Rotat-A, and B-Rotat groups ( $p \geq 0.108$ ). The B-Displac group showed significantly lower enamel bond strength than all other groups ( $p \leq 0.016$ ), except when compared with Displac-A ( $p = 0.162$ ). Results for ARI scores are shown in Figure 1. No significant differences were observed across groups ( $p = 0.644$ ). However, scores 0 and 3 were not observed for the B-Displac group.

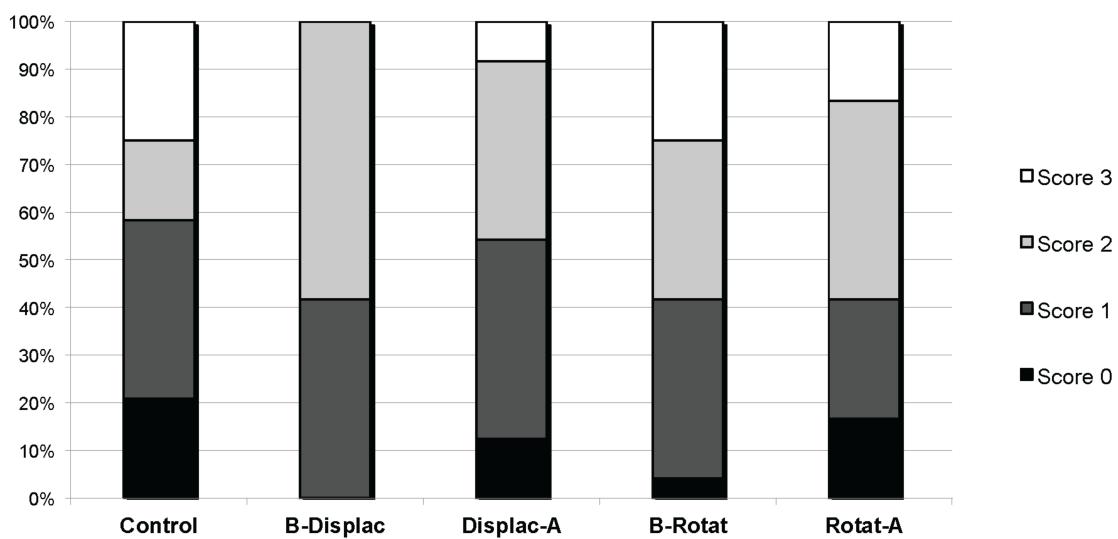


Figure 1. Results for ARI scores. No statistically significant differences were observed across groups.

### 3.5 Discussion

Results of the present study provide evidence that slight bracket displacement to correct positioning of orthodontic devices in relation to the long axis of the tooth can be carried out without jeopardizing the immediate bond strength to enamel if excess adhesive is not removed before displacement. The hypothesis tested was rejected, as B-Displac showed lower enamel bond strength than most of the other groups. Removal of excess adhesive before displacement might lead to a lack of adhesive agent for filling the enamel microporosity left by the acid etching step. When the resin is not removed and the bracket is displaced vertically, the excess adhesive is probably sufficient to infiltrate the etched enamel and create mechanical interlocking for bonding. This situation may only occur when the excess adhesive is removed from around the brackets while the adhesive agent is still unpolymerized (Bishara et al. 2000).

The other situation addressed in the present study was the rotation of the bracket on its own axis. The rotation had no significant influence on enamel bonds. In addition, in such a bonding scenario, the time of removal of excess adhesive did not influence bond strengths. This result can probably be explained by the fact that, given that the bracket was not displaced from its original position in the crown, the adhesive agent available at the bracket-enamel interface was sufficient to infiltrate the enamel microporosity. In addition, rotation may improve the interlocking of the resin with the bracket mesh, as hand pressure is applied again to the bonded device.

In the search for the ideal positioning of the bracket in the center of the clinical crown, clinicians might need slight displacements during the bonding procedures. Results of a previous study (Armstrong et al. 2007) showed that bracket bonding that is guided by measuring the distance from the incisal edge resulted in better placement for anterior teeth compared with localizing the center of the clinical crown. However, the authors stated that archwire bending or bracket repositioning would still be necessary to compensate for the inaccuracies in both techniques. The present study indicates that rotation of the bracket is less harmful to its bond strength to enamel than the vertical displacement of the bracket during fixation. In addition, there is a lower likelihood of interfering with the bond strength if the excess adhesive resin is removed after positioning the bracket.

Table 1. Results of shear bond strength to enamel (MPa) for all groups\*

	Control	Displac-A	B-Displac	Rotat-A	B-Rotat
Mean	9.7	9.1	8.2	9.8	10.1
Standard deviation	1.8	2.1	1.6	2.2	2.0
Minimum	6.9	5.5	5.5	5.9	7.0
Maximum	13.2	13.5	11.3	13.7	13.1
95% confidence interval	8.9-10.5	8.1-10.1	7.5-8.9	8.8-10.8	9.2-11.0
Statistical grouping**	a	ab	b	a	a

\*Groups labeled as -A or B-: excess adhesive resin was removed after or before displacement or rotation of the bracket during fixation.

\*\*Distinct letters indicate significant differences between groups ( $p < 0.05$ ).

*In vitro* testing of dental materials has inherent limitations. It should be acknowledged, for instance, that only immediate enamel bonds were assessed here. Immediate testing was carried out to investigate whether the different positioning scenarios could interfere with the initial attachment of the orthodontic devices to the dental structure. In addition, the bond strength values and ARI scores recorded when debonding loads are applied using a mechanical testing machine might differ from those recorded when debonding pliers are used for debonding (Bishara et al. 1994). Further studies on the longevity of the enamel bonds provided by different bonding approaches are warranted, perhaps using thermal cycling to simulate the aging that orthodontic brackets are subjected in the intraoral environment.

### **3.6 Conclusion**

Displacement of orthodontic brackets during fixation did not seem to affect the bond strength to enamel when excess bonding agent is removed after the final positioning of the bracket on the dental surface.

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## **4 Capítulo 3**

Investigation of a modified photo-activation protocol using two simultaneous light-curing units for bonding brackets to enamel<sup>1</sup>

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#### 4.1 Abstract

This study investigated the effect of a modified photo-activation protocol using two simultaneous light-curing units on the shear bond strength (SBS) of brackets to enamel. Metal brackets were bonded to bovine incisors using the resin-based agent Transbond XT (3M Unitek). Four photo-activation protocols of the bonding cement were tested ( $n=15$ ): Control\_ photo-activation for 10 s on each proximal face of the bracket at a time; Simultaneous\_ photo-activation for 10s on both proximal faces of the bracket at the same time; One side-20s\_ photo-activation for 20 s at one proximal face of the bracket only; and One side-10s\_ photo-activation for 10 s only at one proximal face of the bracket. SBS was tested immediately or after 1000 thermal cycles. Adhesive remnant index (ARI) was classified. Data were subjected to two-way ANOVA and Student-Newman-Keuls' test ( $\alpha=0.05$ ). Pooled means  $\pm$  standard deviations for SBS to enamel (MPa) were:  $10.2\pm4.2$  (Control)<sup>a</sup>,  $9.7\pm4.5$  (Simultaneous)<sup>a</sup>,  $5.6\pm3.1$  (One side-20s)<sup>b</sup>, and  $4.6\pm1.9$  (One side-10s)<sup>b</sup>. Pooled data for immediate and thermal cycled groups were  $6.3\pm2.6^b$  and  $8.8\pm5.2^a$ . A predominance of ARI scores 1-2 and 0-1 was observed for the immediate and thermal cycled groups. In conclusion, simultaneous photo-activation of the orthodontic cement using two light-curing units at each bracket proximal face yielded the same bonding ability compared to the conventional light-curing method. Photo-activation of the bonding agent at one proximal face of the bracket only is not recommended, irrespective of the light-curing time used.

**Keywords:** LEDs; orthodontic brackets; shear bond strength.

#### 4.2 Introduction

The development of adhesive materials has significantly facilitated the clinical practice in orthodontics (1,2). The use of photo-activated resin-based agents in the bonding of orthodontic devices made the process more accurate, as the setting time of the cement can be controlled. Polymerization of the material used for bracket fixation depends on the access to curing light; however, orthodontic devices in general block the direct passage of the light electromagnetic spectrum. For this reason, multiple light exposures of the bracket-enamel interfaces are required to

deliver a minimum radiant exposure (energy dose) for appropriate curing of the bonding material (3).

Multiple light exposures increase the chair time of orthodontic dental treatments, which is inconvenient for the professional, uncomfortable for patients and, often, makes orthodontics unfeasible for children. In general, each orthodontic device applied needs between 20 and 40 seconds of photo-activation for adequate polymerization (4,5). The decrease of such time may cause a considerable reduction in the bond strength to enamel and consequent clinical problems such as premature debonding. The development of new photo-activation materials and/or protocols able to reduce the time to light exposure without interfering in the bond strength is therefore relevant.

Improvements in the polymerization promoting system of resin-based cements was recently indicated as an alternative to optimize the photo-activation protocols in orthodontics (6). However, caution is necessary, as the exaggerated acceleration of the chemical reactions may increase the polymerization stress (7) and interfere with the bonding to enamel. Another approach to accelerate the curing process is the change of light source. High-irradiance light-curing units (e.g.: plasma arc) have already been tested (8,9), but they have not presented a satisfactory response mainly because these units are not cost-effective and generally demand expensive maintenance.

Current curing units based on light-emitting diodes (LEDs) are less expensive and deliver higher irradiance than a few years ago. Thus, clinical approach of simultaneously using more than one LED unit for photo-activation procedures seems feasible in orthodontics. The rationale is to expose two sides of the bracket simultaneously to light, reducing in half the time spent with the photo-activation. Nonetheless, there is no clinical or laboratory evidence available concerning the effectiveness of such approach. There always a risk for increased polymerization stress, thus investigation of that alternative curing method is necessary before clinical application.

The aim of this study was to investigate the effect of using two simultaneous LED-based light-curing units during the photo-activation of orthodontic cement in the bond strength of brackets to enamel. Other curing methods were tested for comparison. The study hypothesis is that the simultaneous photo-activation has no negative impact on the bonding of brackets to enamel.

## 4.3 Materials and Methods

### 4.3.1 Experimental Design

This *in vitro* study involved a 4×2 factorial design, in which the studied factors were: photo-activation protocol of the orthodontic bonding agent (four levels: control, simultaneous photo-activation, photo-activation in only one side of the bracket for 20 s or for 10 s) and storage time of the specimens before the test (two levels: immediate testing or after aging by thermal cycling). The response variables were shear bond strength to enamel (MPa) and failure modes scored by the Adhesive Remnant Index (ARI) method (10). In each group, 15 specimens were tested.

### 4.3.2 Bonding Procedures

In total, 120 bovine permanent incisors recently extracted were used. The teeth had their roots embedded in PVC tubes for the crown buccal face to be in a perpendicular position to the horizontal level. After cleaning the surfaces with prophylactic paste and water, the buccal faces were dried and acid-etched using 37% phosphoric acid for 15 s, washed with air/water spray for 30 s, and dried with compressed air.

Stainless steel edgewise metallic brackets (Morelli Ortodontia, Sorocaba, SP, Brazil) were fixed on the buccal faces using the photo-activated resin-based orthodontic cement Transbond XT (3M Unitek, Monrovia, CA, USA). For all groups, after acid-etching, the bonding agent was applied to the bracket base and the bracket was hand-pressed to the center of the buccal face using a direct bond bracket tweezers. Excess cement was removed from all bracket-enamel margins with a dental explorer. Four different photo-activation protocols were tested (30 specimens per group):

- Control (two sides): the bonding agent was photo-activated for 20 s, 10 s on each proximal face of the bracket, using a LED curing unit (Radii Cal; SDI, Bayswater, Victoria, Australia) with irradiance of 1400 mW/cm<sup>2</sup>;
- Simultaneous (two sides): the bonding agent was photo-activated for 10 s on each proximal face of the bracket simultaneously using two similar light-curing units with same irradiance level;

- One side 20s: the bonding agent was photo-activated for 20 s on only one proximal face of the bracket;
- One side 10s: the bonding agent was photo-activated for 10 s on only one proximal face of the bracket.

#### **4.3.3 Bond strength test and failure analysis**

The same operator carried out all bonding procedures. After bonding, 15 specimens from each photo-activation method group were immediately tested (up to 10 min between bonding and testing). The other 15 specimens were tested after 1000 thermal cycles, which involved alternated immersion in water at  $5\pm5^{\circ}\text{C}$  and  $55\pm5^{\circ}\text{C}$  (30 s dwell time). For the shear bond strength test, a mechanical testing machine was used (DL500; EMIC, São José dos Pinhais, PR, Brazil). A knife-edged chisel was placed at the tooth-bracket interface and a compressive load was applied at a 0.5 mm/min crosshead speed until failure of the bonding. Bond strength values were calculated in MPa considering the bracket base area. After the test, the teeth surfaces were observed in a stereo microscope, at  $\times 40$  magnification, for classification of the ARI scores (10):

- Score 0: no amount of adhesive attached to enamel;
- Score 1: less than half of adhesive attached to enamel;
- Score 2: more than half of adhesive attached to enamel;
- Score 3: all adhesive attached to enamel.

#### **4.3.4 Statistical analysis**

Enamel bond strength data were transformed to ranks and submitted to a two-way Analysis of Variance (photo-activation protocol  $\times$  storage time). All pairwise multiple comparison procedures were carried out using the Student-Newman-Keuls' method. ARI data were analyzed by Kruskal-Wallis test separately for each storage time, while ARI individual comparisons between the storage times within each photo-activation protocol were carried out using the Wilcoxon test. The analyses were carried out using the SigmaStat 3.5 software (Systat Software Inc., San Jose, CA, USA). The significance level  $\alpha=0.05$  was set for all analyses.

#### 4.4 Results

The results of the bonding resistance test are presented in Table 1. The factors ‘photo-activation protocol’ ( $p<0,001$ ) and ‘storage time’ ( $p=0,003$ ) were significant, while the interaction between the factors was not significant ( $p=0,694$ ). Thus, the data are presented in groups for each factor. The control and simultaneous methods were similar to each other, both generating bonding resistance to enamel significantly higher than the other methods. Thus the study hypothesis was confirmed.

Table 1. Means (standard deviations) of enamel bond strength, MPa (n=15)

Photo-activation protocol	Storage time		<i>Pooled data</i>
	Immediately	Thermal cycled	
Control (two sides)	7.9 (2.1)	12.5 (4.5)	10.2 (4.2) a
Simultaneous (two sides)	8.3 (1.9)	11.2 (5.9)	9.7 (4.5) a
One side 20 s	4.9 (2.2)	6.4 (3.7)	5.6 (3.1) b
One side 10 s	4.2 (1.5)	5.1 (2.2)	4.6 (1.9) b
<i>Pooled data</i>	6.3 (2.6) B	8.8 (5.2) A	

Distinct uppercase letters indicate significant differences between the storage times; distinct lowercase letters indicate significant differences between the photo-activation protocols ( $p<0.05$ ).

The IAR analysis results are shown in Picture 1. No statistically significant differences have been observed between the photo-activation protocols in the immediate ( $p=0,589$ ) and thermocycled times ( $p=0,481$ ). In the individual comparisons for each photo-activation protocol (immediate × thermocycled), no differences for the control ( $p=0,173$ ), one side 20s ( $p=0,946$ ) and one side 10s ( $p=0,240$ ) were observed. For the simultaneous group, there was a significant difference between the groups ( $p=0,027$ ), with a predominance (80%) of scores 2 and 3 in the immediate time and predominance of scores 0 and 1 (60%) after thermocycling.

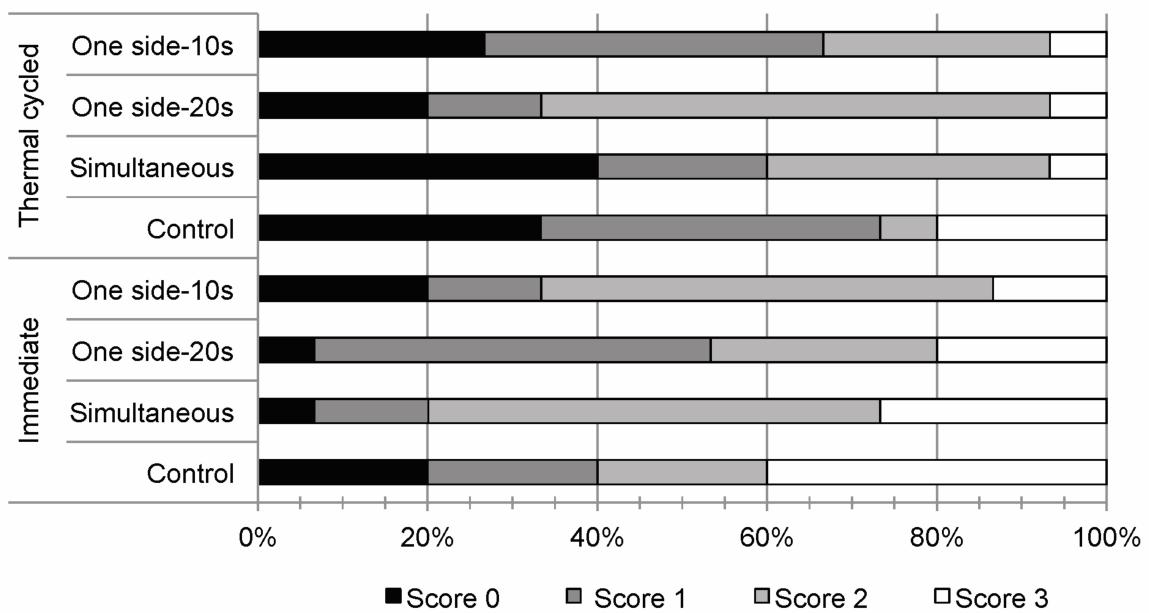


Figure 1. Frequency of ARI scores. For the simultaneous group, there was significant difference between the immediate and the thermal cycled groups, while no other statistically significant difference was detected

#### 4.5 Discussion

The higher bonding resistance observed for the protocols which involved photo-activation in both sides of the bracket (individual or simultaneous) is explained due to higher exposure of the whole cement agent to light because of photo-activation on the two side surfaces. The brackets interfere in the irradiance (light intensity) and the quality of incident light on the cement which is interposed between the bracket and the tooth, even when ceramic brackets are used (11). Thus, when applying light in both side faces, a bigger area of the cement is reached by the light, resulting in a higher degree of C=C conversion, that is, bigger polymerization extension. As the bonding of the bracket to the enamel depends on the correct polymerization of the cementation agent, only one cement area is properly polymerized when the photo-activation is carried out in only one side of the bracket, regardless the usage of 10 or 20s exposure. It has been previously shown that the conversion degree of C=C of a polymer has a direct effect in the resistance of the bonding of resinous materials to dental substrates (12).

The simultaneous photo-activation of the two faces of the bracket using individual light sources brought bonding results similar to the control group. This finding is interesting concerning the reduction of clinical time needed for the brackets

photo-activation. The possible risk associated to the use of this protocol would be causing a higher polymerization stress in the adhesive interface, as both sides would be simultaneously polymerizing and thus, causing stress at the same time. In this scenario there could be some “competition” between the stresses caused in each axial position of the cement in the adhesive interface (13), which could result in disunity in areas in which the adherence were more fragile. Although it might not be ruled out that this mechanism has occurred in the material microstructure and in the ultra morphology of the bonding to the tooth, the results presented here indicate that there was no loss to the bonding of brackets to enamel.

The results of the bonding resistance test also indicate that the bonding resistance to enamel was significantly higher after thermocycling. In general it is observed that the thermocycling has a deleterious effect on the bonding of brackets to enamel (14), which is usually attributed to the degradation of the polymer during ageing and, mainly, the stress caused in the restoring interface due to the constant changes of temperature. The thermal changes tend to cause stress as the different materials which comprise the bonding interface have different thermal expansion coefficients and as a result they reduce (cold) and expand (heat) in a different way during cycling, resulting in stress in the interface. What may explain the positive result of thermocycling in this study is, initially, the low number of cycles, which are probably not sufficient to generate sufficient stress to interfere in the bonding of the materials involved. However, evidence is found in the literature that the bonding resistance of brackets to enamel might not be affected by thermocycling (15-17).

On the other hand, as there was a higher bonding resistance to enamel after thermocycling, a likely explanation is that the cements had a higher C=C conversion during storage comparing to the immediate group. Previous studies show that the C=C conversion degree of polymeric composites tends to increase in the first 24 to 48 hours after photo-activation, mainly in the presence of heat, even in warmer temperatures as in the oral cavity (17). This “late” polymerization may explain the higher retention after ageing, as the adequate formation of the polymeric network is the main factor with a role in the bonding of the cement to the bracket mesh and the dental tissue.

The IAR analysis result for the simultaneous group can also be explained due to higher polymeric conversion after thermocycling, making the cement more interwoven in the bracket mesh and, when tested, to be mechanically retained to the

orthodontic device without leaving any remains (or leaving just a little) remains of cement in the tooth.

From the clinical point of view, the results of the present study are relevant concerning the use of simultaneous photo-activation protocols in two sides of the bracket. It seems reasonable to indicate that the use of two lights sources simultaneously has no deleterious effect on the bonding of orthodontic devices and, therefore, can be stimulated. On the other hand, the simplification of adhesive procedures through light exposure of only one face of the bracket is not a recommended procedure, as it tends to reduce the bonding capacity of the devices to the enamel.

Considering the limitations of the present in vitro study, it has been concluded that the simultaneous photo-activation of the orthodontic cement in both sides of the bracket during bonding to enamel seems not to have any negative effect on the bonding resistance of the orthodontic device to the dental surface. Besides, the photo-activation of the orthodontic cement in only one side of the bracket is not recommended.

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## **5 Capítulo 4**

Sal de Iodônio para reduzir o tempo de fotoativação na colagem de braquetes ortodônticos<sup>1</sup>

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## 5.1 Resumo

Esse estudo avaliou a adição de sal de iodônio para reduzir o tempo de fotoativação na colagem de braquetes ortodônticos. Um cimento experimental foi formulado utilizando monômeros dimetacrilatos, nanopartículas de sílica, canforoquinona e amina. Hexafluorofosfato difeniliodônio foi adicionado em concentrações de 0, 1 e 2 mol%. Braquetes foram colados em incisivos bovinos com tempos de fotoativação de 5, 10 e 20s. Foram analisados a resistência ao cisalhamento e o IRA. Em todos os tempos de fotoativação os cimentos com adição de sal de iodônio apresentaram diferenças em relação ao cimento controle. Incorporação do sal de iodônio apresentou resultados promissores.

**Keywords:** Fotopolimerização. Braquete ortodôntico. Cimento resinoso. Materiais dentários. Cisalhamento.

## 5.2 Introdução

A redução do tempo de polimerização na colagem de dispositivos ortodônticos pode ocasionar perda considerável na resistência de união do braquete ao esmalte dentário e consequentes problemas clínicos. Dessa forma, tem sido um desafio o desenvolvimento de novos materiais que permitam reduzir o tempo de fotoativação, mantendo o grau de conversão de C=C do polímero e, assim, não interferindo na resistência de união dos braquetes a esmalte (OESTERLE et al., 1995; STAUDT et al., 2006). A alteração no sistema de iniciação é alternativa viável para otimização do tempo de polimerização, entretanto, exige cautela pois a aceleração exagerada das reações químicas envolvidas pode acarretar em tensão de polimerização na interface de união (RUEGGEBERG, 1999; STANSBURY, 2000). O uso de um sal de iodônio como co-iniciador de polimerização radicalar de metacrilatos por meio da utilização conjunta com canforoquinona (CQ). Esta associação permite melhor aproveitamento dos radicais livres gerados pela fotoativação (OGLIARI et al., 2007; CRIVELLO; LAM, 1977). Já existem relatos na literatura de testes positivos do uso de sal de iodônio em diversos agentes resinosos fotoativados, e o aprimoramento deste novo sistema de iniciação é fundamental para uma resposta clínica consolidada (COSTA et al., 2014; GONÇALVES et al., 2013).

Assim, este estudo tem como objetivo avaliar a adição de sal de iodônio a compósitos adesivos fotoativados para reduzir o tempo de fotoativação na colagem de braquetes ao esmalte.

### **5.3 Metodologia**

#### **5.3.1 Preparo dos agentes de cimentação**

Um cimento ortodôntico experimental foi formulado pela mistura dos monômeros dimetacrilato do éster de bisfenol-A glicidila, dimetacrilato de trietenoglicol e dimetacrilato de uretano (Esstech, Essington, PA, EUA) na razão em massa de 65:10:25. A matriz inorgânica foi composta por 35% em massa de nanopartículas (7 nm) sinalizadas de sílica (Aerosil, Evonik, Hanau, Alemanha). O sistema fotoiniciador foi composto por CQ (fotoiniciador) e 4-(dimetilamino)-benzoato de etila (co-iniciador), nas concentrações de 0,5 e 1 mol%, respectivamente. Três materiais foram obtidos pela adição das seguintes concentrações do sal hexafluorfosfato de difeniliodônio (Sigma-Aldrich, St. Louis, MO, EUA): 0 (controle), 1 mol% ou 2 mol%.

#### **5.3.2 Fixação dos braquetes ao esmalte**

Para a realização deste estudo foram utilizados incisivos bovinos recentemente extraídos, que tiveram suas raízes incluídas em tubos de PVC de maneira que a face vestibular da coroa ficasse perpendicular ao plano horizontal. Após limpeza com pasta profilática e água, as superfícies vestibulares dos dentes foram secas e condicionadas com ácido fosfórico 37% por 30 s, lavados com spray ar/água por 30 s e secas com ar. Braquetes metálicos de aço inoxodável para incisivos (Morelli Ortodontia, Sorocaba, SP) foram fixados utilizando os três diferentes materiais experimentais. O cimento era aplicado sobre a base do braquete e este pressionado sobre o centro da face vestibular do dente, utilizando-se uma pinça de apreensão. Após remoção do excesso com sonda exploradora, a fotoativação era realizada utilizando fotopolimerizador do tipo LED (Radii; SDI, Bayswater, Victoria, Austrália) com irradiação de 800 mW/cm<sup>2</sup>. Três tempos de fotoativação foram testados para cada face proximal do braquete: 5, 10 ou 20 s, totalizando 10, 20 ou 40 s de fotoativação de cada dispositivo. Para cada grupo foram testados 12 espécimes.

### **5.3.4 Resistência de união e modos de falha**

Os testes foram realizados em máquina de ensaios mecânicos (DL500; EMIC, São José dos Pinhais, PR) com velocidade de 0.5mm/min. Um cinzel era posicionado na face proximal do braquete e uma carga de compressão aplicada até a falha da união. Os valores de resistência de união ao cisalhamento foram calculados em MPa considerando a área da base do braquete. Os dados foram submetidos à Análise de Variância de duas vias e teste de Student-Newman-Keuls ( $\alpha=0,05$ ). Após o teste, o índice de adesivo remanescente (IAR) na superfície dental foi sob aumento ( $40\times$ ) foi classificado conforme previamente descrito (ARTUN; BERGLAND, 1984). Os dados de padrão de falha foram submetidos ao teste não-paramétrico de Kruskal-Wallis ( $\alpha=0,05$ ).

## **5.4 Resultados e Discussão**

Os resultados do teste de resistência de união estão apresentados na Tabela 1. Foi possível observar que a adição de sal de iodônio ao sistema de iniciação proporcionou maior resistência de união dos braquetes ortodônticos ao esmalte para quase todos os tempos de fotoativação quando comparados ao grupo controle. Os resultados mostraram, também, que esse aumento de resistência de união foi mais significativo nos tempos reduzidos de fotoativação. Este resultado se explica por maior grau de conversão de C=C em tempo menores de exposição à luz à luz. Embora o sal não possa absorver energia luminosa na faixa de comprimento de onda emitido pelo LED azul (em geral entre 400 e 500 nm), o sal atua como terceiro componente da mistura. Esta ação em geral envolve reação com radicais inativos da CQ formados durante a polimerização, diminuindo a taxa de terminação e aumentando a taxa de iniciação da polimerização radicalar, consequentemente aumentando a reatividade da polimerização comparado ao material que contém apenas o sistema binário de fotoiniciação (PADON; SCRANTON, 2000; KIM; STANSBURY, 2009).

Tabela 1. Médias (desvio-padrão) de resistência de união dos braquetes ao esmalte, MPa (n=12)

Material	Tempo de fotoativação		
	5 s	10 s	20 s
Controle	2.5 (0.9) C,b	3.5 (0.7) B,c	4.5 (1.4) A,b
1 mol%	4.0 (1.0) B,a	6.1 (1.5) A,a	5.8 (1.4) A,a
2 mol%	3.7 (0.9) B,a	5.2 (1.4) A,b	5.1 (1.1) A,ab

Letras maiúsculas distintas indicam diferenças significativas na mesma linha (entre tempos de fotoativação); letras minúsculas na mesma coluna (entre materiais).

Por outro lado, analisando os valores de resistência de união entre os grupos testes, observa-se que a maior concentração de sal (2 mol%) não foi benéfica para a união dos braquetes ao esmalte, inclusive ocasionando menor resistência de união em algumas condições. Este resultado pode ser explicado pelo aumento da tensão de polimerização em função da maior velocidade da reação, conforme previamente observado (GONÇALVES et al, 2013). A maior disponibilidade de radicais livres associada à situação de confinamento do cimento ortodôntico na interface braquete/esmalte interfere negativamente na união. Em outra análise, os escores do IAR apresentaram prevalência de escores 2 e 3, mostrando que o cimento experimental apresentou boa capacidade de interação ao esmalte dentário independente das variações de tempo de polimerização e concentração de sal de iodônio (Figura 1).

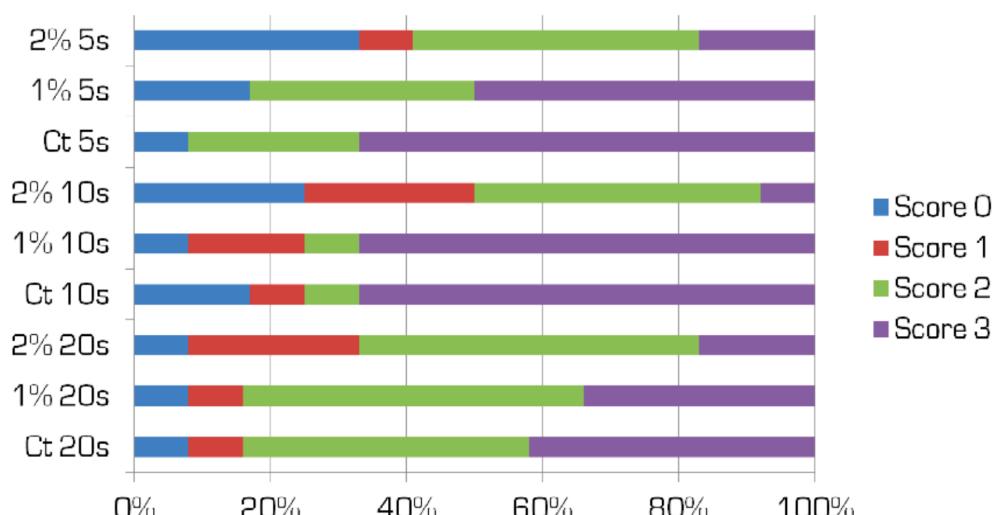


Figura 1. Frequência dos escores do Índice de Adesivo Remanescente. Mostrando uma prevalência de escores 2 e 3.

Analisando a evolução da resistência de união dos diferentes cimentos nos tempos de fotoativação testados, observa-se que os materiais experimentais já apresentaram o máximo de resistência de união (e consequentemente grau de conversão de C=C) no tempo de 10 s, enquanto o cimento controle mostrou progressão da resistência de união em todos os tempos de fotoativação avaliados. Dessa forma, os resultados deste estudo estão alinhados com os resultados relatados na literatura, apontando o uso do sal de iodônio como coadjuvante no sistema de iniciação como uma real possibilidade de reduzir o tempo de polimerização de cimentos utilizados na colagem de braquetes ortodônticos. Entretanto, novos estudos ainda são necessários para esclarecer fatores como concentração ideal e resistência em situações semelhantes ao meio bucal.

### **5.5 Conclusão**

A incorporação de sal de iodônio ao compósito adesivo apresentou resultados promissores para redução do tempo de polimerização requerido para colagem de braquetes ortodônticos.

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## **6 Conclusões**

Respeitadas as limitações do presente estudo, pode-se concluir que:

- Fatores como exposição a agentes corantes, escovação e tipo de dentífrico utilizado podem interferir na estabilidade estética de ligaduras ortodônticas;
- Deve-se evitar o deslocamento do braquete ortodôntico durante o processo de colagem, ao menos após a remoção do excesso de cimento ortodôntico;
- A utilização de duas fontes de luz simultâneas, uma em cada face proximal do braquete durante a fotoativação, parece não representar risco à resistência de união do braquete ortodôntico ao esmalte dental, e pode ser uma alternativa para reduzir em até 50% do tempo clínico despendido com fotoativação;
- A incorporação de sal de iodônio ao compósito adesivo ortodôntico é promissora no desenvolvimento de materiais que permitam redução do tempo de polimerização requerido para colagem de braquetes ortodônticos;
- A associação do protocolo de colagem utilizando duas fontes de luz simultâneas com cimentos ortodônticos que apresentem maior reatividade de polimerização pode representar redução de até 75% no tempo de fotoativação na colagem de braquetes ortodônticos, sendo o conjunto de técnica adequado para aplicação de Ortodontia em Odontopediatria onde o tempo clínico com colaboração do paciente é reduzido;
- Novos estudos laboratoriais e clínicos devem ser conduzidos com o objetivo de otimizar o tempo clínico em Ortodontia, melhorar o desempenho dos materiais odontológicos e oferecer estética mais duradoura aos aparelhos e tratamentos ortodônticos.

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