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Efeito da adição de monômeros funcionalizados em um cimento ortodôntico experimental na prevenção de danos ao tecido dental

Raíssa Coi de Araújo

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Resumo

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O advento da adesão na Odontologia permitiu o uso de acessórios ortodônticos cimentados diretamente sobre a superfície dental. Com estes procedimentos adesivos, surge um importante questionamento acerca do método mais indicado para a remoção dos bráquetes e desgaste do agente de cimentação remanescente sobre o esmalte dental. Várias são as técnicas propostas no que tange aos instrumentos de remoção, mas a literatura aponta que, independente da técnica, sempre há dano à estrutura dental. O uso de acessórios ortodônticos, associados ao excesso de agente de cimentação ao redor dos bráquetes, possibilita um maior acúmulo de biofilme e, consequentemente, desenvolvimento de cárie ao redor dos acessórios. Assim, este estudo tem o objetivo adicionar metacrilatos metálicos coloridos - cobre e prata - a cimento ortodôntico experimental e avaliá-los quanto a influência da cor dos novos cimentos na identificação dos excessos, previamente à fotoativação do produto, e no desgaste do remanescente após a descimentação dos acessórios ao final do tratamento. Outro objetivo foi avaliar o efeito antimicrobiano produzido pela adição dos metacrilatos contra o *Streptococcus mutans,* microorganismo fortemente associado à carie dental. Os grupos testados foram: Cimento ortodôntico experimental sem metacrilato metálico (C), Cimento ortodôntico experimental com Metacrilato de Prata (C_{MAg}), Cimento ortodôntico experimental com Metacrilato de Cobre (C_{MCu}) e referências comerciais - cimento ortodôntico convencional (Transbond XT, 3M Unitek, Monrovia, CA, EUA) e resina composta de coloração distinta da estrutura dental (Resina Composta Filtek Z250 XT D3, 3M ESPE, St Paul, MN, USA). Os materiais foram caracterizados quanto ao grau de conversão, propriedades colorimétricas, propriedades físicas, químicas e mecânicas, viabilidade celular e efeito antibacteriano (através de Teste de Difusão e Ágar e Teste de Contato Direto Modificado). O tempo utilizado na cimentação e remoção dos bráquetes foi avaliado e a superfície do esmalte foi caracterizada em microscopia eletrônica de varredura (MEV), bem como a perda estrutural de esmalte, resultante da remoção do agente de cimentação residual, foi avaliada através da Perfilometria 3D (Rugosidade superficial - Sa e volume de esmalte perdido - V). Os procedimentos experimentais de cimentação, remoção e desgaste do remanescente de cimento foram realizados por operador experiente. Para todos os testes foi considerado o valor $p<0,05$ como estatisticamente significante. Através dos testes realizados, foi possível identificar que os C_{MCu} e C_{MAg} podem ser potencialmente antimicrobianos, uma vez que apresentaram halo de inibição maior que os dos demais grupos. C_{MCu} inibiu o crescimento microbiano no período de 24h, havendo a necessidade de testar este efeito em um período prolongado. Os cimentos coloridos facilitaram a identificação e remoção do material que permaneceu sobre a estrutura dental, reduzindo o tempo de trabalho.

Palavras-chave: Ortodontia, materiais dentários, agentes de cimentação, efeito antimicrobiano.

Abstract

ARAÚJO, Raíssa Coi de. **Effect of functionalized monomers addition in an experimental orthodontic cement to prevent damage to dental tissue.** 2016. 83p. Dissertation (Master degree in Dentistry). Graduate Program in Dentistry. Federal University of Pelotas, Pelotas, 2016.

The advent of adhesion in dentistry allowed the use of orthodontic appliances directly cemented on the tooth surface. With these adhesive procedures, an important question about the most appropriate method for debond brackets and for wearing remnant bonding agents on dental enamel arises. There are several methods and instruments used for that purposes, but the literature indicates that, regardless of the technique, there is always damage to tooth structure. The use of orthodontic appliances associated with the excess bonding agent around the brackets allows a biofilm accumulation and thus caries development around accessories. This study aims to add colored metal methacrylates - copper and silver - on experimental orthodontic cement, and evaluate the influence of the color of new cements when identifying excesses prior to photoactivation of the product, and the remaining wear after debonding of accessories at the end of the treatment. Another objective was to evaluate the antimicrobial effect produced by the addition of methacrylates against *Streptococcus mutans*, a microorganism that is strongly associated to dental caries. The groups tested were experimental orthodontic cement without metal methacrylate (C), experimental orthodontic cement with Silver Methacrylate (C_{MAg}), experimental orthodontic cement with Copper Methacrylate (C_{MCu}) and commercial references - conventional orthodontic cement (TB - Transbond XT, 3M Unitek, Monrovia, CA, USA) and resin composed of distinct coloration of the dental structure (R_{Z250} - Filtek Z250 XT D3, 3M ESPE, St Paul, MN, USA). The materials were characterized as the degree of conversion, colorimetric properties, physical, chemical and mechanical properties, cell viability and antibacterial effect (through Agar Diffusion Method and Modified Direct Contact Test). The time used in cementing brackets and removal of the remnant bonding agent was evaluated, then the enamel surface was characterized by scanning electron microscopy (SEM), and the structural enamel loss resulting from the removal of the residual bonding agent was evaluated by means of 3D profilometry (surface roughness - Sa and volume of enamel loss - V). Experimental procedures of cementing, removal and wear of the remaining cement were performed by an experienced operator. For all tests it was considered $p < 0.05$ as statistically significant. Through the tests, it was possible to identify that C_{MCu} and C_{MAg} can potentially be antimicrobial, since they showed inhibition zone larger than the other groups. C_{MCu} inhibited microbial growth in the 24 hour period, needing to test this effect in a longer period. Colored cement facilitated the identification and removal of the material that remained on the tooth structure, reducing the working time.

Key-words: Orthodontics, dental materials, bonding agents, antimicrobial effect.

Sumário

1 Introdução

O advento dos sistemas adesivos permitiu significativos avanços nos procedimentos clínicos realizados em todas as especialidades da Odontologia. Da mesma forma, o uso de materiais adesivos permitiu uma grande evolução da Ortodontia: a possibilidade de cimentação de bráquetes ortodônticos diretamente sobre esmalte dental.

A partir destas práticas, surgiu a discussão entre os Ortodontistas, acerca das técnicas de aplicação (AL SHAMSI et al., 2007; FJELD; ØGAARD, 2006; HOSEIN; SHERRIFF; IRELAND, 2004), manutenção e remoção dos acessórios ortodônticos (ZARRINNIA; EID; KEHOE, 1995) e, principalmente, acerca dos protocolos de desgaste para remoção do material remanecente na estrutura do esmalte dental (BONCUK; ÇEHRELI; POLAT-ÖZSOY, 2014; CAMPBELL, 1995; CHAN; HIRASUNA; FRIED, 2014; EMINKAHYAGIL et al., 2006; HOSEIN; SHERRIFF; IRELAND, 2004; KRELL; COUREY; BISHARA, 1993; MACIESKI et al., 2011; RETIEF; DENYS, 1979; ZARRINNIA; EID; KEHOE, 1995).

Para discutir estas questões, alguns estudos vêm sendo realizados, com o objetivo de qualificar a superfície dental antes e após a remoção dos dispositivos ortodônticos (ALBUQUERQUE et al., 2010; ALESSANDRI BONETTI et al., 2011; BROSH et al., 2005; FARIA-JÚNIOR et al., 2015; GWINNETT; GORELICK, 1977; TIJANA et al., 2012). Outros, ainda, têm por objetivo avaliar ou quantificar o dano ao esmalte, que ocorre pelo uso dos mais diversos acessórios e instrumentos, tentando restabelecer a superfície danificada (arranhada, rugosa ou irregular) e remover os resíduos de resina (FERREIRA et al., 2013; FITZPATRICK; WAY, 1977; JANISZEWSKA-OLSZOWSKA et al., 2014b; LEÃO FILHO et al., 2015; LEE; LIM, 2008; PATCAS et al., 2015). As cicatrizes resultantes da remoção de resina residual são inevitáveis, e um polimento eficaz, eficiente e confortável ao paciente se faz necessário (CAMPBELL, 1995), apesar não removerem sulcos mais pronunciados (BURAPAVONG et al., 1978). O polimento final com pasta de pedra pomes e água vem sendo considerado uma boa alternativa para amenizar os danos produzidos no esmalte (DAVID et al., 2002; FERREIRA et al., 2013). Pastas de polimento também são utilizadas com esta finalidade, apresentando resultados positivos para melhora na superfície de esmalte. (FERREIRA et al., 2013)

Vários são os métodos para remoção do cimento remanescente após a descolagem dos acessórios ortodônticos: pontas, brocas e acessórios de acabamento e polimento. Através da literatura, é possível identificar que as pontas carbide de tungstênio em baixa rotação são aquelas que causam os menores danos à estrutura dental (JANISZEWSKA-OLSZOWSKA et al., 2014). Entretanto, independente do material utilizado, sempre haverá dano ao esmalte (BROSH et al., 2005; FITZPATRICK; WAY, 1977; GWINNETT; GORELICK, 1977; JANISZEWSKA-OLSZOWSKA et al., 2014; PONT et al., 2010; STRATMANN et al., 1996). Apesar da falta de padronização e estudos controversos, as diversas técnicas apresentadas podem ser utilizadas e irão depender, principalmente, da habilidade do operador (AL SHAMSI et al., 2007; JANISZEWSKA-OLSZOWSKA et al., 2014).

Além da superfície sadia que pode ser perdida pelo uso de pontas e intrumentos de remoção dos cimentos remanescentes, deve-se atentar para o aumento da rugosidade destas superfícies, que pode favorecer o acúmulo de biofilme (SUNDARARAJ et al., 2015). Um estudo revela que rugosidade superficial maior que 0,2µm é capaz de provocar maior retenção de biofilme (BOLLEN; LAMBRECHTS; QUIRYNEN, 1997).

Durante o tratamento ortodôntico há a possibilidade de aumento na retenção de biofilme, principalmente pela presença de fatores retentivos de placa – bráquetes, bandas ortodônticas, tubos e arcos (SUNDARARAJ et al., 2015). A manutenção dos dispositivos ortodônticos em boca causa uma alteração transitória da microbiota, que promove um aumento no número de *Streptococcus mutans* (ROSENBLOOM; TINANOFF, 1991) e, consequentemente, aumento do risco de cárie (SUNDARARAJ et al., 2015). Neste contexto, a utilização de agentes de cimentação com efeito antimicrobiano mostra-se como uma alternativa interessante, reduzindo ou inviabilizando o acúmulo de biofilme na interface bráquete-esmalte.

Alguns antimicrobianos vêm sendo adicionados a cimentos ortodônticos e testados quanto a sua ação antimicrobiana e sua influência nas propriedades físicas e mecânicas desses materiais. Entre os antimicrobianos, podemos citar Cloreto de Benzalcônio (OTHMAN et al., 2002; SEHGAL et al., 2007), Cloreto de Cetilpiridínio (AL-MUSALLAM et a., 2006), Partículas de Prata (AHN et al., 2009), Clorexidina (CACCIAFESTA et al., 2006; CALABRICH et al., 2010; SEHGAL et al., 2007) e Triclosan (SEHGAL et al., 2007).

Para Sehgal et al. (2007), a adição de Cloreto de Benzalcônio foi capaz de inibir o crescimento bacteriano e não interferiu na resistência de união do adesivo. A Clorexidina adicionada ao material teve uma pequena inibição, e a liberação de Triclosan foi considerada abaixo da Concentração Mínima Inibitória.

Produtos derivados da prata podem ter efeitos antibacterianos eficazes contra uma variada gama de microorganismos (MONTEIRO et al., 2009). As partículas de prata, assim como as de cobre (RAFFI et al., 2010), têm ação por contato, alterando a função da membrana e permitindo a entrada das partículas, que interagem com o DNA e provocam a morte do microorganismo (MORONES et al., 2005). Segundo a literatura, as partículas de Prata podem ajudar a prevenir a desmineralização do esmalte ao redor dos bráquetes, sem o comprometimento das propriedades físicas (AHN et al., 2009; LI et al., 2013).

Alguns monômeros antimicrobianos vêm sendo uilizados em diversos materiais odontológicos, como os sistemas adesivos, visando efeito bioprotetor. Esses materiais, ao serem polimerizados, formam, juntamente com os monômeros da matriz orgânica, uma rede polimérica, que retém as moléculas antimicrobianas, evitando sua lixiviação e prolongando seu efeito (IMAZATO, 2003). Alguns exemplos destes monômeros são Brometo de 12-metacriloiloxidodecilpiridínio (MDPB), Cloreto de metacriloxietil cetil dimetilamonio (DMAE-CB), Metracilato de dimetilaminodecil (DMADDM) (COCCO et al., 2015).

Em estudo recente, Henn et al. (2011) incluíram Metacrilato de Zinco em Sistemas Adesivos experimentais e obtiveram resultados positivos para ação antimicrobiana contra *Streptococcus mutans*, um microoganismo fortemente relacionado à doença cárie, muito comum em pacientes em tratamento ortodôntico (GORELICK; GEIGER; GWINNETT, 1982). Como desvantagem, porém, houve redução na resistência de união dos sistemas adesivos, à medida em que a concentração do metacrilato de zinco aumentava (HENN et al., 2011).

Outra propriedade interessante para os cimentos ortodônticos e que vem sendo requerida pelos ortodontistas é a possibilidade de identificação do material no momento de sua aplicação sobre o esmalte, visando a remoção completa dos excessos previamente à polimerização do material (EKHLASSI et al., 2011). Esses excessos são responsáveis pela retenção de placa ao redor dos acessórios ortodônticos, que poderá resultar em lesões de mancha branca, caso permaneçam ali por determinado período (SUKONTAPATIPARK et al., 2001). A literatura relata que 4 semanas são suficientes para que as primeiras lesões de mancha branca apareçam sobre o esmalte dental (ØGAARD; RØLLA; ARENDS, 1988).

A semelhança de cor entre a resina/cimento e o substrato, foi relatada como fator dificultador de sua remoção (SANDISON, 1981). Tendo a vista essa necessidade, o uso de materiais de fácil remoção e de fácil evidenciação faz-se necessário (ZACHRISSON; ÅRTHUN, 1979). O emprego de cimentos coloridos,, ou seja, com coloração destacada em relação à cor do esmalte dental, apresenta-se como alternativa na solução desse desafio clínico.

Com o objetivo de otimizar a remoção do remanescente do agente de cimentação, após o tratamento ortodôntico, e preservar o tecido dental sadio, estudos têm proposto a utilização de “evidenciadores de resina”, pigmentantes naturais ou artificiais que, aplicados na superfície do esmalte, facilitam sua percepção na superfície do dente, permitindo maior precisão na sua remoção (ABDALLAH et AL., 2014). Essa técnica é interessante e pode proporcionar, na prática, uma adequada identificação entre o tecido dental e cimento residual. Entretanto, pigmenta apenas superficialmente o material e, assim que removida a camada mais superficial, necessita de reaplicação. Levando em consideração que o procedimento deve ser repetido algumas vezes, para cada elemento dental que teve um dispositivo cimentado, essa técnica pode resultar em aumento significativo do tempo clínico empregado no procedimento.

Neste contexto, o emprego de um agente de cimentação antimicrobiano e de coloração contrastante ou diferente da coloração da estrutura dental parece uma interessante alternativa para os agentes de cimentação comumente empregados. A coloração diferenciada pode ser obtida com o desenvolvimento de novos materiais coloridos, ou ainda pelo estabelecimento de protocolo de seleção de cor do material na escala de cores contrastante àquela observada na estrutura dental. Dessa forma, o propósito deste estudo é avaliar a influência do efeito antibacteriano e da cor do cimento resinoso empregado na fixação de dispositivos ortodônticos em seu desempenho e aplicação, caracterizando o tecido dental remanescente e sua preservação/manutenção, após a remoção do agente de cimentação.

A hipótese a ser testada nesse estudo é que a utilização de um agente de cimentação antibacteriano e de coloração contrastante à cor da estrutura dentária facilitará a aplicação e remoção do material e permitirá maior preservação do tecido dental.

2 Capítulo 1

Antibacterial potential of novel preventive orthodontic cements

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Highlights

Contrasting cements facilitate excess removal of cement around brackets, preventing biofilm accumulation and enamel damage;

Cements can prevent enamel damage.

Abstract

Objective: The objective of this study was to add metal methacrylates, copper and silver, and evaluate the antimicrobial effect of these products on an experimental orthodontic cements (C_{MAg} and C_{MCu}), comparing them with the experimental orthodontic cement with no addition of methacrylate (C) and as a commercial reference Transbond XT (3M Unitek, Monrovia, CA, USA) and the composite resin Filtek Z250 XT color D3 (3M ESPE, St. Paul, MN, USA). Degree of conversion, Rate and Kinetics of polymerization, Agar diffusion method, Modified direct contact test, Cell viability, Shear bond strenght, Remnant adhesive index and Water Sorption and Solubility were evaluated after an initial screening concentration, based on data of the Degree of conversion and Agar diffusion method. Concentrations that were in the screening were: Experimental Cement + Silver Methacrylate (C_{MAg}) 0.5 mol%, 1 mol%, 2 mol% and 4 mol%; Experimental Cement + Copper methacrylate (C_{MCu}) 0.5 mol%, 1 mol%, 2 mol%, 4 mol%, 8 mol% and 16 mol%. For all the tests, it was considered $p<0.05$ as statistically significant. After the screening, the chosen concentrations were $C_{MCu16\%}$ and $C_{MAg1\%}$. All groups had similar Degree on conversion. In Inhibition zone, experimental groups with methacrylate showed C_{MCu}=14mm^{a}, C_{MAg}=8mm^{ab} and the other groups did not show effect. In modified direct contact test, C_{MCu} did not show bacterial growth. In cell viability test, all groups showed similar results. In the shear bond strength, commercial references showed the greater values (TB=24.5 ±4.7^{A}, R_{Z250}=22.6 ±8.2^{A}, C=18.2 ±6.5^{B}, C_{MCu}=12.1 ±3.9^{C}, C_{MAg}=16.5 ±5.2^{B}), but all groups were into a limit considered reasonable. For Water sorpion, the values are between TB=0.54 ±0.2^{A} and C_{MCu}=4.26 ±0.7^{D}. For solubility, no difference was observed between the groups.

Conclusion: The experimental orthodontic cements showed suitable physical and microbiological outcome, making possible to use it as orthodontic cement. C_{MCu} also showed the best antimicrobial effects, considering both the tests.

Keywords: Orthodontics, dental materials, luting agents, orthodontic

1 Introduction

Accessories bonded directly to the dental structure are widely used in orthodontics due to both popularization of the treatments and increase of demands to an aesthetic smile. The brackets and other orthodontic assessories require proper cleaning procedures because it creates a favorable environment to biofilm accumulation [1,2], which may cause enamel desmineralization, or even exacerbate pre-existing injuries [3], consequently increasing the risk of caries around dispositives [4]. Previous studies have shown the prevalence of these type of lesions up to 68.4% [5]. Besides, desmineralization around brackets occurs fastly, and it takes approximately 1 month for the white spots become evident, even with the use of fluoride toothpaste [1,6]. Since white spot lesions around brackets are difficult to reverse [4], it can lead to aesthetic concerns.

In addition to the cleaning difficulties imposed by the presence of the orthodontic accessories, the increased enamel surface roughness caused by the excess bonding agent has been linked with biofilm accumulation [3]. To facilitate the removal of excess cement, colored bonding materials have been developed in order to facilitate their identification [7]. However, the color of these cements becomes similar to teeth after curing. Therefore, proper identification of the orthodontic cement after bracket debonding is still chalenging [8].

With the purpose to reduce enamel damage, bonding agents with antimicrobial agents to prevent enamel demineralization have been tested, including benzalconium chloride [9,10], cetilpiridinium chloride [11], silver particles [12], chlorhexidine [10,13,14], and triclosan [10]. Materials with silver compounds have shown effective antimicrobial effect against several microorganisms [15]. Silver particles can help preventing enamel demineralization around brackets without being detrimental to the physical properties of orthodontic adhesives [12]. Copper particles have also shown promising results for antibacterial effects [16]. The antibacterial effect of copper and silver ions is based on the adhesion and destruction of the bacterial cell walls, leading to cytoplasm degradation and cell death [17].

Antimicrobial methacrylate monomers have been incorporated into dental materials [18], like MDPB (12-methacryloyloxydodecylpyridinium bromide) [19] and silver methacrylate [20]. These monomers act by contact and are retained in the polymer network after polymerization, prolonging the antimicrobial effect [21]. Zinc methacrylate is another example of metal methacrylates that can be added to dental adhesives to provide antimicrobial properties [22]. Although silver particles are widely tested in dental materials, silver methacrylate is seldom studied for the same purpose, most likely because this monomer may impart a gray shade to the materials [12,20]. In orthodontics, the color alteration caused by metal methacrylates could actually be a positive effect, since it could facilitate removal of excess cement after bracket debonding.

The aim of the present study was to evaluate the effect of the incorporation of metal methacrylates into experimental orthodontic cements and the associated antimicrobial activity, cell viability, and physicochemical properties of the modified materials. The hypothesis evaluated was that the incorporation of these metal methacrylates would impart antibacterial effect to the cements without affecting the biological and physicochemical properties.

2 Materials and methods

2.1 Experimental Design

This *in vitro* study involved a completely randomized design considering the effect of different types and concentrations of metal methacrylates on the antimicrobial activity and physicochemical properties of experimental resin-based orthodontic cements (Fig. 1). A screening of concentration of the metal methacrylates was carried out in pilot studies, testing the following groups: experimental orthodontic cement + silver methacrylate (C_{MAg}) added at 0.5 mol%, 1 mol%, 2 mol%, and 4 mol%; experimental orthodontic cement + copper methacrylate (C_{MCu}) added at 0.5 mol%, 1 mol%, 2 mol%, 4 mol%, 8 mol%, and 16 mol%; experimental orthodontic cement without metal methacrylates as inner control. In this pilot study, degree of C=C conversion and agar diffusion assay analyses were carried out for choosing the best concentration of metal methacrylates to be used in the further analyses. The concentration selected were 1% C_{MAg} and 16% C_{MCu}. With the choosed concentrations, the commercial references (Transbond XT and Composite Resin Z250) were also evaluated in the final experiments. The response variables assessed were polymerization kinetics and rate, direct contact test, cytotoxicity, water sorption and solubility, shear bond strength to enamel and failure modes scored by the adhesive remnant index (ARI).

2.2 Reagents

Bisphenol-A glycidyl dimethacrylate (Bis-GMA), triethyleneglycol dimethacrylate (TEGDMA) and camphoroquinone (CQ) were obtained from Esstech Inc. (Essington, PA, USA). Silica nanoparticles (Aerosil 380, 7nm) were obtained from Evonik Industries AG Inorganic Materials (Hanau-Wolfgang, Germany). Ethyl 4-dimethylamine benzoate (EDAB) and diphenyliodonium hexafluorophosphate (DPIHFP) were obtained from Sigma-Aldrich Chemical Co. (Milwaukee, WI, USA). Copper methacrylate and silver methacrylate were obtained from abcr GmbH & Co. KG. (Karlsruhe, Germany).

2.3 Formulation of experimental materials and commercial references tested

The experimental orthodontic cements were prepared using a 3:1 Bis-GMA:TEGDMA ratio. Silanized silica nanoparticles (20 wt%) were added in resin matrix. The photoinitiator system was composed by 0.5 mol% CQ, 0.8 mol% EDAB, and 0.4 mol% DPIHFP. The metalic compounds were added at molar fractions of 0.5, 1, 2 and 4 % of silver methacrylate, and 0.5, 1, 2, 4, 8, and 16 % of copper methacrylate (please see section 2.1 for details). The chemical structures of the evaluated metal methacrylates are shown in Table 1. Transbond XT light cure adhesive (3M Unitek, Monrovia, CA, USA) (TB) with Transbond XT Primer (3M Unitek) was used as commercial reference since it is considered a gold-standard orthodontic cement. Other commercial reference was the composite resin Filtek Z250 (3M ESPE, St Paul, MN, USA) (R_{Z250}), shade D3, as “colored” commercial reference plus the adhesive system Adper Scotchbond Multi-purpouse (3M ESPE).

2.4 Degree of C=C Conversion (DC) and Polymerization Kinetics (PK)

The degree of conversion was evaluated using Fourier-transform mid-infrared spectroscopy (RT-FTIR; Prestige21 Spectrometer, Shimadzu, Tokyo, Japan) with an attenuated total reflectance device composed of a horizontal multiple-reflection diamond crystal with 45° mirror angle (Pike Technologies; Madison, WI, USA). A preliminary reading of unpolymerized material was taken under the following conditions: 12 scans co-addition, 8 cm^{-1} resolution, Happ–Genzel apodization, and 2.8 mm/s mirror speed. After the first reading, the bonding agents were immediately photoactivated. A support was coupled in order to the spectrometer to hold the curing unit and standardize a 5 mm distance between the fiber tip and the material. The DC was evaluated in the absorbance mode after photoactivation with a LED Curing Unit (Radii Cal; SDI, Bayswater, Victoria Australia) for 60 s at 800 mW/cm^2 irradiance. DC calculation considered the intensity of C=C stretching vibration (peak height) at 1635 cm^{-1} and the C=C aromatic stretching band at 1609 cm^{-1} as internal standard. Three specimens per material were tested under controlled temperature (25±2 °C) and humidity (45±5%) conditions [23–28]. DC was made, initially, in screening of concentration, to select the best concentrations on experimental groups. After this, DC was made to analyse selected concentrations and commercial references. The DC data were analyzed using the software SigmaPlot 12 (Systat Inc, San Jose, CA, USA). Statistical analysis for DC was performed using One Way Analysis of Variance and Tukey test. The level of significance was set at $p<0.05$.

The polymerization kinetics data were plotted and curve fitting performed by using Hill's three parameter non-linear regression. Using these data, the polymerization rate (PR) (R_P (s^{-1})) was calculated as the DC at time t subtracted of DC at time $t - 1$. The coefficient of determination was greater than 0.95 for all curves.

2.5 Antimicrobial tests

2.5.1 Agar Diffusion Method (ADM)

Streptococcus mutans ATCC 25175 were used to evaluate the antimicrobial effect of the bonding agents. The isolated microorganisms were reactivated for 24 hours in Brain Heart Infusion (BHI) agar culture medium. The inoculum was adjusted through an equivalent spectrophotometer (0.1 mL of 108 cells per mL). The solution was seeded on the surface of BHI medium. Filter paper discs (6 mm) were used to dispense the cement on the agar and after were photoactivated for 40 seconds (n = 3). Additionaly, it was evaluated the pure metal methacrylates (powder); and 2% Chlorhexidine (Riohex 2%, Rioquímica®, São José do Rio Preto, SP, Brazil) was used as control. The plates were then incubated at 37°C in the anaerobic environment (Anaerobac, PROBAC DO BRASIL Produtos Bacteriológicos Ltda., São Paulo, SP, Brazil) for 18 hours. After this period, the inhibition zones (IZ) of bacterial growth around the samples were measured with a caliper in millimeters. The tests were made in triplicate. ADM was made, initially, in screening of concentration, to select the best concentrations on experimental groups. after this, ADM was made to analyse selected concentrations and commercial references. Statistical analysis of IZ on screening was performed using Kruskal-Wallis Test followed by Dunn's method to experimental orthodontic cement with silver methacrylate and followed by Tukey test for experimental orthodontic cement with copper methacrylate. For selected concentrations ($C_{MCu16\%}$ and $C_{MAg1\%}$), C, TB and R_{Z250}, IZ was analysed with Kruskal-Wallis test and Tukey test. ($p<0.05$) Correlation between monomer concentration and antimicrobial effect was assessed by Pearson's correlation.

2.5.2 Modified Direct Contact Test (MDCT)

The discs (6 x 1mm) were made with the bonding agents and evaluated in 1h and 24h (n = 3 / group / time). After, they were sterilized by ultraviolet light, during 30 minutes on each side of the sample [29]. The samples were dispensed in a 96-well microplate, 1 sample per well, with a suspension of bacteria (*Streptococcus mutans* ATCC 25175) 20µL on the disks, so as to involve them. It remained so for 1h and 24h at 37 °C. 180µL of BHI were added to the wells containing materials and 3 more wells that served as the control of microbial growth (microbial suspension with culture medium without the material). Then, the plate was taken to the shaker for 10 min. Were removed 100 µL of the bacterial suspension and added to saline for carrying out serial dilutions, thus obtaining 10x dilutions (1), 100x (2), 1000x (3) and 10000x (4). Samples were plated on BHI agar, dividing them into 5 parts, they were placed in 3 aliquots (20 uL) of each dilution. The plates are incubated at 37 ° C in anaerobic atmosphere for 24h. After the incubation period, it was made the counting of Colony Forming Units (UFC). The test was made in triplicate. Statistical analysis between bonding agents was performed using Two Way Analysis of Variance and Student-Newman-Keuls test. ($p<0.05$)

2.6 Cell Viability Assay (CV)

Cell viability was perfomed according to ISO 10993 (2009). Mouse fibroblasts L929 ($20x10^3$ well^{-1}) were maintained in Dulbeccos´s Modified Eagle Medium (DMEM) supplemented with 10 % fetal bovine serum (FBS) 2 % L -glutamine, penicillin (100 U.ml^{-1}) and streptomycin (100 mg.ml^{-1}) using 96-well plates for 24h incubated at 37ºC in a humidified atmosphere of 5 % CO_2 in air until sub confluence was achieved. Specimens of each bonding agent were made by a silicon mold (6 mm diameter and 1 mm depth), and photo-activated by LED Curing Unit (n = 6). These specimens were placed in 24-well plates with 1 mL of DMEM (pH 7.2) at 37ºC. After 24h, 200 µL of eluate of each group were transferred to the 96-well plates previously prepared and incubated for 24h and 48h (n=6). WST-1 was used to assess cell metabolic function by mitochondrial dehydrogenase activity. The absorbance at 540nm was measured via a microplate reader (SpectraMax M5; Molecular Devices, Sunnyvale, CA, USA). Each assay was repeated at least twice. Statistical analysis was performed using Kruskal-Wallis and Tukey test. ($p<0.05$)

2.7 Water Sorption (WS) and Solubility (S)

Ten discs (6 × 1 mm) were made for each material, photoactivated for 40 s on each side and stored in desiccators containing freshly dried silica gel and calcium chloride. After 22h at 37°C and 2h at 23°C the specimens were weighed on a precision scale and this cycle was repeated until a constant mass was obtained (m1). The specimens were immersed in distilled water for 7 days, at 37ºC, then removed, blotted dry and weighed to obtain m2. The specimens were removed from the water, placed again in a desiccator, and the initial cycle was repeated until obtaining a constant mass (m3). Water sorption (WS) and solubility (S) were calculated using the following formulas:

$$\text{Water Sorption Weight\%} = \frac{m2-m3}{m3} \times 100$$

$$\text{Solubility Weight\%} = \frac{m1-m3}{m3} \times 100$$

For WS, the statistical analysis was performed using One Way Analysis of Variance and Tukey test with data transformed to square root and for S, One Way Analysis of Variance. ($p<0.05$)

2.8 Shear bond strength (SBS) to enamel

Metallic brackets (Edgewise Standard; Morelli, Sorocaba, SP, Brazil) were bonded to the buccal faces of bovine incisors (n=10/group), embedded with self-curing acrylic resin in PVC tube. The bond procedure was performed using following the next protocol: prophylaxy with pumice paste and rubber cup (10s), enamel etching with 37% phosphoric acid (15s), washing and drying, application of the Transbond XT Primer (for C, C_{MCu}, C_{MAg}, TB groups) or Adper Scotchbond Multipurpose Adhesive (for R_{Z250} group) with 20s application, 10s drying and 20s photo-activation. According to the group, each material was added on the bracket base, placed over the buccal surface of a tooth and photo-activated for 20s each side. After bonding, the specimens were stored in distilled water at 37ºC for 24h and subsequiently tested in a shear mode. The specimes were positioned at stabilization unit, with the base of the bracket parallel to the strength direction axis applied by the universal testing machine (DL-500, Emic; São José dos Pinhais, Brazil) at a crosshead speed of 0.5 mm/min, using knife-edged chisels. The chisel was supported on the upper side on the tooth-bracket interface and the compressive load was applied until debonding the bracket. The bond strength values are calculated in MPa, considering the area of the bracket. SBS values of the bonding agents were analyzed using One Way Analysis of Variance and Holm-Sidak mehod. Data were transformed to log10 to perform the analysis. ($p<0.05$)

2.9 Adhesive Remnant Index (ARI)

After debond of brackets to the teeth surfaces, the adhesive remnant index was evaluated, as described for Artun and Bergland (1984) [30]. The remnants were classified following the scores:

* Score 0: no material remnant on the surface

* Score 1: less than half material on the surface;

* Score 2: more than half material on the surface;

* Score 3: all bonding agent on the surface, with the impression of the base of the bracket on the material remnant.

A descriptive analysis was performed to describe the results.

3 Results

3.1 Screening of Concentration of the methalic methacrylates

3.1.1 Degree of Conversion

The first trial to define the screening concentration of the metal methacrylates was the degree of conversion. The addition of metal methacrylates in controlled concentrations did not adversely affect the DC of the experimental cements, only for $C_{MAg4\%}$. The results are shown in Fig. 2.

3.1.2 Agar Diffusion Method

The agar diffusion method (ADM) was also used in screening concentration of methacrylates. Fig. 2 shows the inhibition zone of experimental orthodontic cements associated with metal methacrylates in various concentrations of screening. The formulated cements, C_{MAg} 1%, 2% and 4% showed no statistically significant difference between them. For cements with copper, the inhibition zone of $C_{MCu16\%}$ had highest value than the others but with no significant difference from the $C_{MCu8\%}$ and $C_{MCu4\%}$ ($p<0.05$). Besides, these data show that increased incorporation of copper methacrylate in the experimental orthodontic cement were associated with a logarithmic increase in antimicrobial effect ($y=4.9052\ln(x) + 0.4$; $R^2=0.989$, $p<0.05$).

Over the data obtained in degree of conversion and agar diffusion method, we chose to select the concentrations 16% for copper methacrylate and 1% for the silver methacrylate and compare them with control experimental orthodontic cement (C) (with no metal methacrylate) and commercial references. The results presented from now on will refer to the experimental cements with metal methacrylate concentrations chosen in the screening ($C_{MCu16\%}$ now is C_{MCu} and $C_{MAg1\%}$ now is C_{MAg}), experimental cement control (C) and commercial references (R_{Z250} and TB).

3.2 Degree of Conversion (DC), Water Sorption (WS), Solubility (S), Shear Bond Strength (SBS), Polymerization Kinetics (PK) and Polymerization Rate (PR)

Data for degree of conversion, water sorption, solubility and shear bond strength are shown in Table 2. Experimental materials shower higher degree of conversion and water sorption than commercial references ($p<0.05$). Regarding solubility, all materials were statistically similar. For shear bond strength, experimental groups were different statistically of commercial references, however all groups showed higher values than 6-8 MPa, reference values for shear bond strength considered reasonable [31]**.** To analyze the behavior of materials in real time, during the reaction, the polymerization kinetics and the polymerization rate were assessed as showed in the Fig. 3.

3.3 Antimicrobial tests

3.3.1 Agar Diffusion Method

The Table 3 shows the data for the inhibition zone of the pure substances (powder metal methacrylate – M_{Cu} and M_{Ag}), compared to chlorhexidine, a positive control antimicrobial agent. The comparison of chlorhexidine and the metal methacrylates resulted in significant difference between the tested groups. M_{Cu} had the highest inhibition zone, followed by Chlorhexidine and M_{Ag}.

The Fig. 4 represents the inhibition zone of the bonding agents tested. The C_{MCu} showed highest inhibition zone, statistically different than the commercial references.

3.3.2 Modified Direct Contact Test

In the modified direct contact test (MDCT), the selected cement with metal methacrylate (C_{MAg} and C_{MCu}), the experimental orthodontic cement (C) and commercial references (TB and R_{Z250}) were tested and the antimicrobial effect measured in CFU/ml (colony forming unit per mililiter in 10 logarithm). The Fig. 4 represents the direct contact of the bonding agents tested, after 1 hour all the groups were similar and without antibacterial effect. After 24h, C_{MCu} had growth close to zero, however C_{MCu} and TB were similar statistically.

3.4 Cell viability (CV)

Fig. 5 shows the percentage of cell viability assessed after 24h (A) and 48h (B). The untreated group (cell control without eluate resin) was equal to 100%. In 24h, all groups were different to untreated group. The experimental orthodontic cement containing silver methacrylate and copper methacrylate showed 68.6% and 66.2% of cell viability, respectively, and were not statistically different than the experimental material (C) and other commercial materials evaluated ($p<0.05$). In 48h, all groups showed similar cell viability, making possible the stability of the cements over time.

3.5 Adhesive Remnant Index

Data of the adhesive remnant index are presented in Fig. 6. In general the predominant ARI scores were 3 and 2, for C_{MCu} and C_{MAg} there was not observed any ARI 0 score.

4 Discussion

Several researchers have reported that some metals, in their various forms, have antimicrobial effect, including the copper and silver. Due to this, we chose these metals in a new molecular presentation - associated with a methacrylate and forming a metallic monomer - to be incorporated in preventive orthodontic cement in order to improve its antimicrobial effect against *Streptococcus mutans*, a microorganism strongly related to carie [32]. The occurrence of white spot caries lesions, associated with the use of orthodontic appliances is a fact acknowledged by orthodontists and develop extremely quickly and are difficult to reverse without residual damage [4,5]. The combination of factors - brackets, interface cement, and excess material around the accessories - means that there is a highly favorable environment for the development of carious lesions in patients who can not perform the cleaning properly [1,2]. Trying to resolve this adverse effect, alternatives such as adding antimicrobial agents in preventive orthodontic cements could prevent enamel demineralization and promove remineralization of the affected tissue [9–15].

Initially, we carried out the screening of different concentrations of copper and silver methacrylates based on the degree of conversion and inhibition zone to evaluate if the incorporation of these monomers would lead to some antibacterial effect without affecting the degree of conversion. The addition of copper methacrylate from 0.5 to 16 mol% and silver methacrylate from 0.5 to 2 mol% did not affect negatively the degree of conversion of the experimental orthodontic cements, and this probably occurs because the incorporation of monomers in these concentrations not affected the polymerization. Metal methacrylates can copolymerize with other components of organic matrix after curing, different from the incorporation of particles that are not covalently bonded to the polymer network [18,21]. An study showed that similar relationship was obtained with the addition of zinc methacrylate in experimental adhesive system, with an correlation between increasing methacrylate concentration and values for the degree of conversion [22]. On the other hand, the addition of 4% of silver methacrylate promoted a significant reduction in degree of conversion, rejecting our hypothesis. As demonstred by other study, higher concentrations of silver methacrylate change the color of the material [20], harming the passage of light required for full curing that could have affect the polymerization [33].

Some antimicrobial monomers have been added to dental materials with the aim to avoid oral diseases, such as MDPB (12-methacryloyloxydodecylpyridinium bromide) [18]. MDPB added to an adhesive system showed antimicrobial effect on *Streptococcus mutans*, inhibiting metabolic enzyme activity of the microorganisms [34]. In our study, pure metal methacrylates presented antibacterial activity, that was maintained when incorporated in experimental orthodontic cement. The literature suggests that the action of such monomers occurs by contact [21] by three mechanisms: destruction of cell wall, cytoplasmic enzymes denaturation and inhibition of DNA replication [35]. In an ionic form silver and copper could be leached, leading to a transitory effect. After polymerization, metal methacrylates bind covalently to a polymeric network, which maintains the antimicrobial monomers linked to the structure preventing the leaching and extending the effect. [21].

An increase in copper methacrylate concentration improved the antibacterial activity against *Streptococcus mutans* of experimental orthodontic cements, and 16 mol% was the most efficient concentration. It can be explained by the presence of a strong correlation between copper methacrylate concentration and antimicrobial effect. This concentration dependence was also observed in other study with nanoparticles of copper [17]. On the other hand, silver methacrylate showed antibacterial effect only from 1 mol%, constituting the minimum inhibitory concentration once this effect was not potentiated in higher metal methacrylate concentrations. This result was different from other study with metal methacrylate into a bonding agent, in which the highest antimicrobial effect was observed with the incorporation of 30 wt% in the organic matrix [22].

Silver and copper are added in the form of methacrylate and not as ions or nanoparticles, more popularly related to antibacterial effect. The metal methacrylate was tested due to its binding property with other monomers, which minimizes the leaching with the time. This causes the effect to be extended, according to the literature [21], which it is an advantage in an orthodontic cement that will remain in oral cavity during the treatment. Besides, silver is considered relatively inert and may occur the release of ions in the case of interaction of this silver with humidity in the oral environment [36,37], which could result therefore in a higher antimicrobial effect. However, in modified direct contact test the orthodontic cement with silver did not show antibacterial effect after 1 and 24h.

The agar diffusion method is the most employed method in this kind of research despite some limitations. It is a model with low credibility for samples that are difficult to diffuse in the media because there is no relationship between their solubility in water, diffusion power, and antimicrobial study. In some cases, diffusion techniques can be used for antimicrobial screening, but they may not be used as a definitive method because there is no relationship between MIC values and inhibition diameters [38]. Agar diffusion method is a qualitative non-standardized method that is useful only for the detection but not for the comparison of antimicrobial properties of different samples. Comparison of the size of inhibition zones of different materials cannot be used for determination of the relative antimicrobial potencies, since a more diffusible but less active material could give a larger diameter than a non-diffusible but more active one.

A modified direct contact test prevent many of the problems of the agar diffusion method. The test is quantitative and reproducible that allows testing of insoluble materials and can be used in standardized settings. The modified direct contact test may be a more suitable test than the agar diffusion method to evaluate antibacterial properties of definitive cements. Also, this test simulates the oral conditions unlike agar diffusion method. The method also allows for better control of possible confounding factors compared with agar diffusion method [39].

The orthodontic cement with copper methacrylate presented antibacterial effect in both agar diffusion method and modified direct contact test after 24h and had the highest inhibitory effect of microbial growth. Different from other orthodontic cements evaluated, including the commercial references, this potentiated antimicrobial activity could contribute to reduction of demineralization of enamel. Zinc methacrylate added to an experimental adhesive system also gave antimicrobial effect when added in concentrations from 10% wt, agreeing with the findings of this study [22].

About the cell viability, in 24 hours of evaluation, the preventive orthodontic cements with copper and silver showed mild cytotoxicity similar to other bonding agents. At 48 hours, all groups tested showed similar results, with increased cell viability superior to 99%, demonstrating a possible stability of materials in the period tested. In other study, the addition of zinc methacrylate in an adhesive system do not affect negatively cell [22]. Other components, added in orthodontic cements, as benzalkonium chloride and titanium dioxide nanoparticles orthodontic cements also did not result in a cytotoxic effect [40,41].

The experimental orthodontic bonding agents showed significantly lower bond strength values compared to commercial references. C and C_{MAg} were similar, demonstrating that the addition of silver does not interfere with the shear bond strength of the material. C_{MCu} showed values of 11 MPa and even this cement showing a significant lower shear bond strength than the others, however, it is slightly superior to the values considered reasonable to clinical practice suggested in the literature [31]. Shear bond strength to the experimental bonding agents may be related to the amount of filler used. In these cements, we use 20% of inorganic filler particles, while the commercial references TB and R_{Z250} presented, according to the manufacturer, between 70-80% and 60%, respectively. This difference in the amount of filler may have interfered in the shear bond strength without affecting adhesiveness. The maintenance of adhesiveness is proven by more present scores in the evaluation of ARI, demonstrating that there is adherence to the enamel. A previous study demonstrated the addition 1,3,5-triacryloylhexahydro-1,3,5-triazine at concentrations of 15% and 20% wt in an experimental orthodontic cement resulted in antimicrobial effect without affecting the mechanical properties tested [42].

Water Sorption in dental materials means the ability of these materials absorb some solvent, usually water, to the interior, with possible disruption of the intermolecular bonds and weakening the product. The experimental orthodontic cements presented higher water sorption that commercial references and this can be due to the fact that the experimental materials having less amount of inorganic filler [43]. However, the solubility was similar between all materials evaluated it is known that the solubility is related to the degree of conversion of materials [44]. The values of the degree of conversion, although statistically different, were similar between groups. Possibly a similar percentage of subproducts were leached, which may explain the solubility. These effects can predict product stability, but the limitations of these test must be taken into consideration, such as the short-term results and the realization of the same in distilled water, as recommended by ISO 4049. The test could be performed with saliva [45], which has a complex composition possibly reflect different results.

Through tests, subject to the limitations of this study, it became clear that the experimental materials showed satisfactory results, with values close to the results presented by the commercial references. Experimental orthodontic cements showed cell viability similar to the other groups, in addition to presenting appropriate degree of conversion, shear bond strength with values higher than limits considered acceptable. However preventive orthodontic cement with copper 16 mol% showed antimicrobial activity avoiding microbial growth in tested period. With all these results we can realize the great potential of our material with properties similar to the commercial, with the additional antimicrobial effect, our main objective in this study.

This material requires further studies evaluating their long-term and clinical behavior. It is also necessary to evaluate the antimicrobial effect presented in this study is able to last for longer periods of time and is capable of preventing the demineralization around orthodontic appliances.

5 Conclusion

Metallic monomers used in this study had not been previously used in dental materials, so their behavior was unknown. Even with this limitation, the new materials presented adequate results when compared to commercial sealers. This in vitro study showed that copper methacrylate incorporated in novel orthodontic cements presented greater antimicrobial effect, different from commercial references, with adequate physicochemical and biological properties. This material could be used as preventive orthodontic cement being able to reduce the biofilm accumulation and the risk of caries development. The bonding agent with silver did not show antimicrobial effect but showed adequate physicochemical and biological properties. However, additional studies are needed to evaluate the promising results observed in the present study.

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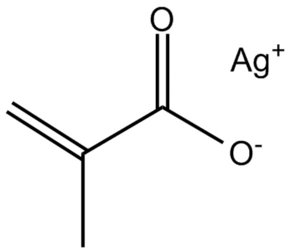
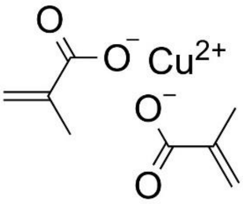
Tables

Table 1 - Chemical structure of the metallic methacrylates tested

Funcionalized monomers	Chemical formula	Structural formula	Molecular weight
Silver methacrylate	$C_4H_6AgO_2$		192.95g
Copper methacrylate	$C_8H_{10}CuO_4$		233.70g

Fonte: abcr GmbH & Co. KG.

Table 2 – Mean ± standard deviation of Degree of C=C conversion (DC), Water Sorption (WS), Solubility (S) and Shear Bond Strength (SBS) to enamel.

Bonding agents	DC (%)	WS (%)	S (%)	SBS (MPa)
TB	42.7 ± 2.2^{B}	0.54 ± 0.2^{D}	0.21 ±0.3	24.5 ± 4.7^{A}
R_{Z250}	37.7 ± 0.8^{C}	1.18 ± 0.3^{C}	0.22 ±0.3	22.6 ± 8.2^{A}
C	49.8 ± 1.2^{A}	3.77 ± 0.5^{AB}	0.50 ±0.4	18.2 ± 6.5^{B}
C_{MCu}	49.4 ± 1.8^{A}	4.26 ± 0.7^{A}	0.25 ±0.6	12.1 ± 3.9^{C}
C_{Mag}	48.3 ± 0.7^{A}	3.37 ± 0.6^{B}	0.16 ±0.5	16.5 ± 5.2^{B}

Different uppercase letters in columns indicate diferences between the bonding agents ($p<0.05$).

Table 3 – Median and interquartile range (q1-q3) of Inhibition Zone of the pure substances of methacrylate monomers and the control Chlorhexidine.

Pure Substance	Median (mm)	q1-q3
Copper methacrylate (M_{Cu})	26[a]	26-27
Silver methacrylate (M_{Ag})	12[c]	12-14
Chlorhexidine	19[b]	18-20

Different lowercase letters mean statistical differences between powder metallic monomer and the chlorhexidine ($p<0.05$).

Figures

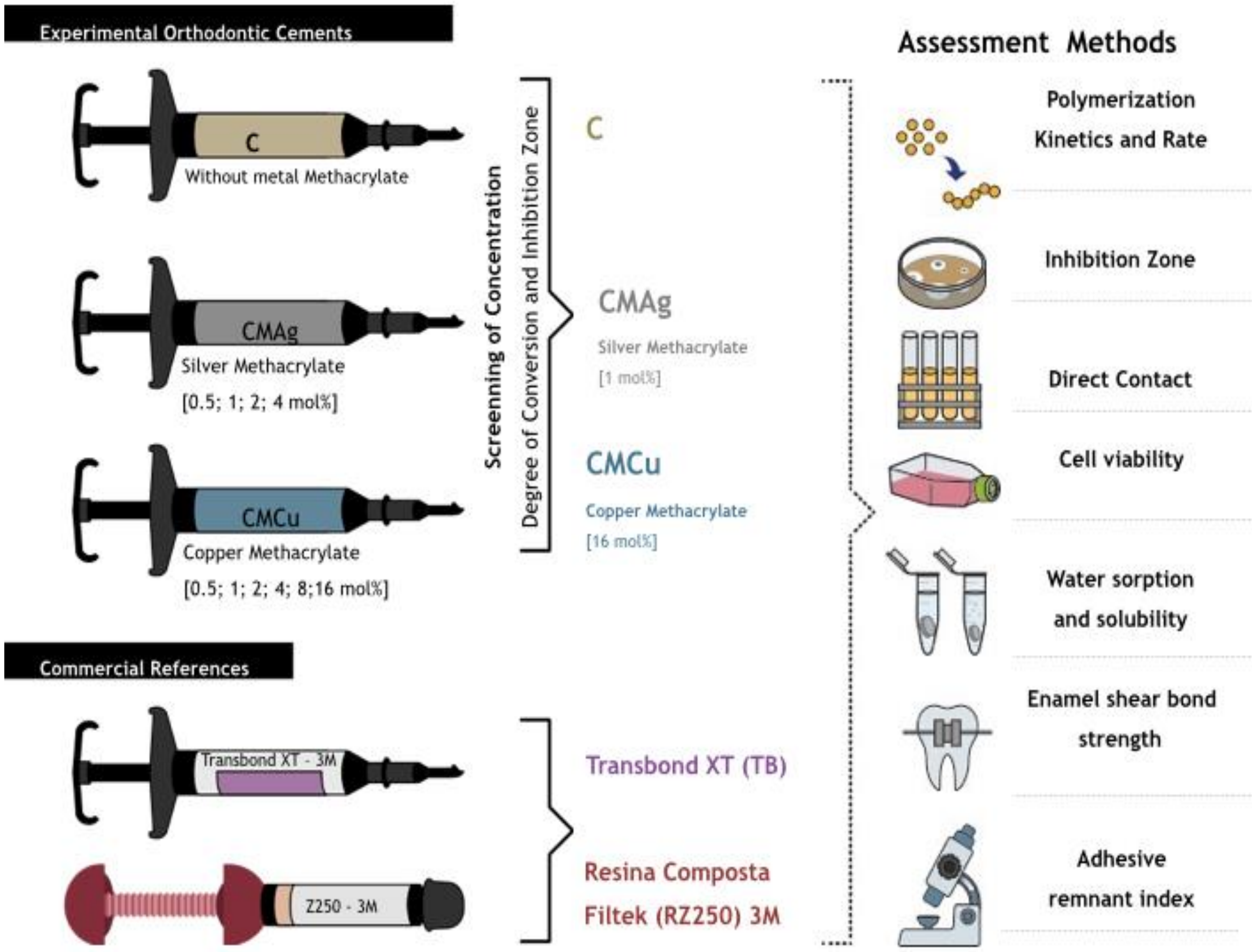


Fig. 1 – Experimental design and response variables tested in the study.

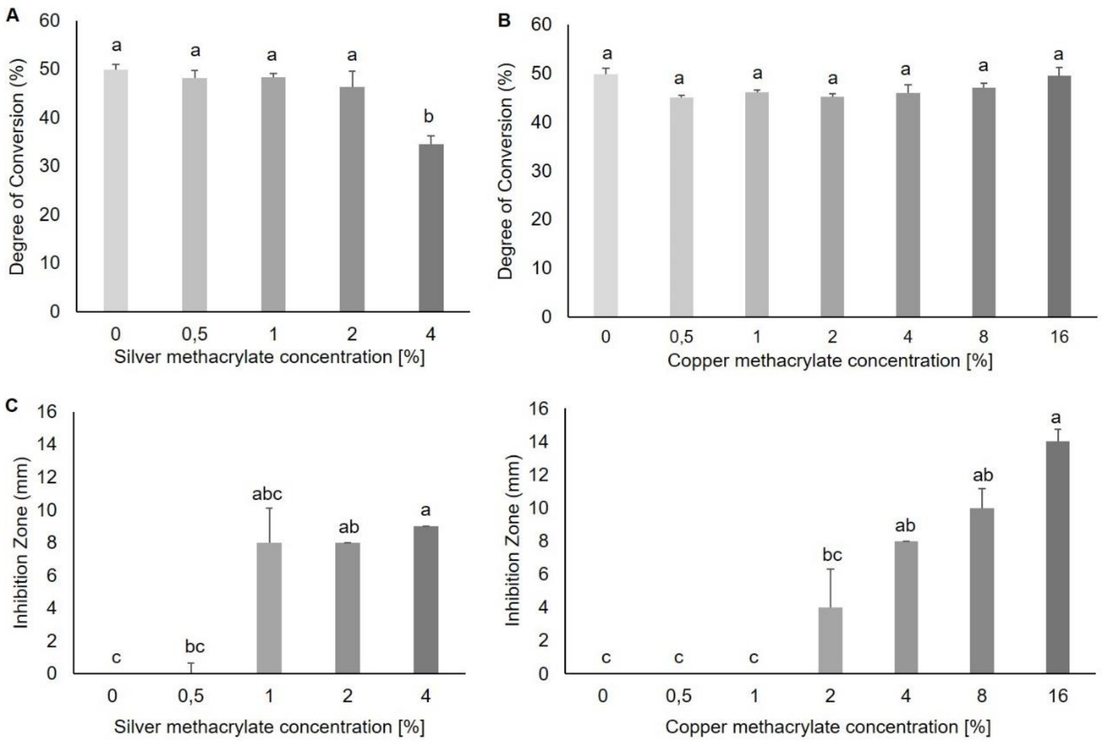


Fig. 2 – Screening test: Degree of Conversion (mean ± standard deviation) and Inhibition Zone (median ± interquartile range) of experimental orthodontic cements with different concentrations of silver (A and C) and copper (B and D) methacrylates. Different letters indicate statistical differences between the experimental orthodontic cements (p<0.05)

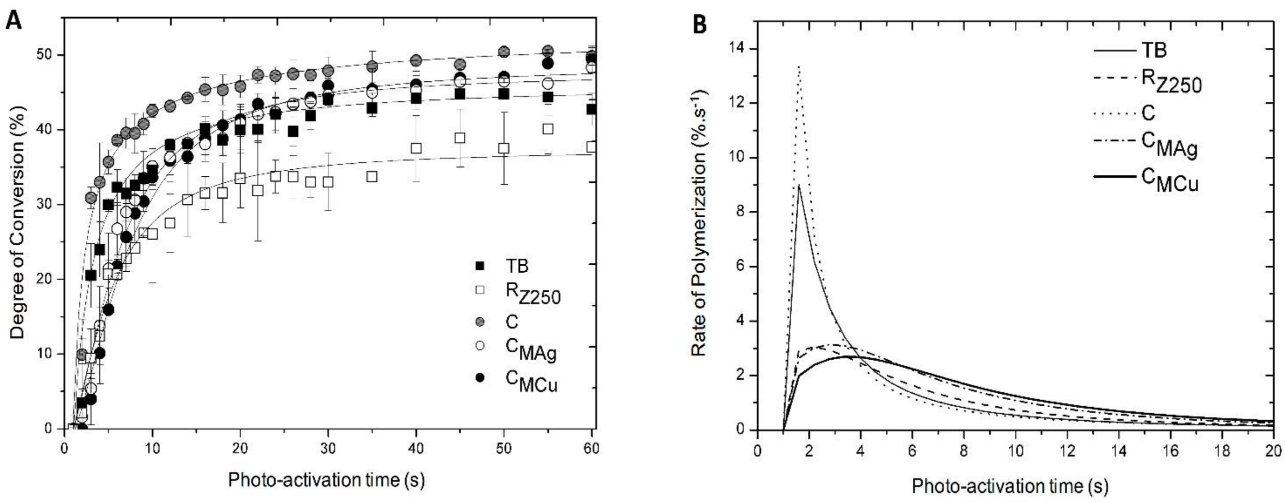


Fig. 3 - Real-time polymerization kinetics (A) and rate of polymerization (B) of the tested bonding agents in 60 s photoactivation time.

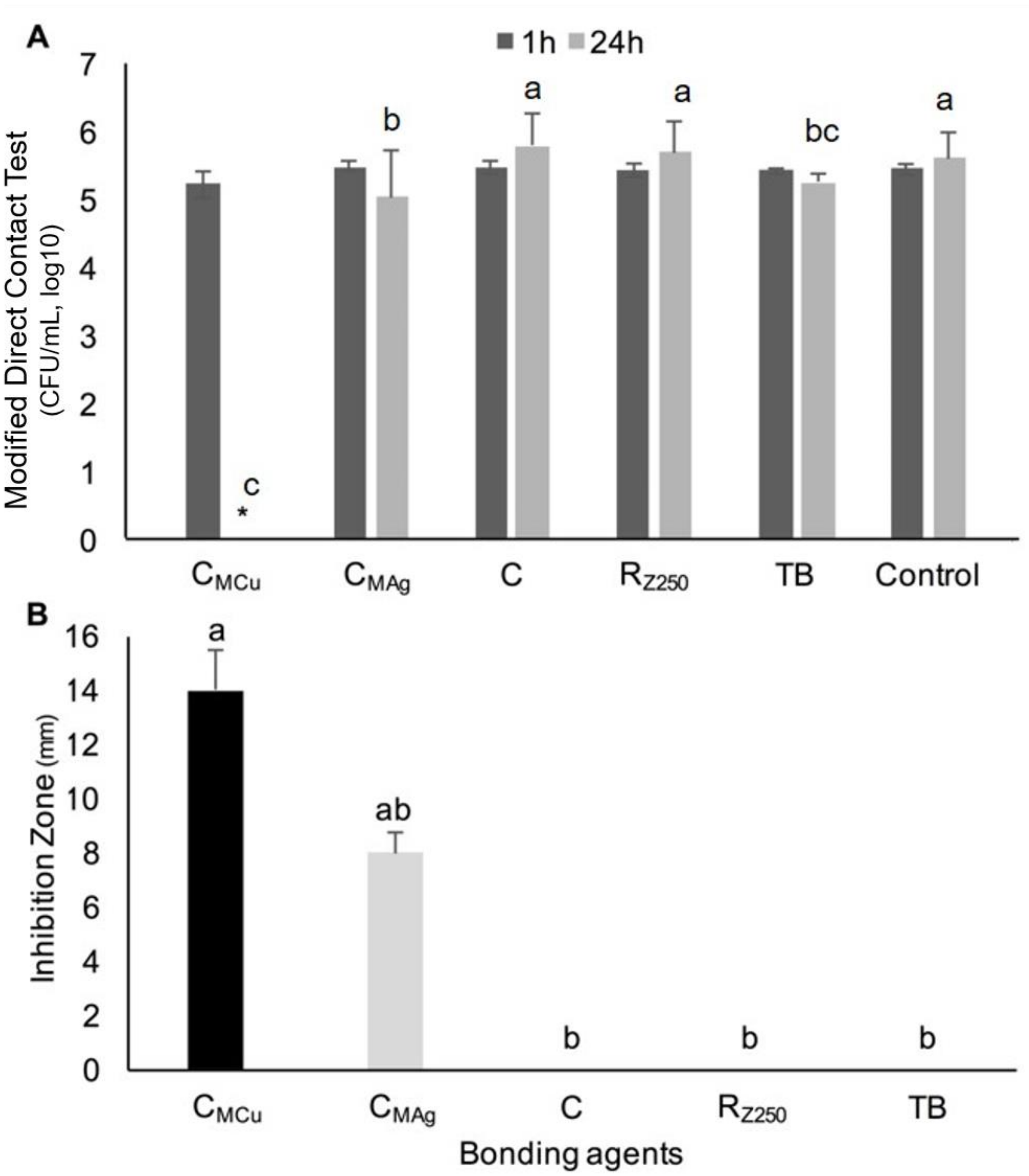


Fig. 4 – Modified Direct Contact Test (A) and Inhibition Zone (B) of the tested bonding agents against *S. mutans*.
*Only C_{MCu} did not show bacterial growth in Modified Direct Contact Test (A).
Different lowercase letters mean statistical differences bonding agents (A and B) ($p<0.05$).

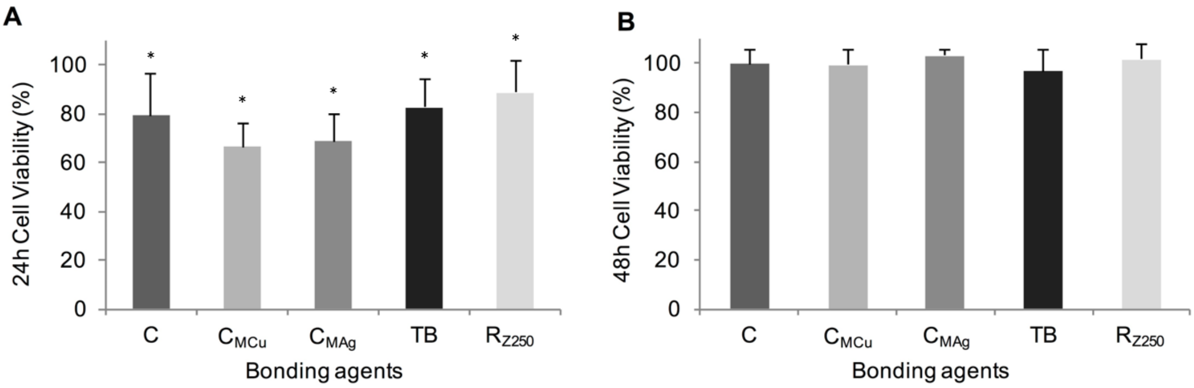
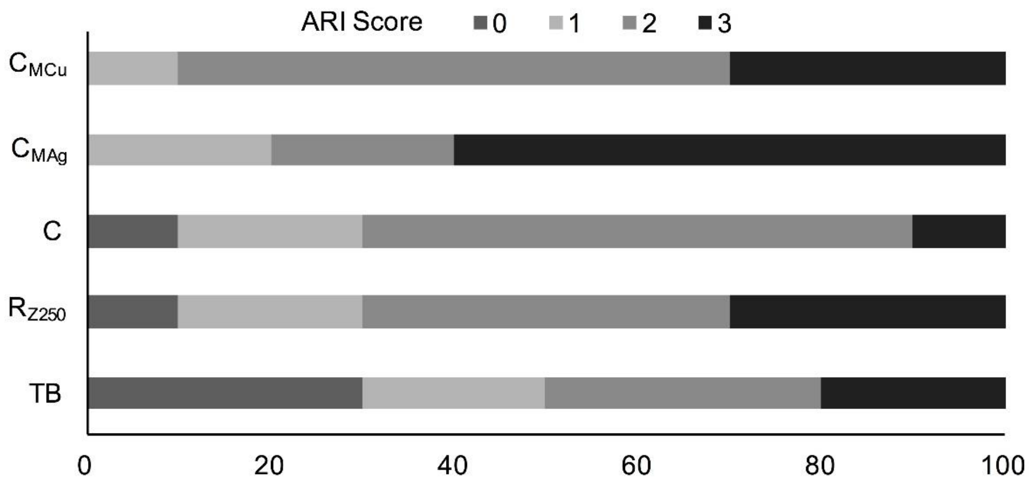


Fig. 5 - Cell Viability and standard deviation (%) of the evaluated cements in 24h (A), with difference between the untreated group and the another groups (*). In 48h (B) cell viability with no statistical diferences observed ($p<0.05$).



Scores used for ARI classification: Score 0: no material remnant on the surface; Score 1: less than half material on the surface; Score 2: more than half material on the surface; Score 3: all bonding agent on the surface, with the impression of the base of the bracket on the material remnant

Fig. 6 - Adhesive Remnant Index (ARI) of the bonding agents evaluated after shear bond strength test.

3 Capítulo 2

Colored orthodontic cement – a novel possibility to prevent enamel damage

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Highlights

Novel contrastant orthodontic cements facilitate the identification of cements in bond and removal process;

Cements may be avoid excess of material around brackets;

Cements can prevent enamel damage.

Abstract

Objective: The objective of this study was to add metal methacrylates on experimental orthodontic cements, copper and silver, and evaluate the influence of cement color change in removal of remaining adhesive, comparing to composite resin Filtek Z250 XT color D3 (R_{Z250} - 3M ESPE, St. Paul, MN, USA) (a commercial reference with detached color in relation to enamel) and references with color similar to tooth: Transbond XT (TB - 3M Unitek, Monrovia, CA, USA) and Experimental orthodontic cement without methacrylate (C). Also, physical and mechanical properties are evaluate and compare to TB, R_{Z250} and C. Color evaluation of the cements, comparing to enamel; dental color evaluation, comparing enamel color pre and post treatment; flexural strength and elastic modulus, surface characterization with Surface roughness (Sa), Enamel Damage Index (EDI) and Adhesive Remnant Index (ARI), quantification of enamel loss by 3D perfilometry (V) and time evaluation of cementation process and bonding agent removal. For all tests it was considered $p<0.05$ as statistically significant. Color of the cements not pigmented enamel surface in tested period and all groups were similar ($\Delta E<2.25$). In surface roughness and enamel loss after the treatment, no differences were observed between groups. EDI was similar between groups. C_{MCu} and C_{MAg} facilitate the material removal, resulting in less time (in seconds) to process (TB=57.4 ±8.3^{A}; R_{Z250}=79.0 ±17.1^{B}; C_{MCu}=32.8 ±3.8^{C}; C_{MAg}=34.0 ±8.6^{C}).

Conclusion: The experimental orthodontic cements with methacrylates showed suitable mechanical properties and aid the process of removal orthodontic bonding agents.

Keywords: Orthodontics, dental materials, bonding agents.

1 Introduction

The introduction of adhesive systems in Odontology promoted an evolution in orthodontics procedures. Brackets were previously welded in metallic bands and bonded using zinc phosphate, but then began to be bonded directly on enamel with advances in adhesion on dentistry. The advantages of this evolution, beyond aesthetics, was the decrease amount of intraoral accessories, enabling improve cleaning the teeth. However, lots of questions appeared about how correctly debond accessories and wear remnant adhesive in the surface after finished the orthodontic treatment. Researchers started the search to the best cleaning technique aiming enamel preservation.[1–12]

Researches suggested wide range of strategies in order to preserve enamel on which the brackets are bonded, such as antimicrobial cements, instruments to cementing agent removal, methods of disclosing for residual cements. Many studies demonstrated that the debond orthodontic accessories and wear of the remnant cement always cause enamel damage independent of the material used[9,10,12–15].

The excess of the bonding agents around the orthodontic brackets can cause plaque accumulation and, consequently, white spot lesions risk[16]. It is reported that 68.4% of orthodontic patients presents white spot lesions[17], and the excess of bonding agents around orthodontic brackets can lead to plaque accumulation and, consequently, caries lesions[16]. Previous studies showed that instruments used to bracket and cement removal are not the main factor on the enamel preservation. The similarity of the cementing agent color with teeth may interfere on the residual material identification, making the removal difficult[18].

Based on these background, the aims of this study are:

a) add silver and copper methacrylate on experimental cement, with the objective to change bonding agent color;
b) evaluate if the color change facilitates the identification of the cement, aiding removal;
c) evaluate if the silver and copper methacrylate addition could affect mechanic properties.

2 Materials and methods

2.1 Experimental design

In this study, three experimental orthodontic cement (C, C_{MCu} and C_{MAg}) and 2 commercial references (Orthodontic Cement Transbond XT - TB - 3M Unitek, Monrovia, CA, USA and resin composite Filtek Z250 color D3 - R_{Z250} - 3M ESPE, St. Paul, MN, USA) were tested for mechanical and optical properties. The concentrations of the metal methacrylates were selected using a screening in a previous study.

2.2 Formulations and Commercial references

The experimental orthodontic cements were prepared in ratio of 3:1 to Bisphenol A glycidyl methacrylate (Bis-GMA - Esstech Inc., Essington, PA, USA) and Tryethyleneglyycol dimethacrylate (TEGDMA - Esstech Inc., Essington, PA, USA). 20% of nanoparticles (silica Aerosil 380, 7nm, Evonik Industries AG Inorganic Materials, Hanau-Wolfgang, Germany). Photo-initiator system was composed of 0.5% of camphoroquinone (CQ - Esstech Inc., Essington, PA, USA), 0.8% for Ethyl 4-dimethylamine benzoate (EDAB - Aldrich Chemical Co., Milwaukee, WI, USA) and 0.4% of Diphenyliodonium hexafluorophosphate (DPIHFP - Aldrich Chemical Co., Milwaukee, WI, USA). Experimental groups were formed, based on a previous screening: experimental orthodontic cement (C), experimental orthodontic cement + 1% silver methacrylate (abcr GmbH & Co. KG., Karlsruhe, Germany) (C_{MAg}) and experimental orthodontic cement + 16% copper methacrylate (abcr GmbH & Co. KG., Karlsruhe, Germany) (C_{MCu}).

Transbond XT adhesive (3M Unitek, Monrovia, CA, USA) (TB) was used as commercial reference to be considered a gold-standard in orthodontic cements. Other commercial reference was composite resin Filtek Z 250 (3M ESPE, St Paul, MN, USA) (RF_{Z250}), D3 color, as “colored” commercial reference.

2.3 Color evaluation

The difference of color between enamel and bonding agents were perfomed with specimens (6x1mm, n=10) made with the aid of two glass plates, polyester strip on a metal matrix, and the bonding agent dispensed into the mold and light cured for 20s on each side (Radii Cal; SDI, Bayswater, Victoria Australia). The enamel samples were made in the same dimensions, cutting bovine crowns cattle central incisors, with drill for glass cutting[19].

The color readings / translucency of the materials tested were performed by measurement of parameters L*, a * and b *, based on the Commission Internationale de l'Éclairage (CIE) system with a spectrophotometer (SP60 - EX-Rite / Grand Rapids - Michigan, USA), on white background. The device was calibrated according to the manufacturer's recommendations, through a white and black pattern that accompanies the device.

Mean color values of the sample bonding agents were compared to the mean color values of the enamel sample. The variation of CIELab color was calculated and indicated the diference of cement and enamel color, following the equation:

$$\Delta E_{00}=[(\Delta L' / k_L S_L)^2 + (\Delta C' / k_C S_C)^2 + (\Delta H' / kHSH)^2 + R_T (\Delta C' / k_C S_C)(\Delta H' / k_H S_H)]^{1/2}$$

The values of CIELab were converted in CIEDE2000 color formula (ΔE_{00}).[20] Cement that presented $\Delta E_{00}>2.25$ in relation to the enamel, were considered considered perceptible to the human eye, desirable for our cements.[21]

2.4 Dental color evaluation

Buccal surface of enamel bovine incisors was evaluated using the same parameters CIEDE2000 to analyze if bonding agents could pigment teeth. Values for L, a and b were obtained with VITA EasyShade (VITA Zahnfabrik, Bad Säckingen, Germany) prior to cementation and after removal of orthodontic bonding agent remaining on the dental enamel.

These data were compared in order to assess whether the material is able to change the color of the enamel. For the second evaluation, the teeth were stored in distilled water at 37 ° C for 24 hours and 30 days (n=3/time). The methodology used was described previously[22]. To compare the difference of color between the times statistical analysis was performed using One Way Analysis of Variance and data were transformed to log10. ($p<0.05$)

2.5 Mechanical properties

2.5.1 Flexural strength and elastic modulus

Bars (10×2×2mm) were made with the bonding agents, inserting cement in a metal matrix, cover with polyester and glass blade and photo-activeted using two light exposure (20s on each extremity of matrix) (n=15). After polimerization, bars were removed of the matrix, polish with sandpaper grit SiC #600 under cooling and stored in water at 37ºC for 24h. After this time, dimensions were measured with digital paquimeter (Mitutoyo, Tokio, Japan) and these were submited to the flexural test - flexure resistance of three points - on universal test mechine (DL500; EMIC, São José dos Pinhais, PR). The load was applied in the central region of the bar, with the 8mm of distance between supports, and the speed 0,5mm/min up to failure. Flexural strength (σf) and elastic modulus (Ef) were evaluated with the test. The comparisons between groups were performed using One Way Analysis of Variance and Holm-Sidak method with data transformed to log10 for flexural strength and Kruskal-Wallis and Tukey test for elastic modulus. ($p<0.05$)

2.6 Surface characterization and quantification of enamel loss

2.6.1 3D Perfilometry

Twenty-five bovine incisors were used in the form of discs, with 8mm diameter. The discs were flat and polished with sandpaper grit SiC # 600, # 1200 and # 2500. After, they were embedded in acrylic resin in aluminum tube support (3x3x1,5cm). After, prophylaxis was performed with pumice paste and rubber cup and specimens subjected to ultrasound cleaning. The topographical analysis of the enamel surfaces were performed using a 3D profilometer (Form Talysurf 60i, Taylor Hobson, Leicester, UK). After planning and polishing the surface, an 3 x 2 mm area on the enamel was delimited Then, the surfaces were scanned with a resolution of 20 nm in the z axis, 60,000 points on the x axis and spacing of 4 µm in the y-axis. After the orthodontic brackets were bonded in the defined área in the enamel, removed after 24h, the surfaces treated according to the experimental protocol and scanned again using the same previous parameters (n=5). The roughness of the surfaces (Sa) and volumn (V) of enamel before and after the finish was obtained by Sa parameter (average of absolute deviations) and three-dimensional topographical images were derived using the following equation:

$$Sa = \frac{1}{A} \iint_A |Z(x,y)| dxdy,$$

Where Z is the height of measured points at the coordinates x and y.

Two Way Repeated Measures ANOVA (One Fator Repetition) and Student-Newman-Keuls method were used to compare the influence of cement (different groups) treatment (before vs after) in Sa and V. For Sa data were transformed to square root. ($p<0.05$)

2.6.2 Optical Microscopy and Enamel Damage Index

Sample submited to 3D-Perfilometry (n=5/group), were submited to Optical Microscopy analisys (MO) (BX41M-LED, Olympus, Tokyo, Japan), with 40x, 100x and 200x magnification. Images were made before cementing brackets and after remotion of bonding agents to the teeth surfaces. The images provided qualitative data to the dental surface.

Through the images obtained in the MO, the surfaces were classified by Enamel Damage Index, suggested by Schuler and Van Waes (2003)[23] and used for other researchers[11,24], by following scores:

* Score 0: Smooth surface without scratches, and perikymata might be visible;

* Score 1: Acceptable surface, with fine scattere scratches;

* Score 2: Rough surface, with numerous coarse scratches or slight grooves visible;

* Score 3: Surface with coarse scratches, wide grooves, and enamel damage visible to the naed eye.

To compare the scores of EDI initial and final in the same group, Wilcoxon Signed test were performed ($p<0.05$). To compare EDI final between groups Chi-square test was performed ($p<0.05$).

2.6.3 Adhesive Remnant Index

After debonding brackets to the teeth surfaces with plier, the adhesive remnant index was evaluated, as described for Årtun and Bergland (1984)[25]. The remnants were classified following the scores:

* Score 0: no material remnant on the surface

* Score 1: less than half material on the surface;

* Score 2: more than half material on the surface;

* Score 3: all bonding agent on the surface, with the impression of the base of the bracket on the material remnant.

A descriptive analysis was performed.

2.6.4 Scanning Electron Microscopy

The topography of the images was qualitatively assessed by scanning electron microscopy - SEM (PhenomProX, PhenomWorld, Eindhoven, Netherlands). The images were taken in low vacuum environment, with 15 KV acceleration and expansion of 250x and 1000x.

2.7 Time evaluation of cementation process and bonding agent removal

The cementation process of orthodontic brackets has been timed, as well as the cement wear process that remained on the enamel. The objective was to verify if the cement color change facilitated the two processes.

The cementation process included all clinical steps for cementation of orthodontic brackets: application of 37% phosphoric acid for 30 seconds, rinsed for 15 seconds with water-air spray and dried with air to obtain whitish surface, applying adhesive (TB, C, C_{MAg} and C_{MCu} groups used Transbond XT adhesive and R_{Z250} group used Scotch Bond adhesive) and drying flow of excess adhesive, light curing for 20 seconds; cementing bracket and photoactivation by 20 seconds on each side (n=6).

The removal process was performed in an environment simulating clinical practice. A device was made to couple the sample on the headrest and coupling of the dental suction unit, under illumination of operating light.

To compare the difference of cement and removal process statistical analysis was performed using One Way Analysis of Variance with data transformed to log10 and One Way Analysis of Variance and Holm-Sidak method, respectively.

3 Results

3.1 Color evaluation

The results for the cement color analysis, in comparison to enamel color are shown in Fig. 1. C_{MCu} (ΔE=23.08) and C_{MAg} (ΔE=15,96) showed the higher difference of color to the enamel.

3.2 Dental color evaluation

The color analysis of the samples, before the bracket bonding and after bonding agent removal, is presented in Fig. 2. No difference was observed before and after treatment, both in 1 hour and 24 hours, showing that in time evaluated no color difference was observed in samples.

3.3 Flexural strength (σf) and elastic modulus (*E*f)

Mechanical properties are presented in the Table 1. Both in σf and *E*f, experimental orthodontic cements were similar and different to commercial references.

3.4 Surface characterization and quantification of enamel loss

3.4.1 3D Perfilometry (Surface roughness and Volume loss)

Table 2 shows the mean values for surface roughness of samples, comparing the roughness before orthodontic brackets cementation (Sa_i) and roughness after removal bonding agent remained (Sa_f). No difference was observed between the groups on each time. There was influence of treatment between Sa_f and Sa_i for all groups. ($p<0.05$) Table 3 shows the volumes (vol/mm^3) of enamel surface before the treatment and after debond of orthodontic brackets and removal remnant orthodontic cement. No difference was observed between the groups on each time. There was influence of treatment between V_f and V_i for all groups. ($p<0.05$)

Fig. 3 shown three-dimensional topographical images of enamel before (3a) and after (3b-f) bonding agents removal.

3.4.2 Optical Microscopy, Enamel Damage Index and Adhesive Remnant Index

Enamel Damage Index was performed based on Optical Microscopy and is shown in Table 4. Adhesive Remnant Index is shown in the same table.

3.4.3 Scanning Electron Microscopy

The images of SEM are shown in Fig. 4 (a-f).

3.5 Time evaluation of cementation process and bonding agent removal

The analysis of bonding time and removal of bonding agents time are presented in Table 5. C_{MCu} and C_{MAg} required less time to remove remnant adhesive. C presented ARI 0 in all specimens during debonding brackets, so it was not possible to calculate the time for removal.

Discussion

The incorporation of metallic methacrylates into experimental orthodontic cement provided a significant color change in the material in relation to the color of the enamel. The addition of copper methacrylate became material blue and the material with silver, brown. This pigmentation aimed to viewing the product, both in the cementation of orthodontic accessories, help cleaning of excess prior to curing and facilitate the identification of remaining cement on enamel, serving as a guide for removal of residual cement, trying to avoid enamel damage. This color change is considered perceptible to the human eye, as experimental orthodontic cements showed ΔE greater than 2.25 (C_{MCu} ΔE=23.08; C_{MAg} ΔE=15.96) when compared with the enamel[21]. The cleaning of residual bonding agent on the enamel after removal of the brackets can not be complete, thus cement residues can remain on the tooth[11], which can be particularly difficult to identify and to view and can facilitate enamel damage[18]. The persistence of any residual material on the tooth could help plaque retention[15], and can result in staining of residual material[26].

In this study, carbide tungstein bur at low speed for removal of residual bonding agent was used, the gold standard for removal of residual cement, reported in the literature as being the least causes damage[15]. Other studies reported that regardless of the instrument used for cement removal, there will always be some damage to the tooth structure and these damages are irreversible[9,10,12–15].

The samples were evaluated for surface characterization after removal of residual bonding agents to surface roughness and the amount of tooth structure loss after removal of the cement. With 3D perfilometer we verified that the addition of metal methacrylates did not interfered significantly in the surface roughness of teeth. In all test groups, an increase in roughness comparing the pre and posttreatment, but there was no difference between the groups tested (TB, R_{Z250}, C, C_{MCu} and C_{MAg}). After treatment, on mean, the groups presented a Saf = 0,277μm roughness. So this increase in roughness may be more related to the accessory used for the removal of cement, than the various cements, agreeing with the findings of Pont et al., 2010[12]. These authors[12] suggests that the use of rotary instruments causes an increase in surface roughness resulting in a surface with scratches and irregularities which may cause an increase in bacterial biofilm retention, even so when using the gold standard instrument for the removal of the remaining cement[15]. Literature also suggests that damage is operator-dependent and, regardless of the material used, there will be damage[11,15]

The pigments added to the experimental orthodontic cements were not able to change the color of the enamel. ΔE (color pre and post treatment) was not more than 2.25, regarded reference limit be identified by the human eye, in any of tested groups. If that color change occurred, not necessarily the explanation would be the pigment. The literature reports that the increase in surface roughness can result in color change of the structure. The literature reports that changes in surface roughness caused for example by polishing, may influence the L * parameter[27] and, consequently, the ΔE.

The literature also points out that the damage to the enamel surface may depend on the operator[12,15,28]. In our case, this is not a factor relevent, because the operator is an experienced orthodontist.

Our results disagree with the results presented by Ferreira et al. (2014)[29]. The authors aimed to evaluate the effect of different polishing methods. When they used a similar polishing system used to us, the surface roughness decreased, the final roughness was lower than in the inicial test. In our study, we found that after cement removal with tungsten carbide bur at low speed and polishing with pumice paste and rubber cup, the surface roughness increased. Perhaps the difference between results are due to the fact that we use different polishing times. The authors used polishing for 40 seconds while we carry out the polishing for 10 seconds only. Besides to be related to a suitable polishing[30], our main objective was to evaluate the efficiency of color change in residual cement removal and not evaluate the polishing, which could interfere with the results we needed check.

The obtained surface roughness has particular importance. A surface is considered aesthetically acceptable when has reflectivity as enamel, having minor surface irregularities than 1 micrometers. The enamel roughness considered pattern is 0,64µm ± 0.25[31]. Patients underwent susceptibility testing roughness, which were evaluated for the ability to detect, with the tongue, the presence of roughness. Values from 0.5 µm could be perceived by the tongue[32]. Another importance of surface roughness is the ability to retain biofilm on the enamel surface[12]. The literature suggests that higher values than 0,2µm for roughness are able to retain biofilm, so increasing the possibility of developing caries and periodontal problems[33].

The act of bonding and debonding of orthodontic brackets that are required to perform orthodontic treatment unfortunately causing an irreversible change in the structure of the enamel and these changes may cause the enamel color alterations[27,34,35]. Modifications to the enamel structure, such as surface roughness, confer change in value represented by the parameter L * CIELab system.[34]

Although the findings of this study, showing the roughness, it has remained around 0,277µm, not reaching the threshold considered critical of 1µm - to change the enamel reflection and negatively affect the aesthetics[36]. Probably this was the reason for no color change of the samples after 24 or 30 days after cement contact with the enamel, although there was an increase increased roughness. The sample color was evaluated prior to cementing of the brackets and after removal of the cement (24 and 30 days after cementation). Through the formula CIEDE2000 was possible to verify that, after 24 hours, none of the bonding agents were able to change significantly the natural enamel color, ΔE for the groups not passed the threshold considered clinically apparent[21]. After 30 days, the same ratio was obtained, showing that none of the bonding agents, even those with addition of metal methacrylates to give distinguishable color, was able to change the natural enamel color for way that is perceptible to the human eye.

The enamel loss after removal of residual cement agent was not statistically different between the groups tested. This can be explained by the fact that it was performed by an experienced professional. As pointed out by Al Shamsi et al.[28] and Pont et al.[12] the damage caused to the structure, may depend on the operator. An active professional that is careful about the quality of services offered to your patients, coupled with many years of experience can take neutralizing effect on operator dependent factor at the procedure avalued. This may have been the case during the course of this experiment in this work. However, if the removal procedure of residual cement agent after removal of orthodontic brackets are made by inexperienced professionals or orthodontists poor expertise, possibly a colored material could have be safer considering lower levels of roughness and loss of tooth enamel after completed surface treatment. The values obtained in our study to ename loss are in disagreement with the literature. Tüfekçi et al. (2004)[37], assessing the amount of enamel loss after the removal of the remaining removing enamel surface of the adhesive have found results for the loss were 0.06mm^3, by using na instrument similar to that used in the present study. In this study the volume loss of enamel was between 0.000619 and 0.002025mm^3. Ryf et al. (2012)[38] also had higher values with a mean volume loss of 0.02mm^3. Such differences may have occurred due to differences between the materials used and problable also the difference between the equipment used to make the measurement[39]. Above all, it is important to consider that an experienced and qualified professional can serve as a confounding factor in studies aiming to develop materials for easier use.

As mentioned there is always a chance that, in the surface cleaning process, cements remain on the structure[11]. If bonding agentes color were similar to the enamel color, the residues that remain on the surface are difficult to view. This can be a disadvantage since material it remains unremoved can aid in biofilm formation, so increased risk of development of caries and periodontal disease, possibility color change and other effects. In this study there was no statistically significant difference for enamel volume loss and colored cements did not have the advantage of a significant reduction of the enamel damage.

For flexural strength, commercial references showed higher values than the experimental cements. C, and C_{MCu} C_{MAg} were similar, demonstrating that the addition of the methacrylate in relation to the internal control, resulted in no significant changes. Even with reduced values for flexural strength, all experimental groups had higher values than recommended by ISO 4049[40] ($\sigma f>50MPa$). The same behavior can be seen for the elastic modulus. The addition of potential antimicrobial agents has been added to the material and tested for their interference on the mechanical properties of the products. Benzalkonium chloride at various concentrations tested did not significantly interfere in the mechanical properties.[41] The addition of benzalkonium chloride, chlorhexidine and Triclosan in a commercial reference did not affect the tested mechanical properties, agreeing with the findings of Othmann et al. (2002)[41,42]. Also, Cetylpyridinium chloride was added to an orthodontic cement without interference diametral strength.[43]

For bonding the orthodontic brackets, colored experimental orthodontic cements needed more time, but the difference was not statistically significant. Probably all excess of material that remains on the enamel was easily identified and removed before curing. For the removal time of the remaining bonding agent on enamel, our groups ranged between 32.8 and 79s, including, in this time, the bonding agent wear with tungsten bur at low speed and the polishing with pumice paste and water and rubber cup (for 10s). C_{Mu} and C_{MAg} had significantly lower values than the other groups, demonstrating that the color change can assist in the removal phase. Krell et al. (1993)[5] found in their study that the time to debonding, wear to the bonding agent and finishing with abrasive discs was, on mean, 113s, higher than those found in our study. Another factor that may have interfered in the time to bonding agent wear is the consistency of the material. The fact that the experimental orthodontic cements having 20% inorganic filler made it to be easier to wear, thereby reducing the working time, while commercial references TB and R_{Z250} have 70-80% and 60% of inorganic filler particles, respectively, and more time was necessary to be totaly removed.

6 Conclusion

The addition of colored metal methacrylate facilitates the residual cement removal proven by less removal time. Even so, it was not able to prevent damage to enamel when performed by an experienced professional because it showed no differences in surface roughness and loss of enamel volume compared to the materials without color. More studies should be conducted to assess if the removal by inexperienced professionals will be improved by the colored material.

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Tables

Table 1. Mean ± standard deviation (SD) of flexural strength in MPa (σf) and median and interquartile range (q1-q3) of elastic modulus in GPa (Ef) of bonding agents (n=12).

Groups	σf Mean (SD)	*E* f Median (q1-q3)
TB	115.9 (19.11)^{A}	3591.9 (2991.4-4165.3)^{A}
R_{Z250}	120.4 (21.55)^{A}	5544.5 (4823.8-6510.7)^{A}
C	75.5 (13.45)^{B}	1443.7 (1226.6-1539.4)^{B}
C_{MAg}	73.5 (7.05)^{B}	1249.3 (1206.0-1488.4)^{B}
C_{MCu}	67.1 (10.03)^{B}	1222.7 (1024.6-1299.7)^{B}

Different letters in the same column indicate significant differences between materials (p <0.05).

Table 2. Mean ± standard error (SE) Initial (Sa_i) and final (Sa_f) surface roughness (Sa, n=5) and difference between stages.

Groups	Sa_i	Sa_f	Difference
TB	0.127 (0.03)^{Aa}	0.234 (0.04)^{Ab}	0.107
R_{Z250}	0.187 (0.09)^{Aa}	0.288 (0.02)^{Ab}	0.101
C	0.103 (0.03)^{Aa}	0.199 (0.06)^{Ab}	0.096
C_{MCu}	0.067 (0.01)^{Aa}	0.338 (0.14)^{Ab}	0.271
C_{MAg}	0.104 (0.02)^{Aa}	0.326 (0.07)^{Ab}	0.223
Grouped averages	**0.117^{a}**	**0.277^{b}**	**0.160**

Difrerent uppercase letters in columns representes difference between groups in the time; different lowercase letters in lines representes diferences between Sa_f and Sa_i. (p<0.05)

Table 3. Mean ± standard error (SE) of initial volume (V_i), final volume (V_f) and the difference between the volumes, in cubic millimeters (mm^3).

Groups	V_i mm^3 (SE)	V_f mm^3 (SE)	Volume difference
TB	0,001042 (0,000228)^{Aa}	0,002065 (0,000209)^{Ab}	0.001023
R_{Z250}	0,001597 (0,00061)^{Aa}	0,002216 (0,000169)^{Ab}	0.000619
C	0,000733 (0,000171)^{Aa}	0,001376 (0,000387)^{Ab}	0.000643
C_{MCu}	0,00053 (0,000077)^{Aa}	0,002555 (0,001004)^{Ab}	0.002025
C_{MAg}	0,000845 (0,000206)^{Aa}	0,002432 (0,000535)^{Ab}	0.001588
Grouped averages	**0.0009494^{a}**	**0.002129^{b}**	**0.001179**

Different uppercase letters in columns representes difference between groups in the time; different lowercase letters in lines representes diferences between Sa_f and Sa_i. ($p<0.05$)

Table 4. Distribution of Enamel Damage Index (EDI) scores before and after treatment and Adhesive Remnant Index (ARI) scores (n=5)

Groups (Index)	Grade 0	Grade 1	Grade 2	Grade 3
$C_{EDI\ initial}$	0	5	0	0
$C_{EDI\ final}$	0	4	1	0
***C** ARI*	***4***	***0***	***0***	***1***
$C_{MAg\ EDI\ initial}$	0	5	0	0
$C_{MAg\ EDI\ final}$	0	1	3	1
$C_{MAg\ ARI}$	***0***	***1***	***1***	***3***
$C_{MCu\ EDI\ initial}$	0	5	0	0
$C_{MCu\ EDI\ final}$	0	4	0	1
$C_{MCu\ ARI}$	***0***	***0***	***0***	***5***
$TB_{EDI\ initial}$	0	5	0	0
$TB_{EDI\ final}$	0	3	2	0
TB_{ARI}	***1***	***0***	***0***	***4***
$R_{Z250\ EDI\ initial}$	0	5	0	0
$R_{Z250\ EDI\ final}$	0	0	3	2
$R_{Z250\ ARI}$	***0***	***2***	***0***	***3***

No statistical difference between EDI initial and final. ($p<0.05$) No statistical difference between groups in EDI final. ($p<0.05$)

Table 5. Mean ± standard deviation of bonding time and removal time of bonding agents, in seconds.

Bonding Agents	Bonding time	Removal
TB	40.4 (5.3)[A]	57.4 (8.3) [A]
R_{Z250}	39.4 (8.2)[A]	79.0 (17.1) [B]
C	38.0 (14.4)[A]	-
C_{MCu}	55.6 (12.7)[A]	32.8 (3.8) [C]
C_{MAg}	44.0 (13.5)[A]	34.0 (8.6) [C]

Different uppercase letters in the same column indicate significant differences between materials ($p < 0.05$).

Figures

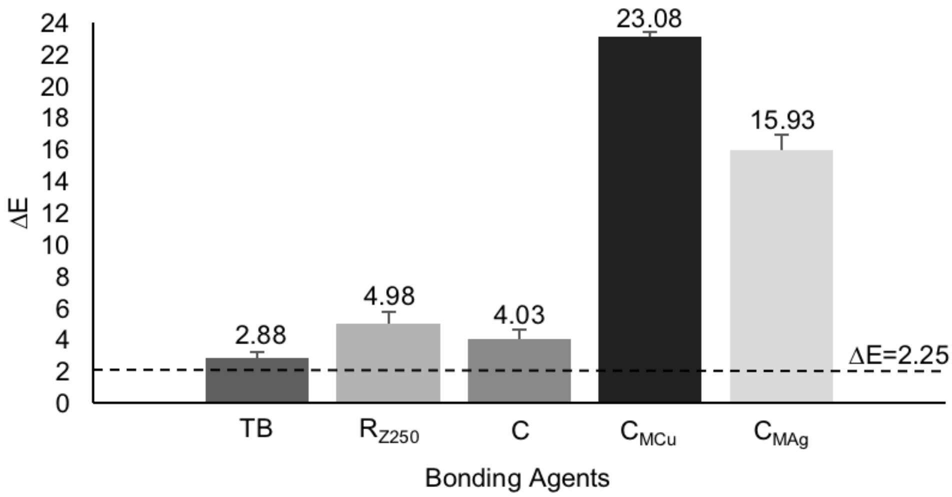

Fig. 1 - Color difference (ΔE) (mean ± SD) between orthodontic bonding agents tested and the enamel surface

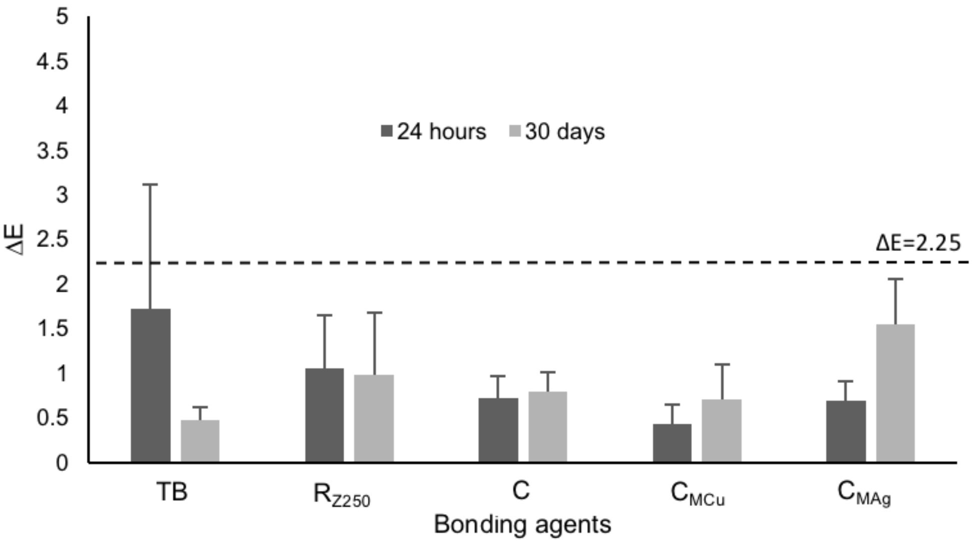

Fig. 2 - Enamel color change (ΔE) (mean ± SD), after removal bonding agent in contact with the enamel for 24 hours and 30 days. No significant difference between the groups.

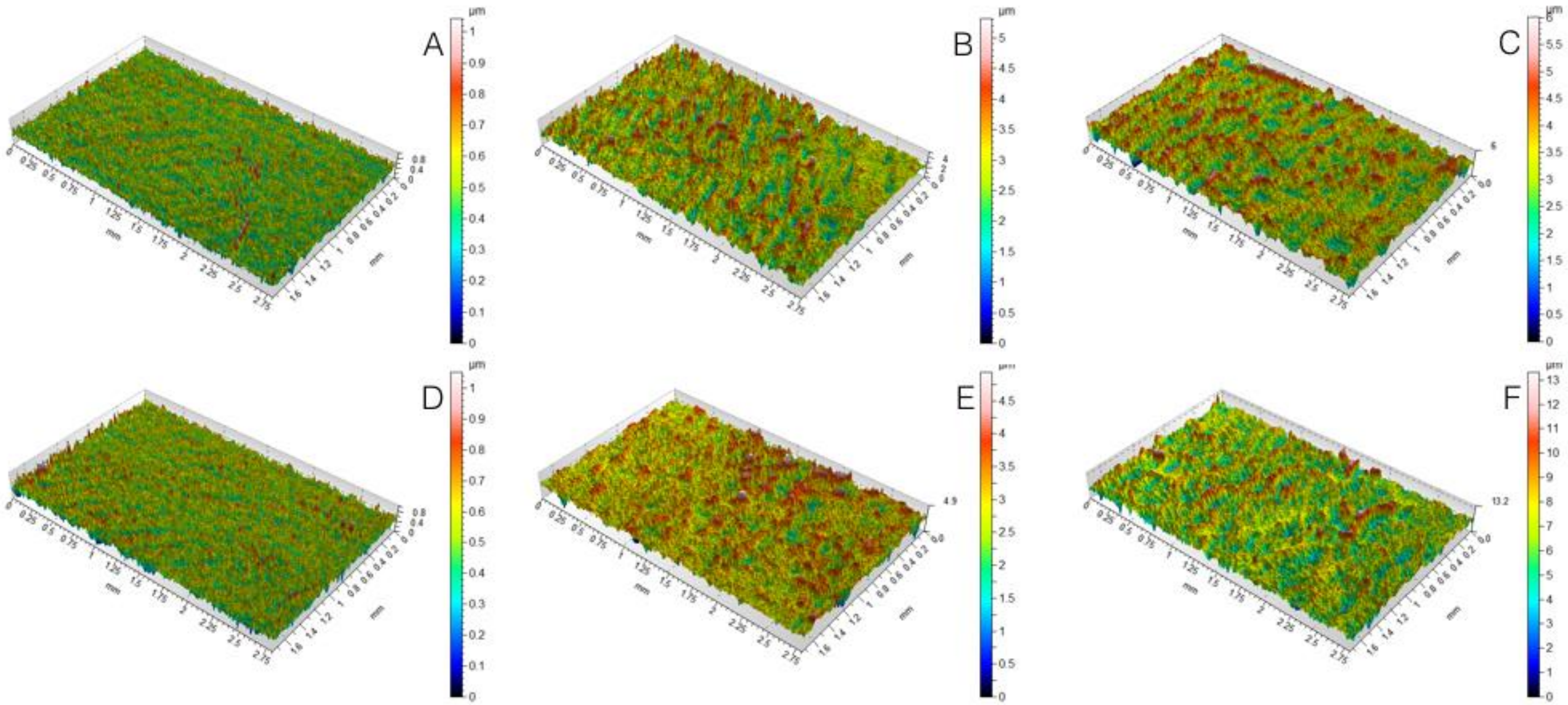

Fig. 3 - Three-dimensional topographical images of enamel before (3a) and after (3b-f) bonding agents removal. A - Grounded enamel; B – TB; C - Z250; D – C; E - C_{MAg} and F – C_{MCu}.

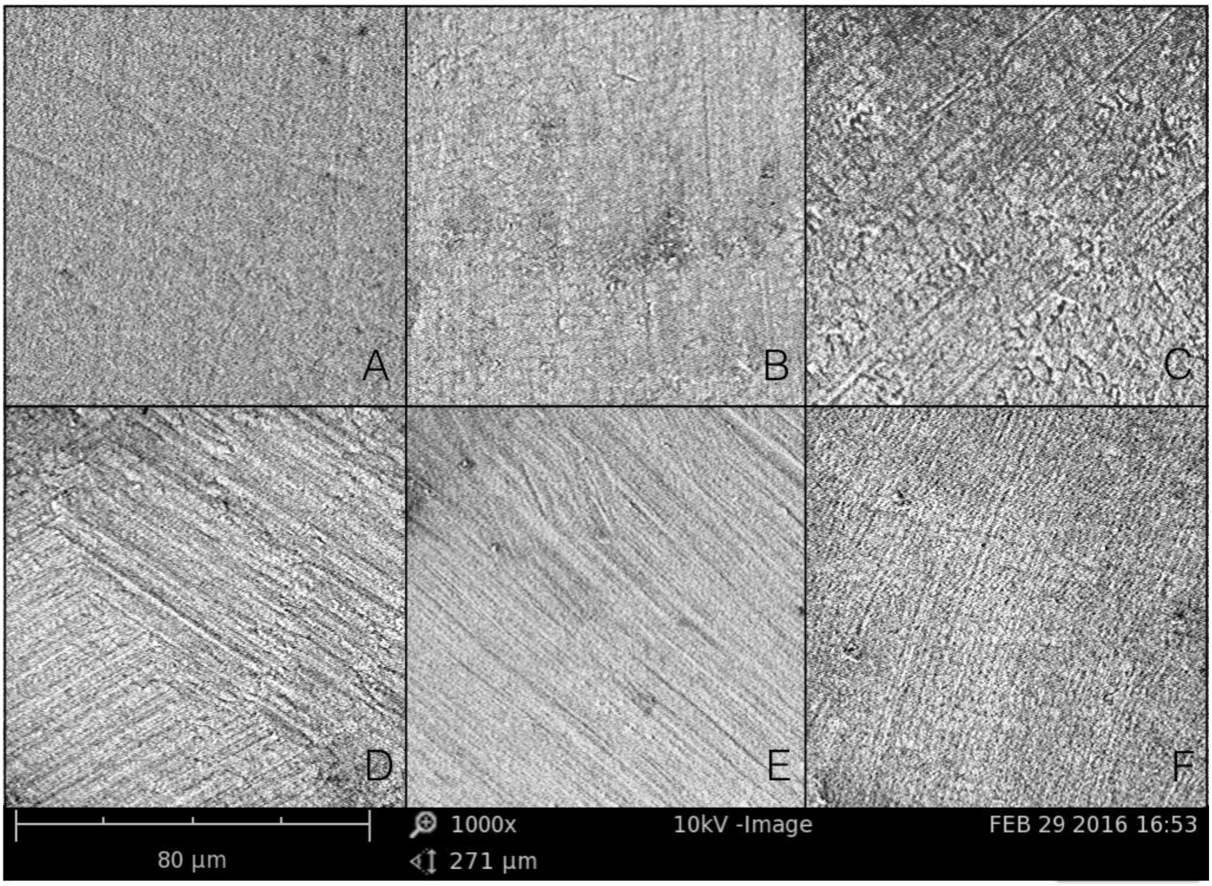

Fig.4 - Characterization of enamel by Scanning Electron Microscopy. A - Grounded enamel; B – TB; C - Z250; D – C; E - C_{MAg} and F – C_{MCu}

4. Considerações finais

A adição dos metacrilatos metálicos possibilitou uma melhora no cimento ortodôntico experimental, possibilitando que ele fique facilmente visível, tanto no momento da cimentação do acessório ortodôntico quanto no momento da remoção do cimento residual, após a descolagem dos bráquetes. O metacrilato de cobre possibilitou, ainda, um efeito antimicrobiano no período testado, contra *S. mutans.*

A partir de agora, novos estudos devem ser realizados com o objetivo de avaliar o efeito do material colorido na remoção do cimento remanescente com operadores inexperientes e o efeito antimicrobiano a longo prazo, proporcionado pela adição dos matcrilatos metálicos.

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Anexo

Anexo A – Depósito de Patente

INSTITUTO NACIONAL DA PROPRIEDADE INDUSTRIAL

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Pedido nacional de Invenção, Modelo de Utilidade, Certificado de Adição de Invenção e entrada na fase nacional do PCT

Número do Processo: BR 10 2016 016883 0

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Dados do Pedido

Natureza Patente: 10 - Patente de Invenção (PI)

Título da Invenção ou Modelo de Utilidade (54): COMPOSIÇÕES ODONTOLÓGICAS ANTIBACTERIANAS CONTENDO METACRILATOS METÁLICOS POLIMERIZÁVEIS

Resumo: A invenção refere-se a materiais odontológicos poliméricos que contenham metacrilatos metálicos na formulação de resinas odontológicas, sistemas adesivos, selantes, cimentos e compósitos. Os materiais apresentam variadas aplicações em odontologia nas áreas de prótese dentária, dentística, ortodontia, odontopediatria, implantodontia, endodontia. Em particular, a presente invenção refere-se a materiais odontológicos antimicrobianos que contenham metacrilatos metálicos na formulação, como metacrilato de cálcio, de estanho, de cobre, de prata, de níquel, de titânio, de ferro.

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Documentos anexados

Tipo Anexo	Nome
Comprovante de pagamento de GRU 200	comprovante de pagamento.pdf
Declaração de período de graça	divulgação não prejudicial.pdf
Declaração de período de graça	divulgação não prejudicial 1.pdf
Portaria	Decreto de Nomeação do Reitor_UFPel.pdf
Procuração	PROCURACAO.pdf
Reivindicação	Reivindicações Metacrilatos Metálicos.pdf
Resumo	Resumo Metacrilatos Metálicos.pdf
Relatório Descritivo	Relatório Descritivo Metacrilatos Metálicos.pdf

Acesso ao Patrimônio Genético

☑ Declaração Negativa de Acesso - Declaro que o objeto do presente pedido de patente de invenção não foi obtido em decorrência de acesso à amostra de componente do Patrimônio Genético Brasileiro, o acesso foi realizado antes de 30 de junho de 2000, ou não se aplica.

Declaração de Divulgação Anterior Não Prejudicial

☑ Artigo 12 da LPI - Período de Graça.

Declaração de veracidade

☑ Declaro, sob as penas da lei, que todas as informações acima prestadas são completas e verdadeiras.

Nota da Dissertação

Efeito da adição de monômeros funcionalizados em um cimento ortodôntico experimental na prevenção de danos ao tecido dental

Effect of monomers functionalized addition in an experimental orthodontic cement to prevent damage to dental tissue

A presente dissertação de mestrado desenvolveu cimentos ortodônticos experimentais, com adição de potenciais agentes antimicrobianos – Metacrilato de Cobre e Metacrilato de Prata. A adição destes produtos visou alterar a coloração dos materiais ortodônticos (e assim facilitar a remoção do cimento remanescente sobre a estrutura dental), além de testá-los para o efeito antimicrobiano sobre o principal microorganismo causador da cárie. Propriedades físicas, químicas e mecânicas dos produtos foram testadas, cmparando-os sempre com referências comerciais padrão-ouro. Análises qualitativas e quantitativas de perda do esmalte foram realizas, a fim de identificar se a alteração de cor do produto realmente facilitou e evitou o desgaste iatrogênico do esmalte.

Campo da pesquisa: Ortodontia, Materiais Odontológicos, Microbiologia

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Data da defesa e horário: 27/07/2016

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Orientador: Prof. Dra. Giana da Silveira Lima
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