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INFLUÊNCIA DA ADIÇÃO DE SILICATO DE NIÓBIO NAS CARACTERÍSTICAS FISICO-QUÍMICAS DE UM ADESIVO ORTODÔNTICO EXPERIMENTAL

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Dissertação apresentada como requisito obrigatório para a obtenção do título de Mestre em Odontologia, área de concentração Clínica Odontológica - Materiais Dentários.

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Orientador

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“Cada um é o artífice de sua própria sorte”

Appius Claudius Caecus (340 – 273 a.C.)

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RESUMO

PINTO, L. **INFLUÊNCIA DA ADIÇÃO DE SILICATO DE NIÓBIO NAS CARACTERÍSTICAS FISICO-QUÍMICAS DE UM ADESIVO ORTODÔNTICO EXPERIMENTAL.** Dissertação – Faculdade de Odontologia da Universidade Federal do Rio Grande do Sul, Porto Alegre, 2021.

O objetivo desta dissertação foi sintetizar e caracterizar o silicato de nióbio (SiNb) por meio do método sol-gel utilizando o pentacloreto de Nióbio ($NbCl_5$) e o tetraetilortosilicato como precursor da sílica para ser utilizado como partícula de carga bioativa em uma resina adesiva ortodôntica com capacidade de minimizar o surgimento de lesões de mancha no esmalte dentário nas margens de acessórios colados para o tratamento ortodôntico. O silicato de nióbio produzido foi caracterizado utilizando a difração de raios X e a granulometria a laser. A resina adesiva ortodôntica foi desenvolvida utilizando Bis-GMA e TEGDMA com a incorporação de diferentes concentrações de SiNb (10, 20 e 30% em peso). Um grupo foi formulado sem a adição de SiNb (0%) e utilizado como controle. Esta resina foi avaliada quanto ao grau de conversão, amolecimento em solvente, citotoxicidade, resistência à flexão e resistência de união. A caracterização do silicato de nióbio mostrou moléculas de estrutura amorfa e partículas com tamanho médio de 11,43 μm e uma área de superfície de 509,204 m^2/g. A avaliação da resina demonstrou uma redução no grau de conversão dos grupos com maiores concentrações de carga de SiNb (20 e 30%). Essa redução também foi vista nos valores de resistência à flexão nos grupos experimentais quando comparados ao grupo controle ($p<0,05$). Nos testes de citotoxicidade, amolecimento em solvente e resistência ao cisalhamento a adição do SiNb como partícula de carga não influenciou o comportamento do material, não demonstrando diferenças estatísticas entre os grupos experimentais e grupo controle ($p>0,05$). Conclui-se então que a utilização do silicato de nióbio como carga bioativa em uma resina adesiva ortodôntica pode ser uma opção interessante para o uso na prática clínica pela sua baixa influência nas propriedades mecânicas do material e visando sua capacidade de deposição mineral para evitar o surgimento de lesões de mancha branca no esmalte dentário durante o tratamento ortodôntico.

Palavras-chave: Adesivo ortodôntico, Lesão de mancha branca, Nióbio.

ABSTRACT

PINTO, L. **INFLUÊNCIA DA ADIÇÃO DE SILICATO DE NIÓBIO NAS CARACTERÍSTICAS FISICO-QUÍMICAS DE UM ADESIVO ORTODÔNTICO EXPERIMENTAL.** Dissertação – Faculdade de Odontologia da Universidade Federal do Rio Grande do Sul, Porto Alegre, 2021.

The objective of this dissertation was to synthesize and characterize niobium silicate (SiNb) using the sol-gel method using Niobium pentachloride (NbCl5) and tetraethylortosilicate as a precursor to silica to be used as a bioactive charge particle in an orthodontic adhesive resin with the ability to minimize the appearance of white spot lesions on dental enamel on the margins of glued accessories for orthodontic treatment. Niobium silicate was characterized using X-ray diffraction and laser diffraction. The orthodontic adhesive resin was developed using Bis-GMA and TEGDMA with the incorporation of different SiNb concentration (10, 20 e 30% by weight). A group was formulated without the addition of SiNb (0%) and used as control. This resin was evaluated for degree of conversion, softening in solvent, cytotoxicity, flexural strength and shear bond strength. The characterization of niobium silicate showed amorphous structure molecules and an average particle size of 11.43 μm and a surface área of 509.204 m^2/g. The evaluation of the resin showed a reduction in the degree of conversion of the groups with higher concentrations of SiNb load (20 and 30%). This reduction was also seen in the flexural strength values in the experimental groups when compared to the control group ($p<0,05$). In cytotoxicity, softening in solvent and shear bond strength tests, the addition of SiNb as a particle of charge did not influence the behavior of the material, showing no statistical differences between the experimental groups and the control group($p>0,05$). It is concluded that the use of niobium silicate as a bioactive filler in an orthodontic adhesive resin can be an interesting option for use in clinical practice due to its low influence on the mechanical properties of the material and aiming at its mineral deposition capacity, to prevent the appearance of white spot lesions on tooth enamel during orthodontic treatment.

Key-words: Orthodontic adhesive, White spot lesion, Niobium.

SUMÁRIO

INTRODUÇÃO

Durante o tratamento ortodôntico, um efeito colateral não desejado é o surgimento de lesões de mancha branca (WSL). As lesões de mancha branca são descritas como o primeiro sinal de lesão de cárie que pode ser detectada a olho nu (JULIEN, BUSCHANG, CAMPBELL, 2013; FEJERSKOV, NYVAD, KIDD, 2003). Os bráquetes, bandas, fios e outros acessórios ortodônticos interferem negativamente nos procedimentos de higiene bucal, causando acúmulo de placa bacteriana, o que agrava o risco de desmineralização no esmalte (CHAPMAN et al. 2010). As lesões de mancha branca podem se desenvolver quatro semanas após a colagem dos acessórios ortodônticos (BAESHEN, LINGSTROM, BIRKHED, 2011). A prevalência relatada varia consideravelmente dependendo do método/critério de medição empregados, estudos demonstram que cerca de 37% dos pacientes tratados tinham pelo menos uma lesão de mancha branca desenvolvida após tratamento ortodôntico (WILLMOT, BROOK, 1999), enquanto 24% dos dentes tratados desenvolveram pelo menos uma lesão de mancha branca (MIZRAHI, 1982), sendo os dentes mais afetados os primeiros molares superiores e inferiores, incisivos laterais superiores, incisivos laterais inferiores e caninos inferiores (MIZRAHI, 1983) . A desmineralização do esmalte não viola apenas o princípio estético do tratamento ortodôntico, mas também prejudica a saúde dos dentes.

Com o intuito de prevenir essa desmineralização, agentes antimicrobianos adicionados a adesivos ortodônticos foram testados, como os monômeros antimicrobianos MDPB (UYSAL et al. 2011), DMADDM (MELO et al. 2014) e TAT (ALTMANN et al. 2014), além de clorexidina (BISHARA et al. 1996), nanopartículas de prata (BLOCHER et al. 2015), triclosan (MALKOC et al. 2005), oxido de zinco (SPENCER et al. 2009) e dióxido de titânio (POOSTI et al. 2012). Apesar de alguns autores terem observado uma redução nos valores de resistência de união em adesivos com agentes antimicrobianos (ALTMANN et al. 2015), uma meta regressão recente demonstrou que a adição desses agentes não influencia significativamente na resistência de união do material com o esmalte dentário. Considerando isso, o desenvolvimento de um adesivo ortodôntico com adição de agentes que possam induzir deposição mineral no

esmalte dentário pode representar uma alternativa eficiente para a prevenção do surgimento de lesões de mancha branca.

O desenvolvimento de partículas bioativas com alto teor de sílica pode ser uma alternativa para garantir a interação adequada entre carga inorgânica e a matriz polimérica, mantendo o equilíbrio entre propriedades mecânicas e biológicas dos compósitos odontológicos. O nióbio tem sido estudado na área biomédica devido sua capacidade de potencializar a deposição mineral, objetivando a remineralização dos tecidos duros (COLLARES et al. 2014; ALTMANN et al. 2017; BALBINOT et al. 2018; BALBINOT et al. 2019; OBATA et al. 2012; PRADHAN et al. 2016). A adição de nióbio a biomateriais tem demonstrado sucesso na redução da citotoxicidade (DSOUKI et al. 2014) e aumento na atividade da fosfatase alcalina (OBATA et al. 2012), induzindo a deposição mineral na superfície do esmalte dentário, além de não influenciar significativamente nas propriedades mecânicas dos materiais. Esses resultados estão relacionados a uma ligação estável entre Si-O-Nb encontrados nessas partículas, com a presença de sílica disponível para silanização e do nióbio com a capacidade de promover deposição mineral (ALTMANN et al 2017; COLLARES et al. 2014; ALTMANN et al. 2017; OBATA et al. 2012; CARNEIRO et al. 2018) .

Pensando na capacidade bioativa da utilização do nióbio em compósitos odontológicos, o objetivo deste estudo foi de desenvolver e testar as propriedades de uma resina adesiva ortodôntica utilizando o silicato de nióbio (SiNb) como partícula de carga inorgânica com a intenção de conferir ao material a capacidade de induzir deposição mineral no esmalte dentário, reduzindo assim o surgimento de lesões de mancha branca.

OBJETIVO

Com base no exposto, o objetivo do presente estudo foi desenvolver uma resina adesiva ortodôntica experimental com silicato de nióbio e a posterior caracterização das propriedades do material.

ARTIGO

A presente dissertação de mestrado apresenta-se na forma de um manuscrito.

INFLUÊNCIA DA ADIÇÃO DE SILICATO DE NIÓBIO NAS CARACTERÍSTICAS FISICO-QUÍMICAS DE UM ADESIVO ORTODÔNTICO EXPERIMENTAL

INTRODUCTION

Tooth enamel demineralization around orthodontic brackets is a common issue in clinical practice. The prevalence of these caries lesions, known as white spots, in patients during orthodontic treatments varies from 25% to 46% (1, 2, 3). White spot lesions can be visually detected as opaque white lesions present in tooth enamel (4). These lesions usually occur in the cervical third of the crown, mainly in the first molars, lateral incisors, and canine teeth (5). White spot lesions usually develop after four weeks without biofilm removal (6).

The high prevalence of white spots on the enamel of orthodontic patients is partly attributed to the irregular surface of the brackets and the presence of orthodontic wires, elastic bands, orthodontic bands, and other accessories. These increase plaque retention, making oral hygiene difficult for patients and limiting the ability to clean teeth through salivary flow and the action of oral muscle movements. Consequently, the plaque pH is decreased due to the presence of fermentable carbohydrates, accumulation and maturation of that plaque, and colonization of aciduric bacteria such as *Streptococcus mutans* and *Lactobacilli* (7).

To prevent this demineralization in the most susceptible sites, orthodontic adhesives, sealants, and varnishes have been suggested as an adjuvant method (8). To reduce bacterial colonization, antimicrobial agents have been added to orthodontic adhesives such as antimicrobial monomers (methacryloyloxy dodecylpyridinium bromide; MDPB) (9), dimethylamino dodecyl methacrylate

(DMADDM) (10), and 1,3,5-tryacryloylhexahydro-1,3,5-triazine (TAT) (11), in addition to chlorhexidine (12), silver nanoparticles (13), triclosan (14), zinc oxide (15), and titanium dioxide (16). Although some authors have observed a reduction in the bond strength of adhesives with antimicrobial agents (14), a recent meta-regression analysis demonstrated that the addition of these agents did not influence the bond strength to tooth enamel (17). The development of an orthodontic adhesive with the ability to reduce bacterial growth and provide more favorable conditions to mineral deposition could be an effective approach to challenge the appearance of white spot lesions.

Niobium appears to be a promising material for the development of this material. Niobium-containing glasses have drawn great interest for dental use due to their structural versatility and several possible useful applications. When used as a component of bioactive glasses, they have shown the ability to obtain higher biocompatibility, lower hydrolytic degradation (11), and reduced cytotoxicity (18). The presence of niobium in bioactive glasses for developing materials for dental use can also stabilize pH (19), increase microhardness (20), and stimulate mineral deposition (11, 19, 21). The chemical resistance of silicates can also be improved with the addition of niobium, which exerts favorable effects on their chemical and physical properties (22).

In an attempt to develop a material with the necessary characteristics to prevent white spot lesions, this study aimed to evaluate the physical, chemical, and biological properties of an experimental orthodontic adhesive resin with the addition of niobium silicate (SiNb).

MATERIALS AND METHODS

SYNTHESIS OF NIOBIUM SILICATE (SiNb)

The SiNb particles were synthesized by the sol-gel method, as described elsewhere (23), with tetraethylorthosilicate (TEOS; Aldrich Chemical, St Louis, MO, USA) as the silica precursor and niobium pentachloride ($NbCl_5$; CBMM, Araxá, MG, Brazil) as the inorganic modifier. To produce the sol, hydrochloric acid (HCl, 1M; Aldrich Chemical, St Louis, MO, USA) was mixed with $NbCl_5$ for 30 minutes and then with TEOS for 1 hour. The sol was kept in a room at 18 °C for 7 days until the gel was formed. The gel was exposed to a heat treatment at 100 °C for 24 hours, then 500 °C for 24 hours. The particles were slowly cooled for 24 hours, ground, and dispersed in absolute ethanol to be sieved, controlling the size of the particles.

CHARACTERIZATION OF NIOBIUM SILICATE:

X-Ray Diffraction

X-ray diffraction, used to determine the atomic and molecular structure of SiNb, was performed on the SiNb powder by diffractometer (X'PertPRO, PANalytical MPD, Netherlands), using CuKα radiation at 40kV-40mA with angles between 5 °C and 100 °C and a step size of 0.02 for 2 seconds.

Surface Area and Particle Size (Laser Diffraction)

The SiNb powder surface area was evaluated by the Brunauer-Emmett-Teller (BET) method, based on isothermal data of nitrogen adsorption. The nitrogen adsorption measures were obtained by a Quantachrome Nova 1200 (Instrument of Quantachrome Instruments Corporate Headquarters, Boynton Beach, USA). The particle size range was obtained by a laser diffraction particle size analyzer (CILAS 1180, Orleans, France).

FORMULATION OF EXPERIMENTAL RESINS

The experimental orthodontic adhesive resins were formulated with 75 wt% of bisphenol glycidyl methacrylate (Bis-GMA) and 25 wt% of triethylene glycol dimethacrylate (TEGDMA). Camphorquinone (CQ), ethyl 4-dimethylamino benzoate (EDAB), and diphenyliodine hexafluorophosphate (DPIHFP) were added as a photoinitiator system at 1 mol%, along with 0.01 wt% of butylated hydroxytoluene (BHT) as a polymerization inhibitor (Sigma-Aldrich, St. Louis, MO, USA). Additionally, 5 wt% of vaporized silica (AEROSIL 200-non silanated, Piscataway, NJ, USA) was added to adjust the viscosity. Different concentrations of silanized SiNb were used to formulate four groups of experimental resin: 0% (control), 10 wt%, 20 wt%, and 30 wt%.

DEGREE OF CONVERSION

The degree of conversion (DC) was measured by Fourier-transform infrared spectroscopy. A spectrometer (Vertex 70, Bruker Optics, Ettlingen,

Germany) was used, equipped with an attenuated total reflectance device (Platinum ATR-QL, Bruker Optics, Ettlingen, Germany) composed of a horizontal diamond crystal with a mirror angle of 45 °C. The experimental orthodontic adhesives were dispensed directly onto the diamond crystal, contained in a polyvinylsiloxane matrix for standardization (5 mm in diameter and 1 mm in height). A spectrum was obtained before, and another immediately after photoactivation for 20 seconds, with a Valo Cordless (Ultradent Products Inc, South Jordan, UT, USA), with a standardized distance of 5 mm. The DC was then calculated based on the absorbance values for 1,610 cm (bond C=C in the aromatic ring) and 1,640 cm^{-1} (bond C=C in the aliphatic chain), as internal standard (24).

SOFTENING IN SOLVENT

For the solvent softening test, five samples from each group (n=5), made with a polyvinyl siloxane matrix with a 6 mm diameter and 1mm thickness and photoactivated for 20 seconds on both sides with a Valo Cordless (Ultradent Products Inc, South Jordan, UT, USA), were embedded in acrylic resin (VIPIFlash, Vipi Industry, Pirassununga, SP, Brazil). The samples were polished with silicon carbide sandpaper with granulations of 600, 1200, and 2000 and felt discs saturated with alumina suspension (alumina, 0.5 µm; Arotec, Cotia, SP, Brazil), then washed with distilled water in an ultrasound washer (Cristófoli, Campo Mourão, PR, Brazil). Three indentations were made (at 10 grams for 5 seconds) in each specimen (HMV 2, Shimadzu, Tokyo, Japan) to obtain the initial Knoop hardness value (KNH1). The samples were immersed in a solution of 50%

water and 50% ethanol (Labsynth, Diadema, SP, Brazil) for 2 hours and washed with distilled water. After the immersion period, another three indentations were made to determine the final Knoop hardness values (KNH2). The percentage of Knoop hardness variation (ΔKNH%) was calculated for each sample (25).

FLEXURAL STRENGTH

The flexural strength of the developed experimental orthodontic adhesives was tested according to the ISO 4049 standard. Five specimens were used per group (n=5). The experimental and control orthodontic adhesives were inserted in a metallic matrix (12 mm x 2 mm x 2 mm) with polyester matrices on both sides. Photoactivation was performed for 20 seconds (Valo Cordless, Ultradent Products Inc, South Jordan, UT, USA) in two windows at the top and bottom of the samples. The samples were then stored in water for 24 hours at 37 °C and subjected to a three-point flexural strength test using a universal testing machine (Shimadzu EZ-SX, Shimadzu Corp., Kyoto, Japan) with a crosshead speed of 0.5 mm/min. Flexural strength was calculated in MPa according to the following formula:

$$\sigma=\frac{3Fl}{2bh2}$$

where F is the maximum load, l is the distance between the supports, b is the width of the sample, and h is the height of the sample.

CYTOTOXICITY

A culture of gingival fibroblasts was used to perform the cytotoxicity test. The cells were obtained from a healthy donor aged over 18 years old, who may be female or male. The cells were grown in Dulbecco's modified Eagle medium (DMEM), supplemented with 10% fetal bovine serum and 1% penicillin at 37 °C with 5% CO_2. The eluates were prepared by immersing samples (3 mm in diameter x 1 mm in thickness) from each experimental group (n=3) in 1 mL of DMEM for 24 hours. The cells were then placed in 96-well plates at a concentration of 5 x 10^3 and treated with 100 µL of an eluate. After 72 hours, the cells were fixed with 10% trichloroacetic acid (TCA), incubated at 4 °C for 1 hour, washed six times with running water, and dried at room temperature. Then, 4% sulforhodamine B (SRB; Sigma-Aldrich, St. Louis, MO, USA) was added to color the cells, and the plate was incubated for 30 minutes at room temperature. The plates were then washed four times with 1% acetic acid to remove the excess unbound dye and dried at room temperature. Trizma solution was used to resuspend the cells, and the microplates were measured at 570 nm (Multiskan EX Microplate Reader, MTX Lab Systems, Vienna, Austria).

SHEAR BOND STRENGTH

Clean bovine teeth, free of fractures and stored in distilled water at 4 °C for no more than 3 months, were used for the shear bond strength test (n=12). The roots were incorporated in acrylic resin in metal molds (20 mm in diameter and 15 mm in height) with the vestibular face perpendicular to the acrylic base. The vestibular surface was conditioned with 37% phosphoric acid (Atacktec,

CaiTech Industry LTDA, Rio do Sul, SC, Brazil) for 30 seconds, rinsed with water for 30 seconds, and dried with air spray for 30 seconds. TransbondXTPrimer (3MUnitek, Monrovia, CA, USA) was applied to the bonding surface and photopolymerized for 20 seconds using a Valo Cordless (Ultradent Products Inc, South Jordan, UT, USA). Metal brackets of upper central incisors (Roth Max, Morelli, Sorocaba, SP, Brazil), with an area of 11.65 mm^2, were used (control and experimental). The teeth were randomly divided into four groups, and the orthodontic adhesives were applied to the base of the brackets and placed in the center of the vestibular surface of the tooth. To standardize the thickness of the adhesive, the brackets were subjected to 300 gF, and the excess adhesive was removed. The adhesives were then light-cured for 40 seconds (10 seconds for each side of the support) and stored in water for 7 days. The specimens were subjected to shear bond strength testing in a universal testing machine (Shimadzu EZ Test EZ-SX, Kyoto, Japan) using a knife wire chisel (0.1 mm) at 180° to the vestibular surface of the tooth, supporting it on the adhesive-enamel interface, with a crosshead speed of 1 mm/min.

STATISTICAL ANALYSIS

The normality of the data was tested using the Shapiro–Wilk test. The softening in solvent test KNH1 and KNH2 data were compared using a paired t-test. One-way analysis of variance was applied to analyze the degree of conversion, cytotoxicity, antimicrobial activity, and ΔKNH% in the softening in solvent and shear bond test data. All analyses were performed considering a significance level of $p<0.05$.

RESULTS

The X-ray diffraction test results showed an amorphous molecular structure in SiNb powder (Figure 1). The laser diffraction test results showed a BET surface area of 509.204 m²/g and an average particle size of 11.43 µm with a diameter at 10% of 3.58 µm, 9.63 µm at 50%, and 21.92 µm at 90%.

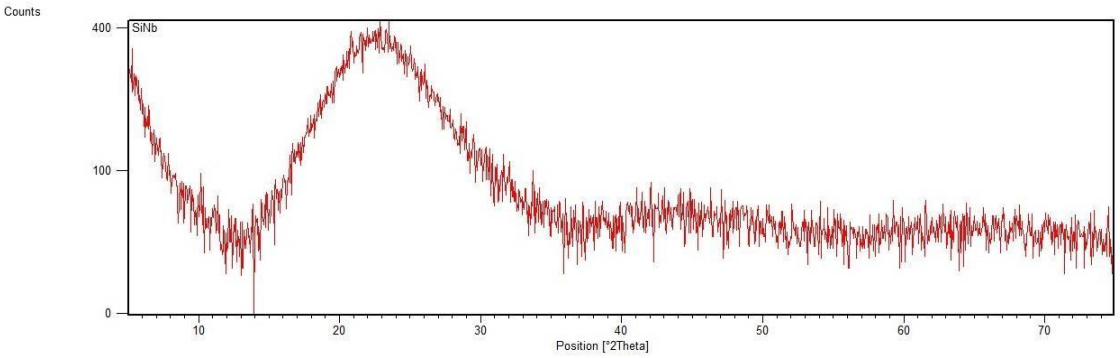

Figure 1: X-Ray difraction graph

The DC testing results are presented in Table 1. A reduction in the DC of experimental orthodontic adhesives with SiNb filler was observed compared to the control group; the groups with 20% and 30% had the lowest DC. In cytotoxicity testing, the 10% SiNb group presented the highest cell viability values (88.99%), with statistically significant differences compared to the 20% and 30% groups ($p<0.05$). However, all groups with SiNb showed no statistically significant difference to the control group ($p>0.05$).

Group	*DC (%)*	*Cell Viability (%)*
G_{CTRL}	53.02 (±0.89) ^{A}	76.60 (±8.01) ^{AB}
$G_{10\%}$	51.46 (±0.57) ^{B}	88.99 (±16.23) ^{A}
$G_{20\%}$	48.60 (±0.56) ^{C}	60.88 (±11.34) ^{B}
$G_{30\%}$	47.83 (±0.37) ^{C}	66.75 (±10.66) ^{B}

Table 1 – Results of Degree of Conversion and Citotoxicity test.

In the softening in solvent test (Table 2), the experimental groups showed no statistically significant difference compared to the control group ($p>0.05$). However, all groups showed a significant reduction in Knoop hardness values when tested after immersion for 2 hours in ethanol. The 20% and 30% SiNb groups presented the highest values of Knoop hardness decrease after ethanol immersion ($p<0.05$).

No statistically significant differences existed between groups in shear bond strength ($p>0.05$). In the flexural strength test results, no statistically significant difference existed between the experimental groups with SiNb ($p>0.05$). However, they showed significant differences when compared to the control group, which obtained much higher results (Table 3).

Group	***KHN1***	***KHN2***	***∆KHN%***
G_{Ctrl}	29.15 (±1.49) ^{ABa}	23.84 (±1.58) ^{Ab}	17.93 (±2.04) ^{A}
$G_{10\%}$	27.82 (±0.73) ^{Ba}	21.91 (±2.01) ^{Ab}	19.45 (±7.52) ^{A}
$G_{20\%}$	31.41 (±2.62) ^{Aa}	22.73 (±1.13) ^{Ab}	27.04 (±8.44) ^{B}
$G_{30\%}$	31.61 (±1.74) ^{Aa}	23.56 (±2.04) ^{Ab}	25.22 (±10.21) ^{B}

Table 2 – Results of Solftening in solvent test *(Different capital letters indicate statistically significant difference in the same column and different lowercase letters indicate statistically significant difference in the same row)*

Group	***SBS (MPa)***	***Flexural Stregth(MPa)***
G_{CTRL}	17.37 (±3.20) ^{A}	70.63 (±16.29) ^{A}
$G_{10\%}$	16.41 (±2.22) ^{A}	48.72 (±7.36) ^{B}
$G_{20\%}$	18.56 (±2.30) ^{A}	47.06 (±2.84) ^{B}
$G_{30\%}$	18.66 (±3.48) ^{A}	47.55 (±6.15) ^{B}

Table 3 – Results of Shear Bond Strength and Flexural Strength.

DISCUSSION

The prevalence of white spot lesions increases significantly during orthodontic treatment (1). In an attempt to reduce the occurrence of this type of lesion around brackets and other accessories, this study aimed to develop an orthodontic adhesive resin with bioactive properties to induce remineralization in sites demineralized by the presence of biofilm. Adhesive materials with improved properties such as remineralization potential have been widely evaluated in studies of polymers used in dentistry (26, 27, 28, 29, 30). In this study, SiNb was synthesized aiming at this potential as a bioactive filler in dental composites, improving biological properties and not compromising physical-chemical properties. SiNb particles developed using the sol-gel method were incorporated into an orthodontic adhesive resin formulated with a combination of Bis-GMA and TEGDMA at concentrations of 10%, 20%, and 30%.

Although bioactive properties are desired in dental materials, the addition of bioactive fillers may impair the formation of polymeric matrices (24), influencing the degree of conversion of the material. The groups with higher concentrations of SiNb (20 wt% and 30 wt%) showed a slight decrease in DC values. This can be explained by the dispersion of the light emitted by the photopolymerizer due to the greater presence of SiNb particles, impairing the transmission of light through the material and reducing the light availability in the polymer, thus reducing its degree of conversion (31). Inadequate polymerization is directly related to material degradation and cytotoxic effects due to leaching of non-activated monomers during adhesive application, which can represent a reduction in cytotoxicity values in the groups with higher concentrations of SiNb.

However, no group showed statistically significant differences compared to the control group.

The addition of different bioactive particles to polymeric materials has been studied, and some impact on the mechanical properties of the materials has been reported (26, 30, 32). In the flexural strength test, the groups with SiNb showed statistically significant differences compared to the control group ($p<0.05$). The values obtained were lower than those recommended by ISO 4049: 2009, which corresponds to some previous results in the literature that the addition of inorganic fillers in higher concentrations can result in increased mechanical failure between polymer matrix and inorganic filler due to little interaction between them (33). However, this should not pose a considerable problem for clinical application of the material, since it would be used for bonding orthodontic brackets fixed on enamel and used for a reduced time, only during the treatment period, with subsequent removal.

Although the addition of inorganic filler to polymeric matrix negatively influences the degree of conversion and flexural strength, it also increases the hardness of the material. The solvent softening test results showed a slightly higher initial Knoop hardness in the groups with higher concentrations of inorganic filler, 20 wt% and 30 wt%, than in the control and 10 wt% groups. However, after the storage period in ethanol, the groups presented no statistically significant differences in hardness values, causing higher variations in the Knoop hardness in the groups with 20 wt% and 30 wt%, which can be explained by greater degradation of crosslinks due to inadequate polymerization. In the shear bond strength test, the material was tested in a situation simulating clinical reality. The material was used to bond central incisor brackets on the buccal surface of

bovine incisors. The addition of SiNb did not change the results obtained in the experimental groups when compared to the control group. Although no consensus exists that values obtained in vitro serve to predict the clinical success of the material (34, 35) due to the variation of the test parameters (17), the higher shear bond strength values demonstrated would probably lead to greater retention of brackets in the dental enamel during orthodontic treatment (36, 37).

CONCLUSION

Although more studies are needed, the use of SiNb as a bioactive filler could be a viable alternative to orthodontic adhesive resin development.

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CONSIDERAÇÕES FINAIS

O surgimento de lesões de mancha branca no esmalte dentário ao redor de acessórios colados durante o tratamento ortodôntico é um problema muito comum na prática clínica (JULIEN, BUSCHANG, CAMPBELL, 2013). Na tentativa de solucionar esse problema, vários agentes com capacidade antimicrobiana e remineralizante foram empregados em resinas adesivas ortodônticas e tiveram suas propriedades avaliadas (ALTMANN et al. 2015).

O nióbio (Nb) é um material que tem se apresentado como promissor para ser utilizado como partícula de carga devido ao seu potencial de induzir formação mineral (COLLARES et al. 2014; ALTMANN et al. 2017). Neste estudo o nióbio foi utilizado para a sintetização de um silicato (SiNb) (BALBINOT et al. 2020) com o intuito de ser adicionado como partícula de carga em uma resina adesiva ortodôntica experimental.

Foi formulada uma resina experimental utilizando uma base de Bis-GMA e TEGDMA e esta resina teve suas propriedades avaliadas em diferentes concentrações (10,20 e 30%). A adição de partículas bioativas à materiais poliméricos tem sido estudada e tem demonstrado influenciar negativamente nas propriedades mecânicas dos materiais (SAURO et al. 2012; DUMINIS et al. 2018; BALBINOT et al. 2019). A adição do SiNb como partícula de carga influenciou negativamente no grau de conversão do polímero e na resistência a flexão, entretanto, não teve influência significativa no comportamento do material quando avaliado nos testes de amolecimento em solvente, citotoxicidade e resistência ao cisalhamento.

Pensando na utilização clínica do material para colagem de acessórios ortodônticos em esmalte dentário, os resultados apresentados pelo material nos

testes nos permite considerar o silicato de nióbio como uma alternativa viável para ser utilizado no desenvolvimento de resinas adesivas ortodônticas com propriedades de indução de deposição mineral no esmalte dentário, apesar de serem necessários mais estudos para avaliar a capacidade de induzir deposição mineral e melhor compreender os efeitos da adição do SiNb nas propriedades dos materiais. Os próximos estudos a serem realizados com esse material compreendem uma avaliação da capacidade de induzir deposição mineral e uma avaliação da viscosidade desse material com adições de diferentes concentrações de SiNb. Posteriormente, realizar um estudo comparativo utilizando um material comercial (Transbond XT) para comparar com o material experimental com a concentração que apresentou os melhores resultados. Por fim realizar um estudo in situ, onde o material seria testado em meio bucal para comprovar os resultados encontrados nos testes laboratoriais.

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