

# EFFECT OF CPP-ACP ON THE BOND STRENGTH OF SILORANE AND METHACRYLATE BASED RESTORATIVE SYSTEMS

## ABSTRACT

This study evaluated the influence of the previous application of casein phosphopeptide amorphous calcium phosphate (CPP-ACP) on the bond strength of two restorative systems to dentin. Self-etching adhesive systems Clearfil SE Bond (Kuraray) and Filtek™ LS System Adhesive (3M ESPE) were used in combination with two micro-hybrid composites Filtek™ Z250 (3M ESPE) and Filtek™ LS (3M ESPE), respectively. Twenty-eight sound human third molars had the occlusal surface worn until the total exposure of dentin and were randomly divided into 4 groups (n = 7): G1 (Clearfil + Z250), G2 (CPP-ACP + Clearfil + Z250), G3 (LS system) and G4 (CPP-ACP + LS system). The application of the paste containing CPP-ACP was held in the dentin of G2 and G4 prior to adhesive protocol, which followed the manufacturer's recommendations. Two cylinders of a single increment of resin composite were built on each dentin surface using tygon's matrix with 1.26 mm of diameter x 1 mm high and light-cured with LED Bluephase 16i (Vivadent) apparatus. Microshear bond strength test was conducted in a universal testing machine (EZ Test, Shimadzu) with a speed of 0.5 mm/min. The data were subjected to two-way ANOVA test with a significance of 5%. There was no significant difference on bond strength between restorative systems with or without the previous use of a CPP-ACP paste. It was concluded that the application of CPP-ACP in the dentin prior to adhesive protocol did not influence the bond strength of the restorative systems tested.

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## KEYWORDS

Composite resins. Dentin. Shear strength. Dental materials.

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## INTRODUCTION

The dentin exposure caused by loss of enamel or cementum in the cervical region of the teeth is a common problem, and may be related to attrition, erosion, or tooth abrasion.<sup>1</sup> These diseases are known as non-carious cervical lesions (NCCL), being responsible for most cases of dentin hypersensitivity.

Treatment of these diseases constitutes in the obliteration of dentinal tubules exposed with the use of desensitizing agents such as potassium oxalate, application of adhesives, or restoration of the lesion with composite resin.<sup>1</sup> Other forms of treatment of these lesions and remineralization of exposed dentin are based on the fluoride solutions or application of pastes containing amorphous calcium phosphate.<sup>2</sup>

In order to obtain greater efficiency in the restorative treatment of NCCL, self-etching adhesive systems are used. This class of adhesive partially demineralizes the dentin and promotes the infiltration of the monomer concurrently reducing the risk of post-operative sensitivity.<sup>3</sup> This system promotes permeability of the smear layer without removing it completely, infiltrating it and partially dissolving the hydroxyapatite, creating a hybrid layer that incorporates minerals.<sup>4</sup> The self-etching systems reduce the over acid conditioning of dentin, and the penetration of non-polymerized monomers in the dentinal tubules, thus reducing tooth

sensitivity caused by pulp inflammation.<sup>5</sup>

Another advantage is the smallest number of steps presented in this technique, which reduces the chance of operator error, increasing the success rate of these procedures. Studies have been demonstrated that the bond strength of a two-step self-etching systems in dentin is very close to that found in conventional adhesive systems.<sup>6</sup>

The absence of enamel around the perimeter of NCCL is one of the responsible factors for the failure of class V restorations. Studies show that the quality of adhesion to dentin is lower than to the enamel,<sup>7</sup> so it seems reasonable to attempt to mineralization of this tissue, increasing the quality of adhesion and reducing the chances of failure due to leakage.

Recent studies have shown that the use of casein phosphopeptide amorphous calcium phosphate (CPP-ACP), which is a substance derived from milk protein, promotes the release of calcium phosphate bio-available to assist in enamel remineralization of human teeth.<sup>8</sup> The application of this substance, prior to the application of etch-and-rinse and self-etching adhesive systems can increase the bond strength of resin-based materials to enamel.<sup>9</sup>

However, other authors reported that the application of CPP-ACP may reduce the bond strength to enamel when associated with the use of self-etching adhesive systems.<sup>10</sup> But there are few studies in the literature evaluating the effect of the paste containing

CPP-ACP in the bond strength and stability to dentin.

Another factor that impairs the quality of class V restorations is the polymerization shrinkage of composite resins, which can create stress in the adhesive interface between the composite and tooth substrate.<sup>11</sup> Shrinkage stress generated can lead to cusp deflection and rupture of the bond in this interface, with consequent predisposition to leakage, installation of secondary caries and post-operative sensitivity.<sup>12</sup> An attempt to minimize the failure at the dentin-restoration bond interface is the development of resin monomers that have lower polymerization shrinkage. The monomeric system of chemical compounds derived from oxirane and the siloxane component, called silorane, has led to a lower polymerization shrinkage when compared to the methacrylate.<sup>13-15</sup> The resin-based silorane manufactured commercially by the name Filtek LS (3M ESPE) is used in conjunction with self-etching adhesive system which consists of acidic primer and adhesive, both photopolymerizable. Few studies reported in the literature have evaluated the effect of the paste containing CPP-ACP on the bond strength to dentin, especially in association with the use of new restorative systems with low polymerization shrinkage and silorane-based adhesives.

The aim of this study was to evaluate the influence of the paste containing CPP-ACP,

prior to the use of self-etching adhesive systems and different composite resins on bond strength to dentin, using the microshear bond strength test. The null hypothesis tested was that the use of CPP-ACP prior to the adhesive restorative procedure would not affect the bond strength of self-etching adhesive systems tested.

## MATERIAL AND METHODS

This research was approved by the Ethics Committee (FOP-UNICAMP, protocol # 101/2011). Twenty-eight third molars fully erupted, extracted from young patients for orthodontic reasons were selected. The teeth were stored for 24 h in a solution of 2% thymol to disinfect. The roots were debrided with periodontal curettes and cleaned using pumice and water paste with prophyl brush in a hand piece<sup>16</sup>, and after stored in distilled water for 1 month to the time of testing.

### 1. PREPARATION OF SAMPLES

The teeth had the occlusal surface worn with a 360-grit silicon carbide sandpaper (Norton, Guarulhos, SP, Brazil) in a polishing machine (APL-4, Arotec, São Paulo, SP, Brazil) until the total exposure of the dentin surface, and they were evaluated in a stereomicroscope Stereoscan 440 (Leica, Cambridge, England).

The teeth were embedded in

polystyrene resin (Piraglass, Piracicaba, SP, Brazil), with the occlusal surface exposed. To standardize the smear layer, the polished dentin surfaces were worn again for 1 minute with 600-grit sandpaper. The teeth were randomly divided into four groups (n = 7), according to the use or not of CPP-ACP paste on dentin and the type of resin monomer (methacrylate and silorane), in association with their self-etching adhesive systems: G1 (Clearfil + Z250), G2 (CPP-ACP + Clearfil + Filtek Z250), G3 (LS System Adhesive + Filtek LS) and G4 (CPP-ACP + LS System Adhesive + Filtek LS).

The application of paste containing CPP-ACP (MI Paste, GC Corporation, Tokyo, Japan) in the dentin surface was performed for 3 minutes and followed by washing in running water for 30 seconds<sup>9</sup> in G2 and G4, prior to the application of the adhesive protocols according to the manufacturer's instructions. The adhesives were light cured for 10 s using Bluephase 16i LED unit (Vivadent, Burs, Austria) with light intensity of 1390 mW/cm<sup>2</sup>. Arrays of Tygon (Tygon tubing, TYG - 030, Saint-Gobain Performance Plastic, Maime Lakes, FL, USA), with approximately 1 mm of height and 1.26 mm in diameter, were used to make up the resin cylinders. The composites were inserted into the mold in a single increment and a polyester strip was placed on the matrix.

The tip of the LED was pressed against the polyester strip and the light exposure was made for 20 s, according to manufacturer's recommendation. Two cylinders were made for each sample. The samples were stored for 24 h in distilled water at 37° C before the microshear bond strength test.

## 2. MICROSHEAR BOND STRENGTH

Specimens were fixed in particular device for the microshear test coupled to a universal testing machine (EZ Test, Shimadzu Co., Kyoto, Japan). Each cylinder was wrapped in adhesion area of composite with an orthodontic wire (0.3 mm in diameter). The test was conducted at a speed of 0.5 mm/min until failure of the samples. Values were obtained for maximum resistance provided by the machine in kgf. To calculate the bond strength values (MPa) it was used the following formula:  $MPa = Kgf \times 9.8 / \text{Adhesion area (mm}^2\text{)}$ .

The values were submitted to the two-way analysis of variance (p<0.05).

## RESULTS

The analysis of variance (ANOVA) of two factors showed no statistically significant difference neither for the factor restorative system (P<0.05) nor for the factor treatment with the paste containing CPP-ACP (P<0.05)

and not for the interaction of the factors ( $p < 0.05$ ). The bond strength values are shown in Table 1.

Table 1. Means (standard deviations) of microshear bond strength test (MPa).

Resin Composite	CPP – ACP	
	With application	Without application
Filtek Z250	7.23 (2.96) Aa	5.73 (2.50) Aa
Filtek LS	6.25 (2.73) Aa	5.22 (1.48) Aa

\* Similar letters (uppercase in the row and lowercase in the column) are not statistically different ( $p > 0.05$ ).

## DISCUSSION

According to the results of this study the application of the paste containing CPP-ACP did not influence significantly on the bond strength values of any self-etching adhesive used. Therefore, the null hypothesis tested was accepted. The CPP-ACP is the association of amorphous calcium phosphate (ACP) to a protein of milk, casein phosphopeptide, and it has been studied because of their ability to increase the concentration of calcium and phosphate ions, which assist in remineralization of dental tissues by the deposit of apatite, and their ability to control dentinal hypersensitivity.<sup>17,18</sup>

Because of that, there are some suggestions that the use of CPP-ACP may cause a higher rate in dentin mineralization hindering the conditioning of self-etching adhesives in relation to groups which were not treated with the paste.<sup>17</sup> However, other studies have shown that prophylactic application of CPP-ACP paste on enamel prior

to self-etching adhesive systems can increase the bond strength of resin-based materials to enamel.<sup>9</sup>

The absence of difference may be due to acid-base reaction between the hydroxyapatite deposited by the CCP-ACP paste and acidic monomers present in the self-etching adhesive. Self-etching adhesives tested has low pH and may be classified into mild, moderate and aggressive,<sup>19</sup> that not remove the smear layer but aggregates it inside the adhesive layer, while the pH is stabilized by the release of ions from the dentin. In this case the calcium reagent was possibly deposited by CCP-ACP paste, but it presented no influence on bond strength, in disagreement with the researches that assessed the application of CCP-ACP paste in enamel.<sup>9, 10</sup> Furthermore, the use of self-etching adhesive systems have the advantage of lack of washing steps and drying while maintaining the optimum moisture dentin and additionally the entire demineralized dentin is filled by the adhesive monomer system,

keeping the residual hydroxyapatite, which can improve bonding and durability of the restorative procedure.<sup>4</sup>

Some authors have reported that nanocomplexes formed with the use of CPP-ACP could promote the protection of the tooth hard tissues adjacent to pH changes, reducing demineralization and remineralization of the tooth in helping.<sup>17,18</sup>

Regarding the type of resin-based materials used to restore the dentin, no significant difference was found on the bond strength. Although the Filtek LS resin composite has a coefficient of post-gel shrinkage of about 0.11 and the Filtek Z250 resin, about 0.51<sup>20</sup> the immediate results showed no adhesive failure that could have been caused by polymerization shrinkage stresses generated in the bonding interface.

There is a study showing that in hydrophobic resins the polymerization stress can be stored in the adhesive interface by up to four weeks,<sup>21</sup> which can cause decreased adhesive strength in the composites with high volumetric shrinkage polymerization coefficient, such as Filtek Z250. In this case, the paste containing CCP-ACP probably could influence the bond strength. But more studies evaluating the aging of the samples must be done.

Thus, this study showed that the previous application of CPP-ACP with self-etching adhesive systems and restored using

silorane- or methacrylate-based resin did not influence on the bond strength of restorative systems tested. This indicates that the CPP-ACP paste may be used prior to bonding procedures as a promoting remineralization and reducing demineralization and post-operative sensitivity without decreasing the bond strength.

## CONCLUSION

It can be concluded that the paste containing casein phosphopeptide amorphous calcium phosphate can be associated with self-etching adhesives and methacrylate- or silorane-based composite resins without a decrease in the the bond strength to dentin.

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