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Synthesis of a 45S5 bioactive glass cementitious resin bonded composite material for orthodontic applications

Síntesis de un material compuesto de resina cementante con vidrio bioactivo 45S5 para aplicaciones de ortodoncia

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Resumen

Se obtuvo un material compuesto base vidrio bioactivo 45S5 y resina comercial cementante para aplicaciones de ortodoncia, tomando como control dos resinas comerciales. La síntesis del vidrio bioactivo se realizó mediante sol-gel, estabilizando el gel seco por medio de la asistencia de microondas durante 6 minutos, obteniendo un material amorfo con buena estabilidad dimensional y transparente. Se preparó un material compuesto por resina cementante Transbond y 8% de vidrio bioactivo 45S5. El material se sometió a pruebas de degradación para determinar el efecto de la saliva artificial en la degradación del material durante un mes. La degradación de las muestras se analizó mediante SEM/EDS y variaciones de pH del fluido. La bioactividad de los compuestos se llevó a cabo exponiendo el material a fluido fisiológico simulado (SBF), encontrando la formación de capas de apatita biológica en la superficie de los materiales. Las propiedades mecánicas del material se analizaron uniendo los brackets con el material compuesto a las piezas dentales de humanos previamente preservadas y se midió la resistencia al corte y el esfuerzo de tensión de la interfaz diente-resina. Los análisis se realizaron por triplicado. Los datos de la prueba mecánica se analizaron mediante el análisis estadístico ANOVA. Encontrando que el material compuesto sintetizado exhibe mejores propiedades de bioactividad, mecánicas y de degradación en comparación con las resinas comerciales.

Palabras clave: Material compuesto, Vidrio bioactivo, Resina cementante, Ortodoncia.

Abstract

A composite material based on 45S5 bioactive glass and commercial cementitious resin for orthodontic applications, was obtained, taking two commercial resins as control. The bioactive glass was synthesized using sol-gel and stabilizing the dry gel using microwave assistance for 6 minutes, obtaining an amorphous material with good dimensional stability and transparency. A composite material was prepared using Transbond cementitious resin and 8% 45S5 bioactive glass. The resulting composite was subjected to degradation tests to determine the effect of artificial saliva on this material degradation over a period of one month. Sample composite degradation and the fluid pH variations were analyzed via SEM/EDS. Composite bioactivity was determined by exposing the material to simulated physiological fluid (SBF), showing the formation of biological apatite layers on the materials' surface. The composite´s mechanical properties were analyzed by adhering the brackets with the composite material to previously preserved human dental pieces and the shear strength of the composite was determined along with the tension strength between the resin teeth interface. Analyzes were performed by triplicate. A Statistical ANOVA analysis was performed to determine significant mechanical test results. These data showed that the synthesized composite material exhibited better bioactivity, mechanical and degradational properties compared to the commercial resins.

Keywords: Composite material, Bioactive glass, Cementitious resin, Orthodontics.

Introduction

In medicine, odontology uses materials to repair or replace dental pieces. A branch of odontology is orthodontics, which deals with teeth alignment using metal brackets that are fixed with adhesives to ensure correct treatment.

Even considering the advances in the materials used in orthodontics, these still show a significant disadvantage: the surface of the teeth have to be abrased to apply orthodontics adhesives to fix the brackets in place, which has been shown to develop caries or white or brown stains (Al et al., 2011; Seyhan-Cezairli et al., 2019) which are caused by organic acids generated by bacteria during treatment (Gao et al; 2023). According to a 2011 study by Al, a sample of 230 patients was taken to verify the incidence of caries and stains. This study found that approximately 70% s had developed these problems. Factors such as treatment duration, oral hygiene, overall health, and even the patient's gender would be contributing factors to these problems (Al et al., 2011). Another reported problem in the literature is the demineralization of dental enamel (Gao et al;

2023), mainly in the bracket area. This problem appears in the abraded surfaces created to apply the bracket adhesive, which can foster the growth of oral microorganisms such as Streptococcus mutans (SM) (Seyhan-Cezairli et al., 2019).

Currently, resin-based compounds, such as methylmethacrylate and polymethylmethacrylate polymer resins, are used as adhesives and restoring materials (Zafar, 2020). These resins are classified as acrylic and compound, showing desirable advantages such as stronger adhesion and better aesthetic presentation (Vallittu et al; 2015). However, they also have some disadvantages, such as polymerization contraction, stress at the tooth-restoration interface, and potential damage to the skin or eyes as they are light-cured by UV light. The resins contribute nothing to stop bacterial growth.

Degrazia and Leitune found an alternative in 2016 (Degrazia et al., 2016); they modified the existing adhesives by adding external agents to improve their properties and reduce their deficiencies. They proposed using silver nanoparticles (AgNP) in concentrations of 0%, 0.11%, 0.18%, and 0.33%, dissolved in distilled water to improve particle distribution in Transbond XT Primer, a commercial adhesive. Their results show a decrease in SM growth in the case of 0.11% and 0.18% concentration. Also, the study found that the shear stress is affected in the case of 0.33% concentration. In addition, using silver nanoparticles reduces the resin's toxicity compared with other agents such as gold and zinc. Current efforts are dedicated to developing materials with better mechanical, adhesive, and antibacterial properties. One such proposal is the use of biomaterials such as glass ionomer cements (GIC) (5) and bioactive glasses (Kim et al., 2021).

Glass ionomer cements (GIC) are made from calcium fluoraluminumsilicate powder and a liquid mixture of 50% polyacrylic acid and 50% water (Vallittu et al; 2015). This material shows three advantages: biocompatibility, anticarcinogenic action, and a specific adhesion to the enamel and dentine. These advantages are obtained by conditioning the dental tissue with 10% polyacrylic acid. On the other hand, the widely commercialized bioactive glass 45S5 is composed of 46.1 mol% of SiO_2, 24.4 mol% Na_2O, 26.9 mol% of CaO, and 2.6 mol% of P_2O_5 (Manafi et al., 2019). Given the fluoride and calcium ion liberation generated by prolonged contact with bodily fluids, this bioactive glass hosts better bioactive properties than GIC. Bioactive glass also shows better mechanical properties, such as a improved adhesion in the resin-tooth interface and antibacterial properties (Pirayesh, 2010).

For the reasons above, this research centers on preparing and characterizing orthodontic adhesives containing 45S5 bioactive glass in their composition.

Materials and methods

The synthesis of the bioactive glass was prepared following the method reported by Pirayesh (Kokubo and Takadama, 2006) by preparing a solution of nitric acid and water. Then, 33.5 mL of tetraorthosilicate was added. The reaction in the solution occurred for one hour under agitation to favor hydrolysis. After agitation, the following reagents were added in order: 2.9 ml of tetraethyl phosphate, 20.13 gr calcium nitrate, 4 mL water, and 13.52 g sodium nitrate. Between each reagent, intervals of 45 minutes under agitation elapsed until a transparent sol was obtained. The gelation process took five days, during which the solution was placed in a closed container at ambient temperature. Once the gel was obtained, it was aged in a closed container for one day at 70 °C. The aged gel was then dried at 120 °C for one day. Finally, to stabilize the glass, a microwave oven was used, in which the glass was exposed to microwave radiation for 6 minutes to eliminate residual and saline nitrates and other undesirable compounds. Once the 45S5 bioactive glass was obtained, it was crushed in a mortar.

Samples of size 3 x 3 x 2 mm were prepared for the TransbondTM XT and ACTIVA BioACTIVE-BASE/LINER brand resins, which were the control resins marked as Transbond and ACTIVA, respectively. A compound was prepared with the TransbondTM XT resin by mixing it with the 8% in weight of the 45S5 bioactive glass powder. This compound was marked as Transbond45. The samples were photocured using an RTA MINIS halogen light lamp for 5 seconds to initiate the polymerization process and then for 20 seconds to complete the light curing process. Then, the samples were weighed, and as well as the Petri dishes and 2 mL tubes (used as containers), were UV sterilized.

VIARDENER artificial saliva was used in the degradation test. The samples were weighed and placed in test tubes containing 5 ml of the artificial saliva. The tubes were placed in an ECOSHEL incubator at 37 °C for four weeks.

In each of the four weeks, three samples of each resin were taken, washed with distilled water, dried~~,~~ and weighed. Images of the samples were taken with a HITASHI scanning electron microscope, and the pH of the saliva was registered with a HANNA HI4221 potentiometer. The artificial saliva was replaced in the samples each week.

The sample preparation for the tensile and shear resistance tests was performed with ASTM D412. First, test tubes were prepared using COMEX epoxy on which the teeth were placed to be subjected to the universal machine loads. For the tensile resistance at the resin-tooth interface measurements, three test tubes were given a rectangular form, each with cavities in which three teeth were placed in horizontal positions and covered with the epoxy but leaving an exposed and accessible area in which the bracket was placed, as shown in Fig. 1A1. The test tubes used in the shear resistance test were also made of the epoxy and with a small perforation in which the teeth were placed in a vertical position. The teeth were covered up to the root by the epoxy to fix them in the test tubes, as shown in Fig. 1B1.

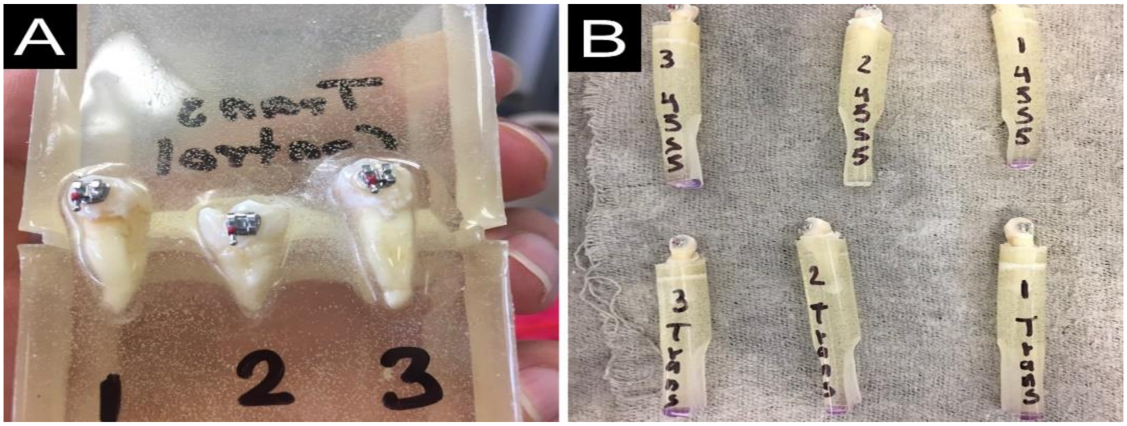

Figure 1. Teeth prepared for tensile test (A) and teeth prepared for shear strength test (B).

The stainless-steel brackets were adhered with the Transbond and ACTIVA cementing resins and were labeled as controls 1 and 2. The Transbond45 resin was identified as Experiment 1. All test tubes were stored for 24 hours before the tests were initiated. The shear resistance tests were performed with a Sintech 20/D universal machine at an approach speed of 0.5 mm/min at a 500 N pressure, following ISO 29022:2013.

The tensile resistance measurement was made by applying a tensile force using a 12 stainless steel orthodontics cable tied to the bracket flaps. The cable was pulled upwards at 90° angle with respect to the tooth-resin interface, while the epoxy test tubes were maintained in a horizontal position. An approach of 0.5 mm/min was used.

The simulated body fluid (SBF) used in the bioactivity tests was recreated following the method proposed by Kokubo and Takadama (Kokubo and Takadama, 2006). Samples of 2 x 2x 3 mm were three times exposed to the SBF solution for 3, 7, 14, and 21 days and placed in the ECOSHEL incubator at 37 °C. Three samples were taken and observed by SEM/EDS each week with a HITASHI SU5000. Infrared spectra were also obtained. The chemical analysis was performed on a Nicolet 6700 spectrophotometer in the 500-3000 cm^{-1} region, with 16 cm^{-1} resolution.

Results and discussion

Figure 2 shows SEM images and their respective EDS for each resin and compound material subjected to degradation tests in artificial saliva for 28 days. Figures 2A1 and 2C1 show the morphology of the Transbond resin before and after its exposure to the artificial saliva. These images show particle sizes between 2.3 and 2.3 µm, which can be attributed to silicon before the exposure. Figures 2A2 and 2C2 show the images of the ACTIVA resin, with particle sizes between 1.5 and 2 µm. In both cases, the size of the crystals is maintained. These results suggest a slow resin degradation. On the other hand, the particle size of the Trabsbond45 compound material shows an average particle size of 1.64 µm during the period investigated. However, the arrangement and distribution (compared to the resins) are due to the mechanical mixing of the bioactive glass, diminishing the size of the larger crystals.

The EDS spectra of the Transbond resin (2B1) show the presence of Si, which is the main component of the resin, and also of C and O, which can be attributed to polymers such as polyethylene glycol dimethacrylate, bisphenol A diglycidyl ether dimethacrylate, and bisphenol A dimethacrylate, which are used as adhesives in their composition. The EDS images of the ACTIVA resin show the presence of Si, Ba, P, and Ca, which are attributed to its composition: a mixture of diurethane and methacrylates with modified polyacrylic acid which contains amorphous silica and sodium fluoride (Vouzara et al; 2020; Pulpdent, 2019). The EDS of the compound material (2B3) shows the components of the Transbond resin along with the increment of the presence of O and C, attributed to the bioactive glass.

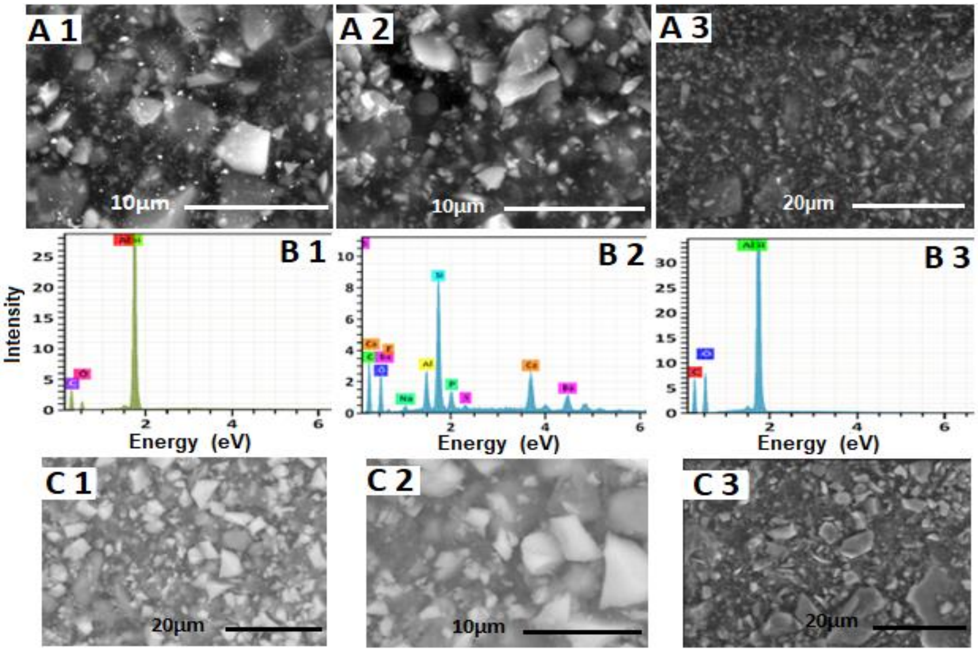

Figure 2. SEM and EDS of the compound material a 0 (A) and 28 (C) days after exposure to artificial saliva. A1 and C1 are the SEM images, and B2 is the EDS image of the Transbond resin. A2 and C2 are the SEM images, and B2 is the EDS image of the ACTIVA resin. Finally, A3 and C3 are the SEM images, and B3 is the EDS image of the compound material Transbond45.

The percentages of weight loss for different periods are shown in Fig. 3A. The Transbond resin retains its initial weight for the first two weeks and loses between 2 and 4 % of its total weight. The ACTIVA resin lost about 5% of its initial weight during the first week, which was maintained for the rest of the study, making it the one that lost more weight. In contrast, the Transbond45 resin kept its initial weight and average particle size of 1.64 µm, which suggests that this material is the most stable and that adding bioactive glass to the resins impacts on the degradation of the material.

Figure 3B shows pH variations in the artificial saliva where the samples were placed during the degradation analysis. The initial pH value was 7.4, which increased to 7.9 after the first week. After that, the pH stabilized between 7.5 and 7.6 for the Transbond resin and the Transcbond45 compound. This pH behavior is typical for this type of resin, which tends to have a higher pH during the first days then settles down. The pH behavior results from the ion exchange between the material and the fluids (Bauer, 2019). Also, Fig. 2 shows that the Transbond45 sample with bioactive glass behaves similarly to the Transbond resin, keeping a stable pH. These results support the orthodontics application of the material, as studies have shown that in an acidic environment, low pH fosters caries development (Barrios et al; 2017), besides leading to rapid and gradual decomposition of the material during orthodontic treatment. One could think that a neutral pH is sufficient to prevent degradation, along with low salt content and adequate body temperature. However, material degradation can happen by many mechanisms in which by products of degradation can be liberated, altering the local pH and stimulating additional reactions, along with continuous or cyclic tension, which can produce abrasion or deformation of the adhesives (Silva et al; 2023).

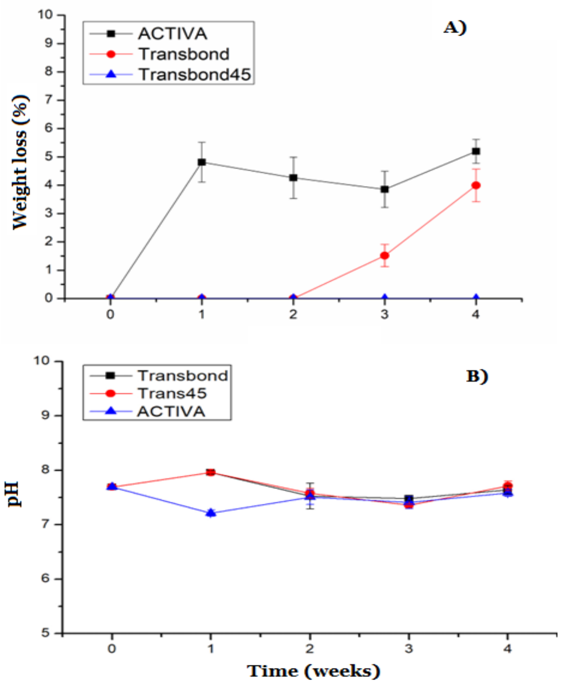

Figure 3. Weight loss and pH behavior of Transbond, Transbond45, and ACTIVA cementing resin solutions.

Figure 4 shows the results of the tensile and shear resistance tests before the brackets were detached from the teeth. The results of these mechanical properties are essential, given that the adhesion of the brackets to the surface of the dental piece is a basic procedure during orthodontic treatment. The adhesion should fix the orthodontic anchors in place, preventing undesirable effects that could harm the enamel surface (Nawrocka et al; 2023).

Figure 4A shows that the compound material Transbond45 had greater resistance to tension than the Transbond and ACTIVA resins. In the cross-section tension test, the Transbond45 resin withstood a maximum load of more than 6.1 MPa before rupture. Also, the shear resistance test (Fig. 4B) aimed to reproduce various dynamics of mandibular mechanics. The test found no significant differences (between 29 and 42 MPa) in the compound resin Transbond45, Transbond, and ACTIVA. Furthermore, all resins show adequate adherence as required in orthodontic procedures (Seyhan-Cezairli et al., 2019).

Figure 4C shows the Young´s modulus or elastic modulus of the materials. A significant difference can be observed in the resistance to deformation in the compound material (45 MPa) and the other resins (11 and 27 MPa). The results obtained for the shear modulus (Figure 4D) show no significant difference in the shear resistance of the adhesives and the compound material, as the materials required the same load to break the bond between the dental piece and the brackets by applying antiparallel forces. These results indicate that adding bioactive glass to the Transbond resin modified its Young modulus, having a tensile resistance of approximately 6 MPa. According to Ariztizabal (Skallevold et al; 2019), adhesives that exceed 9.7 MPa would damage the enamel, and at 14 MPa or more, the enamel can fractured or detached. These results agree with other research; using filler particles for a resin improves mechanical properties (Seyhan-Cezairli et al., 2019; Profeta and Prucher, 2014).

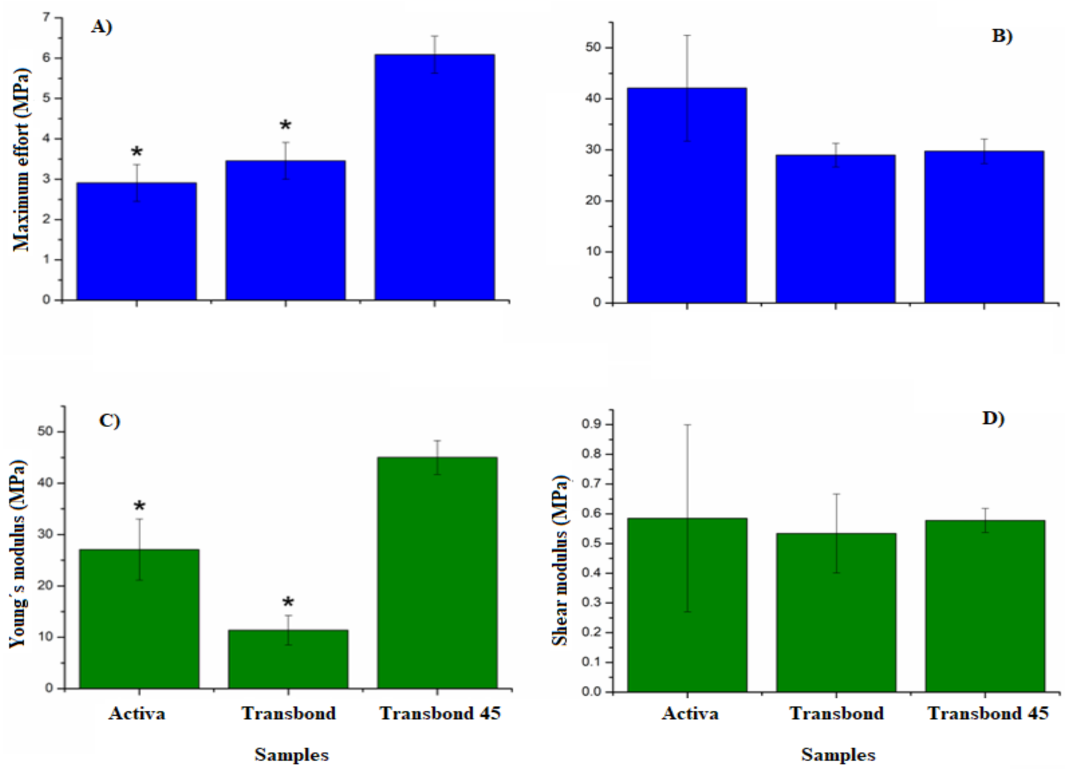

Figure 4. A) Maximum stress registered before yielding of the resin and composite material by the tensile load. * Significant difference for the Transbond45 with $\rho=0.0005$ and 0.0001. B) Maximum stress registered before yielding of the resins and compound material by the shear load. No significant difference between the resins is observed ($\rho=0.0751$). C) Young modulus of the resins and compound material. * A significant difference can be observed in the Transbond sample with $\rho=0.0051$. D) Shear modulus obtained for the resins and compound material. No significant difference is observed between the resins ($\rho=0.9394$).

In the assessment of the amount of residual resins on the dental surface, the Adhesive Remnant Index (RAI) was found. This index is of relevance in clinical studies. Its value is scaled as follows: 0 denotes the absence of adhesive residual on the dental piece, 1 indicates 50% of adhesive residual on the dental piece, 2 means that 50% or more of the adhesive remains on the dental piece, and 3 indicates that 100% of the adhesive remains attached to the dental piece (Alsamak et al., 2023).

Figure 5 shows the images of the brackets and dental pieces after detachment. Some adhesive remains are still attached to the bracket base and the tooth. As shown in Figure 5 (A and B) of the Transbond resin, there is an acceptable resin-tooth interface, but this is different for the resin-bracket interface. From these observations, the ARI index would be 3, which explains the prompt detachment from the bracket, as the statistical analysis shows. Figures 5 C, D, and F show stronger interfaces for the ACTIVA and Transbond45 resins, as they show evidence of adhesion both in the bracket and to the tooth surface, which places them at 1 on the ARI scale. From the statistical data and the images shown, it can be deduced that adding the 45S5 bioactive glass improves the tensile and shear resistances and the ARI of the Transbond resin by reinforcing the resin's inorganic matrix with particles of varying sizes. A lower ARI means that at the end of the orthodontic treatment, the orthodontist should have few problems removing the remaining adhesive from the dental pieces, producing little to no damage to the enamel.

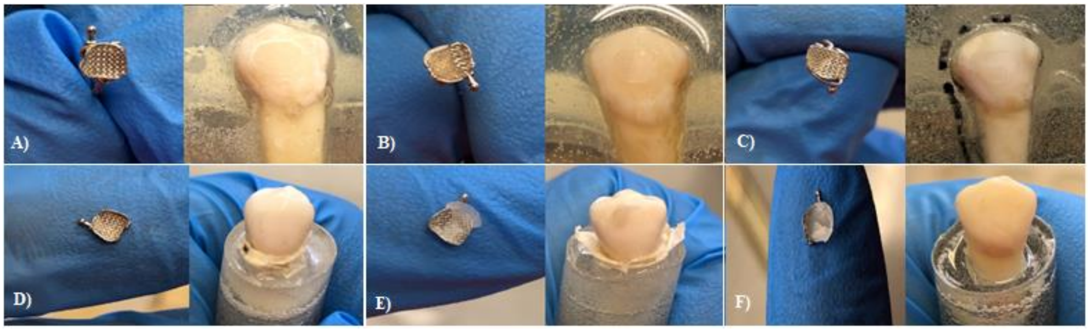

Figure 5. Bracket and dental piece images after detachment. A) Tensile load in the Transbond resin. B) Shear load in the Transbond resin. C) Tensile load in the Transbond45 compound material. D) Shear load in the Transbond45 compound material. E) Tensile loan in the ACTIVA resin, and F) Shear load in the ACTIVA resin.

Figure 6 shows the SEM and EDS images for the 21 days during which the samples were exposed to the SBF. These images show the deposition of the biological hydroxyapatite layer on the surface of the material and its increment with time. The layer shows a morphology similar to that of coral. The ACTIVA resin completely covers the surface, while the Transbond and Transbond45 exhibit less surface coverage. The EDS analysis shows the presence of P and Ca, which are characteristic of forming a hydroxyapatite layer in bioactive materials.

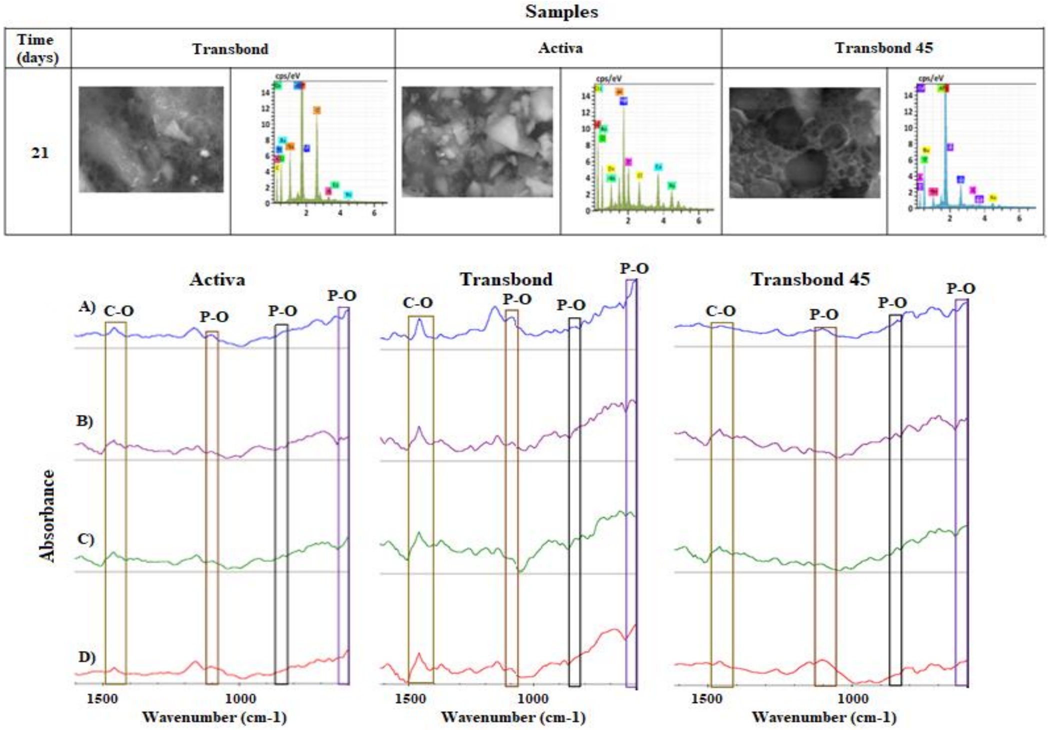

Figure 6. Sem and EDS images of the morphology and composition of the hydroxyapatite layer after 21 days of sample exposure to the SBF. Infrared spectra of the ACTIVA, Transbond, and Transbond45 compound material exposed to the SBF for A) 3, B) 7, C) 14, and D) 28 days**.**

Figure 6 shows the infrared spectra of the material exposed to the SBF. In the images, The absorption bands attributed to the functional groups present in hydroxyapatite can be identified. The phosphate groups can be observed at 660, 850, and 1100 cm^{-1}

(Alvarez-Monsiváis et al; 2022). The absorption bands for the carbonates groups can be observed at 870, 1420, and 1480 cm^{-1}. Finally, the absorption band associated with calcite appears at 1400 cm^{-1}.

Conclusions

In this study, the use of 45S5 bioactive glass was tested to improve mechanical properties, bioactivity, and degradation of Transbond resin used as an adhere metallic bracket. The results show that the Transbond45 samples were stable in their degradation and lost less weight than the Transbond and ACTIVA samples. The mechanical properties of the Transbond45 samples improved by adding the bioactive glass to the resin, as they withstood a more significant tensile load, increasing by approximately 5 MPa compared to the Transbond and ACTIVA resins. Also, the ARI of the Transbond45 resin was determined to be 1, thus improving the value of 3 of the base resin. This study shows that the Transbond45 resin is a compound material suitable as an adhesive in orthodontic applications, especially since the Transbond base resin is among the most widely used and commercially available resins.

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