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Bond strength of a three-step adhesive system modified by beta- TCP particles

Avaliação da resistência de união de um sistema adesivo convencional de três etapas modificado por partículas BETA-TCP

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ABSTRACT

Objective: This study aimed to evaluate the bond strength and fracture pattern of a three-step dentin adhesive system, Scotch Bond Multipurpose (SBMP) (3M ESPE), with or without beta-tricalcium phosphate (β-TCP) particles added to the primer solution. **Material and Methods:** Twelve human molar teeth were used, each sectioned perpendicularly into dentin discs. These discs were randomly allocated into three groups: G1 (control), G2 (primer modified with β-TCP 0.5%), and G3 (primer modified with β-TCP 2%). Each group consisted of four discs for testing. The dentin discs were treated according to the adhesive system protocol. After storage in distilled water, the discs were treated with 35% phosphoric acid gel, the primer (modified or not), and SBMP adhesive, followed by light-curing (Valo Ultradent). Twenty-four hours later, each restored dentin disc was sectioned to obtain specimens for tensile strength testing. Fracture pattern analysis was performed using scanning electron microscopy (SEM). **Results:** Statistical analysis of the tensile strength results showed no significant difference between the control and modified adhesive systems. The fracture pattern observed using SEM was predominantly mixed. **Conclusion:** The addition of β-TCP particles to the adhesive primer solution did not affect bond strength or the fracture pattern.

KEYWORDS

Dentin adhesives; Beta Particles; SEM; Tensile strength; Tooth remineralization.

RESUMO

Objetivo: Este estudo teve como objetivo avaliar a resistência de união e o padrão de fratura de um sistema adesivo dentinário de três etapas, Scotch Bond Multipurpose (SBMP) (3M ESPE), com ou sem partículas de fosfato de beta-tricálcio (β-TCP) adicionadas à solução primer. **Material e Métodos:** Foram utilizados doze dentes molares humanos, cada um seccionado perpendicularmente em discos de dentina. Esses discos foram aleatoriamente distribuídos em três grupos: G1 (controle), G2 (primer modificado com β-TCP 0,5%) e G3 (primer modificado com β-TCP 2%). Cada grupo consistiu de quatro discos para teste. Os discos de dentina foram tratados de acordo com o protocolo do sistema adesivo. Após armazenamento em água destilada, os discos foram tratados com gel de ácido fosfórico a 35%, primer (modificado ou não) e adesivo SBMP, seguido de fotopolimerização (Valo Ultradent). Vinte e quatro horas depois, cada disco restaurado foi seccionado para obter espécimes para teste de resistência à tração. A análise do padrão de fratura foi realizada utilizando microscopia eletrônica de varredura (MEV). **Resultados:** A análise estatística dos resultados de resistência à tração não mostrou diferença significativa entre o sistema adesivo controle e os modificados. O padrão de fratura observado por meio da MEV foi predominantemente misto. **Conclusão:** A adição de partículas de β-TCP à solução primer adesiva não afetou a resistência de união nem o padrão de fratura.

PALAVRAS-CHAVE

Adesivos dentinários; Partículas Beta; MEV; Resistência à tracção; Remineralização dentária.

INTRODUCTION

Composite resins, used in dentistry as restorative materials, are based on methacrylate monomers, inorganic filler particles and require an adhesive system to bond to dental tissues. Among the factors that can affect adhesive integrity, the polymerization shrinkage of these materials, causing microleakage and secondary caries, are disadvantages that can lead to restoration failure [1].

The development of composite materials with antibacterial properties can result in a reduction of their mechanical properties [2,3]. However, since antibacterial properties are important, especially at the interface between the restorative material and the tooth, it is more effective to incorporate antibacterial agents into the materials used as a base for restoration, such as adhesive systems [4]. Nevertheless, the use of these antibacterial materials alone does not guarantee the prevention or treatment of dental caries [5].

Calcium phosphate-based biomaterials are known for their high biocompatibility [6]. However, the combination of bioactive β-tricalcium phosphate (β-TCP) particles with water results in the precipitation of dicalcium phosphate dehydrate. This process can lead to microinfiltration, increasing the risk of dental caries and eventually resulting in restoration failure [4].

In three-step adhesive systems, dentin tissue is treated with 35% phosphoric acid to achieve proper interconnection between the composite and the tooth structure. This conditioning leaves a demineralized collagen layer, within which fluid and hydrophilic adhesive systems can penetrate. However, this penetration occurs with varying degrees of bonding and sealing. To achieve the antibacterial action of the adhesives, it would be beneficial for compounds based on calcium phosphate, for example, to be released, aiding in the remineralization of dentin tissues when conditioned [4].

Current adhesive systems are designed for superior clinical performance in adhesion-based aesthetic restorations, but they lack important antibacterial properties. Even materials that exhibit some antibacterial effects have not demonstrated the expected clinical significance. This is due to the lack of investment in antimicrobial formulations and the scarce evidence in the literature regarding their real benefits in clinical practice, especially in the prevention of caries recurrence [2].

Remineralization of areas affected by adhesive conditioning can improve the quality and longevity of the restorative material-dentin interface. Thus, developing materials that release bioactive ions is a goal of dental biomaterials research [7].

Biocompatibility is a common property of calcium phosphate-based materials, including hydroxyapatite, bioactive ceramics, tricalcium phosphate (α-TCP and β-TCP). The reason for the biocompatibility of calcium phosphate biomaterials is that this material is the main inorganic constituent of hard tissues, and free calcium and phosphorus ions can be used in metabolism [6].

The current goal in the manufacture of dental adhesives is to create a durable adhesion to dentin and protect the collagen fibrils exposed after acid action, either alone or using self-etching adhesives. The incorporation of minerals into this demineralized dentin is interesting because the mineral can act to reduce degradation at the adhesive interface [8].

Studies have shown that various particles have been incorporated into adhesive systems, aiming to promote ionic exchange and mineral precipitation with the hybrid layer. These particles include bioactive glasses, Portland cement, and calcium phosphate [9,10] however, the incorporation of these minerals can significantly reduce the mechanical properties of these composites [7].

The growing interest in minimally invasive treatments has led to the development of modalities that minimize the removal of dentin tissue. For these interventions, restorative materials with antibacterial properties would be highly beneficial and would aid in the remineralization process of these affected dentin tissues. Therefore, the aim of this study was to evaluate the bond strength and fracture pattern of a three-step dental adhesive, with or without the addition of β-TCP particles in the system's primer solution.

MATERIAL AND METHODS

For this study, 12 caries-free and/or restoration-free human molars were used, which were extracted for orthodontic, surgical, or periodontal reasons. After removal, they were cleaned and stored at 0°C.

The study was carried out in accordance with all the criteria of the Ethics Committee of the Pontifical Catholic University of Minas Gerais (PUC Minas) and approved under number 60187016.0.0000.5137.

Experimental groups, application of adhesives, and production of restorations

Four teeth were used for each experimental condition, as follows: G1, Scotchbond adhesive system primer without modification (control group); G2, Scotchbond multipurpose adhesive system primer (3M ESPE) modified with 0.5% by weight of β-TCP; and G3, primer modified with 2% by weight of β-TCP.

Before the adhesion and restoration procedure was performed with a composite resin, each tooth was fixed onto a lightly heated acrylic plate. The teeth attached to the plate were sectioned in a precision cutting machine (Isomet 100, Buehler Ltd., Lake bluuff, IL, USA) equipped with a diamond impregnated disc (15LC Series Diamond, Isomet Buehler-Microstructural Analysis Division, Lake Bluff, Illinois, 60044-USA) with abundant irrigation and a rotational speed programmed to 300 rpm. Two perpendicular cuts along the long axis of the tooth were produced. The first cut removed the occlusal portion and the second cut removed the roots of the teeth, approximately 2 mm above the cementoenamel junction. The final result was dentin discs approximately 4 mm thick with enamel on the edges. The pulp chamber area was filled with Scotchbond Multipurpose adhesive and Z100 composite resin (3M ESPE) (Table I), was adapted to close the hole in the pulp region in all discs and subsequently stored in distilled water.

After the first described step, each adhesive from each experimental condition was applied to the dentin surface according to the procedures described in Table I. The photopolymerization of the adhesive systems was carried out using a light-curing device (Valo, Ultradent) with an intensity of 1,400 mW/cm^2, as reported by the manufacturer. The increments of composite resin (Z100, Color A3, 3M ESPE) were applied directly onto the surface of the restored tooth, being light-cured for 20 seconds each, so that the final result was dentin discs restored with composite resin.

Table I - Composition and mode of application in dentin of the three-step adhesive system Scotchbond Multipurpose

Composition	Method of application (dentin)
Primer 2-hydroxyethyl methacrylate (HEMA) polycarboxylic acid and water.	1. Conditioning with 35% phosphoric acid for 15 seconds; 2. Rinse for 15 seconds; 3. Dry for 5 seconds;
Adhesive 2-hydroxyethyl methacrylate (HEMA), Bis-GMA and photoactivators.	4. Preparation: apply the primer to the conditioned enamel and dentin and dry gently for 5 seconds; 5. Adhesive application: apply the adhesive to prepared dentin and enamel; 6. Light cure for 10 seconds.

Production of test specimens

After applying the adhesives and preparing the composite resin restoration, all disks were stored in distilled water for 24 hours. Each restored dentin disc was attached to an acrylic base using a heated blade and secured to a precision cutter equipped with a diamond disc. A series of perpendicular cuts along the tooth axis in the distal-mesial direction were made under abundant water irrigation. The dentin and resin plates were then removed from the acrylic base and repositioned one by one with sticky wax to make cuts perpendicular to the initial cuts. Thus, specimens with the geometric shape of a toothpick and a cross-sectional area of 0.8±0.4 mm^2 were produced. Sticks from the peripheral region were discarded. Approximately 10 to 15 toothpicks were obtained from each restored dentin disc. All sticks produced from each disc were stored in distilled water at 37°C for 24 hours [11].

Tensile test

For each disc, five toothpicks were randomly selected to evaluate tensile strength. For the execution of the tensile test, each toothpick was attached to the Geraldelli device with cyanoacrylate based glue. This device was then coupled to a universal test machine (Bisco, Schaumburg, IL, USA) with a 500N load cell (Static Load Cell, Instron, Norwood, MA, USA). The tensile test was conducted at a velocity of 0.5 mm/min until total specimen rupture, obtaining union strength values in Newtons (N).

The conversion of the values into megapascals (MPa) was performed after measuring the cross-sectional area of the specimens using an electronic digital caliper (Vonder PDV 1500, O.V.D

Importadora e Distribuidora LTDA, Curitiba, PR, Brazil), according to the following formula:

$$\frac{\textit{Resistance value in Newtons } (N) = \textit{ MPa}}{\textit{Specimen area in mm}^2} \qquad (1)$$

Each experimental condition consisted of 20 toothpicks, originating from the four discs (each disc supplied five toothpicks). Thus, the number of samples (n) in the present study was 20.

Fracture pattern

The specimens with the highest and lowest resistance values of each disc were considered for evaluation of the fracture pattern. Thus, eight specimens from each experimental condition were evaluated: four discs for each condition, and two sticks with the highest and lowest MPa values were removed. To facilitate classification, the two fractured sides of the composite resin and dentin were fixed in double-sided carbon tape stubs and dehydrated for 2 hours inside a silica gel- containing container. The specimens were then covered in gold using a metallizer (Denton Vacuum, DESK V, Standard model, JEOL, USA) and were observed using a scanning electron microscope (SEM; JEOL, JSM - IT 300, USA) with an acceleration of 30 KV, WD of 14 mm, spotsize of 55 nm, and a high vacuum.

Each selected test body was classified according to the following denominations: Type I, adhesive failure at the base of the hybrid layer; Type II, cohesive failure in dentin; Type III, cohesive failure in the hybrid layer; Type IV, cohesive failure in the adhesive layer; and Type V, cohesive failure in the composite resin.

Production of primer solutions

Two solutions of experimental primer were produced. The primer base solution was that of the three-step adhesive system, Scotchbond Multipurpose. For each solution, 1 ml was extracted from the primer vial and placed in a 1.5 ml Eppendorff flask. We followed the previous recommendations of Mehdikhani and Borhani to synthesize β-TCP nanoparticles. In brief, calcium nitrate tetrahydrate (($Ca(NO_3)_2 \cdot 4H_2O$) (98%, Merck, Kenilworth, NJ, USA) and diammonium hydrogen phosphate (($(NH_4)_2HPO_4$) (99%, Merck) were reacted to produce β-TCP nanoparticles. Initially, a white precipitate was formed by adding 500 mL of 0.6 mol $Ca(NO_3)_2$ (pH = 7.3) dropwise over 2–3 hours into 500 mL of 0.4 mol $(NH_4)_2HPO_4$ solution (pH = 4) that was vigorously stirred beforehand. During this reaction, aliquots of 0.1 M sodium hydroxide (99%, Merck) were used to maintain the pH of the system. The resultant white precipitate was stirred for 12 hours and then washed with distilled water (DW) and ethanol to enhance its dispersion properties. Using gentle suction, this mixture was filtered through a filter glass. The filtration cake was dried at 80°C for one day. The next day, the dried powders were ground using a mortar and pestle and calcined in an alumina crucible at 700°C for 2 hours [11]. These solutions, as well as the original primer solution (control) were used to prepare the conditioned dentin with phosphoric acid.

The results obtained in the tensile tests from different groups were evaluated statistically: normality test D'agostino and Pearson, Analysis of variance (ANOVA) and Tukey test with level of significance 95%. The analysis of variance (ANOVA) will compare the bond strength of an adhesive system modified with the addition of β-TCP particles at concentrations of 0,5% and 2,0% to the unmodified adhesive system. It will evaluate whether the observed differences between the sample means are statistically significant, using the F-test to determine if the effect of the additives is significant compared to the system without any β-TCP particle addition.

RESULTS

Tensile test

There was no statistically significant difference between the groups in the traction test after 24 hours or storage time. Results of data analyzed by ANOVA (Table II and Graph 1).

Fracture pattern

The fracture pattern in analyzed specimens was predominantly mixed, representing adhesive and cohesive failure.

Table II - Mean values of standard deviation obtained through the tensile tests in megapascals

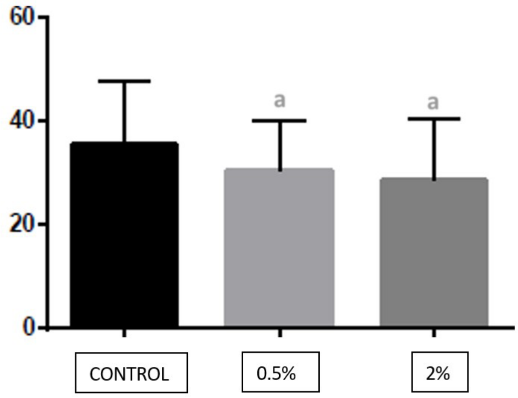
Adhesive	24 Hours
SMBP control	35.37 (12.54) A
SBMP + 0.5% β-TCP	30.50 (9.53) A
SBMP + 2% β-TCP	28.51 (11.86) A

SBMP, Scotchbond Multipurpose; β-TCP, beta-tricalcium phosphate; A, there is no statistical difference.

SEM analysis of each body showed that cohesive failure was predominant in relation to adhesive failure, varying between cohesive defects in the dentin, cohesive in the hybrid layer, cohesive in the adhesive layer, and cohesive in the composite resin (Figures 1, 2 and 3).

The SEM images showed that the majority of specimens had mixed fracture patterns, and adhesive defects could be observed at the base of the hybrid layer in the presence of cohesive faults, which are located in the dentin, hybrid layer, adhesive, and composite resin (Figures 2).

Only a purely adhesive failure was found in a fractured specimen in the control group. The other samples presented mixed patterns (Graph 2).



Graph 1 - Mean values and standard deviation obtained through the tensile tests in megapascals.

DISCUSSION

Ensuring the longevity of adhesive restorative procedures means that efforts were made to stabilize the dentin region after acid etching and exposure of collagen fibers. The concept of remineralization based on the interaction of bioactive ions was recently developed [12] and the central objective would shield the demineralized collagen layer [13] after using phosphoric acid on dentin. There should be no damage or a decrease in the region of the union. The incorporation of bioactive inorganic fillers can enhance the mechanical properties of dentin adhesives. The presence of essential remineralization sources, such as calcium and phosphate, ensures that the dentin adhesive can remineralize the adhesive-dentin interface [14,15].

The present study confirmed that β-TCP at concentrations of 0.5% and 2% did not cause a decrease in bond strength values with dentin. The presence of these two ion sources ensures the remineralization of the adhesive-dentin junction, extending the lifespan of the composite restoration [11,16].

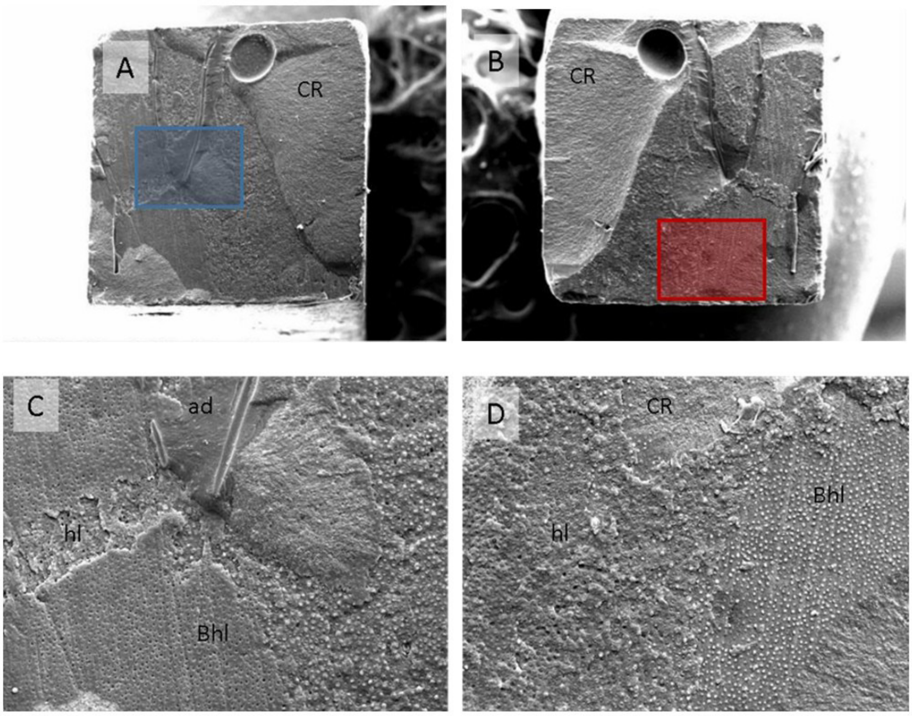


Figure 1 - SEM images of the Scotch Bond Multipurpose adhesive system, showing mixed fracture patterns. Two homologous bodies were observed after the fracture in smaller and larger increase, in the left and right columns (dentin side). CR, composite resin; hl, hybrid layer; Bhl, base of the hybrid layer; ad, sticker. The semi-transparent blue box in the image (A) represents the largest increase shown in (C). The semitransparent red image box (B) represents the increase shown in (D).

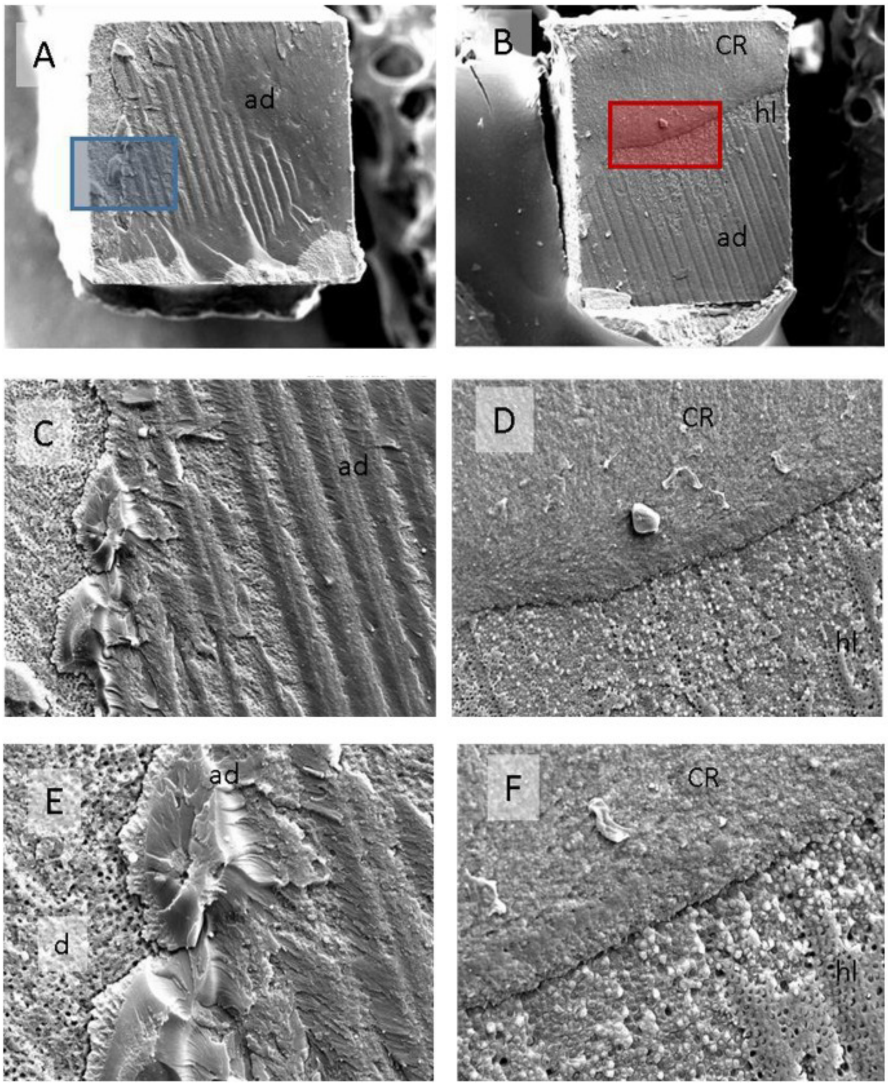


Figure 2 - SEM images of the Scotch Bond Multipurpose adhesive system, modified with 0,5% β-TCP by weight, showing a mixed fracture pattern. CR, composite resin; hl, hybrid layer; Bhl, base of the hybrid layer; ad, sticker. The semi-transparent blue box in the image (A) represents the largest increase shown in (C). The semitransparent red image box (B) represents the increase shown in (D). The image (E) represents the magnification of image (C) and the image (F) represents the magnification of image (D) (x500 increase).

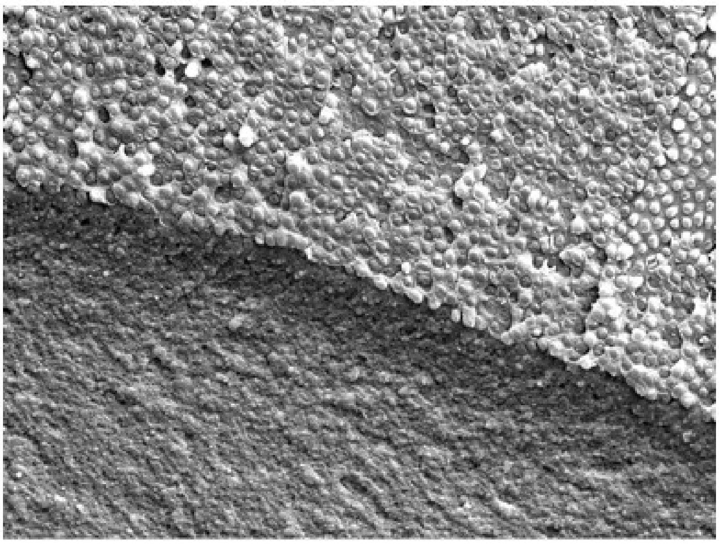
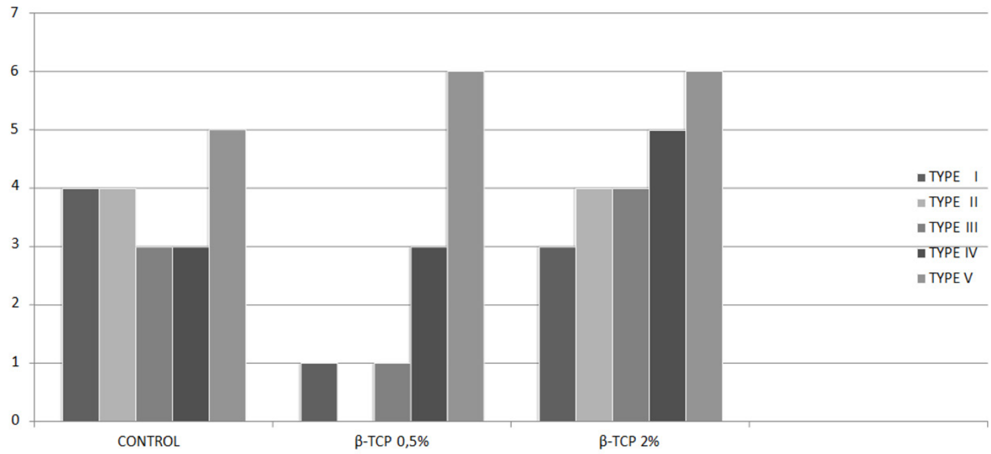


Figure 3 - SEM image of the Scotch Bond Multipurpose adhesive system, modified with 2% β-TCP by weight, showing a mixed fracture pattern.



Graph 2 - Evaluation of fracture patterns obtained using scanning electron microscopy (SEM). All fracture patterns were present on the analyzed discs, and the predominant fracture pattern was type V. Type I, adhesive at the base of the hybrid layer; Type II, cohesive failure in dentin; Type III, cohesive failure in the hybrid layer; Type IV, cohesive failure in the adhesive; Type V, cohesive failure in the composite resin.

The justification for using these concentrations of β-TCP (0.5% and 2%) is that this component must dissolve in the primer solutions of the adhesive systems. When β-TCP is added in concentrations greater than 2% by weight, the solutions become very viscous, often making it impossible to fully dissolve the β-TCP. This prevents the correct execution of adhesive procedures according to the manufacturer's recommendations. A previous study suggested that when incorporating inorganic fillers into adhesives, the weight concentration should not exceed 10%, as this may compromise bond strength due to a significant increase in viscosity [17,18].

In an in vitro study, adhesives capable of releasing fluoride ions were tested to evaluate the deposition of crystals in gaps between restorative materials and dental tissues (enamel and dentin). Positive results were observed over the storage period, suggesting that such adhesives could repair flaws resulting from these procedures [19,20].

Other studies that have sought modifications in adhesive systems to make them bactericidal and induce remineralization include Bapna et al. In 1988 and 1992, studies have been conducted to evaluate the antibacterial properties of Scotch Bond adhesive and other adhesive systems, as well as to assess bond strength [21-23]; other authors have tried to introduce antibiotics and bactericidal agents into the adhesive or primer systems with good results from the biological and mechanical points of view [24].

Since, adhesion in various parts of the tooth can be influenced by different functional monomers, methacrylates, solvents, the pH of adhesive systems, in addition to the thickness of the dentin [25].

Since then, new strategies have been used to reduce degradation in adhesive restorations. The current strategy to reduce degradation at the adhesive interface is called biomimetic remineralization [26] and it consists of inducing the formation of apatite crystals in the exposed collagen regions and in the internal spaces of the adhesive layer [27]. This process intends to imitate the natural process of remineralization by filling the collagen of the demineralized dentin in the interfibrilar and intrafibrillary regions [28].

The biomimetic remineralization process is modulated by non-collagenous analogues of dentin phosphoproteins [12,28,29]. They are responsible for maintaining the calcium phosphate present in the nanometer-sized dentin, sufficient to penetrate the demineralized collagen fibrils [12].

Bioactive Portland cement (calcium silicate) is the most widely used substance for biomimetic remineralization, with promising laboratory results regarding remineralization of dentin and union interface [30,31]. However, when its dissolution occurs, there is formation of spaces that are filled by water, leading to degradation [31]. Another alternative, which was used in the present study, is using materials based on β-TCP, which may induce the formation of new hydroxyapatite crystals [4].

The use of calcium phosphate-based compounds when combined with water leads to the precipitation reaction of another calcium phosphate-based product, dicalcium phosphate dihydrate [32]. Dentin adhesives when applied close to the dental pulp can lead to severe pulpal inflammation [33], while calcium phosphate-based biomaterials are highly biocompatible [6].

Katoh et al. [34] suggested that the development of adhesives containing calcium phosphate powder as a material for direct pulp capping could lead to the formation of restorative dentin on the exposed pulp. The present study shows that, under our experimental conditions, there was no mechanical damage associated with both materials in a formulation that maintains its mechanical properties associated with β-TCP, a biomaterial highly that is biocompatible and capable of acting in the direction of biomimetic remineralization.

Previous studies have evaluated the relationship between the release of bioactive ions, such as fluoride, and the influence on the mechanical properties of these materials, and it can be seen that those materials with the highest fluoride release had the worst mechanical properties [35]. However, Profeta et al. [31] obtained promising mechanical effects by adding calcium/sodium phosphosilicate in the adhesive protocol in composite resin restorations. At different storage times (24 hours and 6 months), the results of the tensile tests of the test and control groups were similar. Furthermore, the authors demonstrated that bioactive additives of calcium phosphate particles could increase bond strength through mineral deposition in the region, acting against the enzymatic degradation of the hybrid layer.

Other authors also obtained satisfactory results in mechanical tests using β- TCP. One of the criteria evaluated by Sauro et al. to include β-TCP particles in photopolymerizable resins was the microtraction assay. The results were positive when incorporating bioactive biomaterial particles into restorative materials [7,36].

The present study confirmed the nature of the faults using SEM images. Adhesive failures that result from material or adhesive procedure failures were uncommon. This suggests that, from the mechanical point of view, β-TCP did not negatively affect the results.

Further studies are needed to confirm the idea of modifying consolidated adhesive systems to improve the biological remineralization and protection capabilities of both the pulp dentin complex and the adhesive interface.

CONCLUSION

The effects of bonding strength with the addition of β-TCP particles were investigated in this study, revealing promising results from a mechanical perspective. The microtraining tests showed that adhesive failures, predominantly adhesion failures, occurred in the experimental groups that used β-TCP. However, no significant statistical differences were observed.

Additional laboratory studies are necessary to better evaluate the β-TCP concentrations proposed in this study, especially over long storage periods.

Author's Contributions

LRAA: Methodology and writing – Original Draft Preparation.SMM: Methodology and Writing Review & Editing. MY: Methodology. ERAL: Data Curation . REFQN: Supervision. ANGA: Project Administration, supervision and formal Analysis.

Conflict of interests

The authors of this manuscript certify that there is no financial or personal interest of any nature or type in any product, service and/or business that is presented in this article.

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Regulatory Statement

This study was conducted according to all the requirements of the Ethics Committee of the Pontifical Catholic University of Minas Gerais (PUC Minas) and received approval under number 60187016.0.0000.5137.

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