

# Mestrado Integrado em Medicina Dentária

# Effect of water aging on dentin bond strength after tooth fragment reattachment Original Scientific Article

Mara Filipa Oliveira Gorito

Advisor: Doutora Alexandra Vinagre Co-Advisor: Prof. Doutor João Carlos Ramos

Coimbra, julho 2019

Faculdade de Medicina da Universidade de Coimbra Mestrado Integrado em Medicina Dentária

# Effect of water aging on dentin bond strength after tooth fragment reattachment

# **Original Scientific Article**

# Gorito M<sup>1</sup>, Vinagre A<sup>2</sup>, Ramos JC<sup>3</sup>

1) 5th year student of Integrated Master in Dentistry, Faculty of Medicine, University of Coimbra

2) Assistant Lecturer of Integrated Master in Dentistry, Faculty of Medicine, of University of Coimbra

3) Assistant Professor of Integrated Master in Dentistry, Faculty of Medicine, of University of Coimbra

Área de Medicina Dentária, FMUC, Coimbra, Portugal Avenida Bissaya Barreto, Blocos de Celas 3000-075 Coimbra Tel.: +351 239 484 183 Fax: +351 239 402 910 Author's e-mail: maraoliveiragorito@gmail.com

# Contents

Abstract	5
Introduction	7
Materials and Methods	10
Results	13
Discussion	15
Conclusion	20
Acknowledgements	21
References	22

## 1.Abstract

**Introduction:** Dental trauma is a serious worldwide public health issue more frequent in phases of individuals' growth and development. Regardless the occasion, the absence of treatment may lead not only to aesthetic concerns but also to functional and emotional disturbances. There are still very few publications that provide us with information about the procedure for applying the fragment "bonding" materials that we should choose to ensure clinical longevity.

**Purpose:** To compare the dentin microtensile bond strength of three different tooth reattachment techniques and evaluate failure modes, after two years water aging.

**Materials and methods:** Two years ago, fifteen third molars were selected, prepared and randomly divided into three groups (n=5). Each tooth was transversely sectioned at the crown level due to obtain a detached crown fragment. Afterwards, the bonding protocol with resin composite was applied in three different ways according to three groups: conventionally with a layer of resin composite (group I), pre-heated resin composite at 50°C (Group II) and resin composite applied with ultrasonic vibration (Group III). For all groups a controlled 30 N force was applied. After bonding the teeth were cross-sectioned and sticks with 1 mm<sup>2</sup> were obtained. They were then tested in tension in a universal testing machine at 0,5 mm/min. The mode of failure was evaluated with optical microscopy. Data was analyzed with the IBM SPSS software and significance level was set at  $\alpha = 0.05$ .

**Results:** The following microtensile bond strengths were registered (mean in MPa  $\pm$  SD): Group I – 21,8  $\pm$  8,7; Group II - 20  $\pm$  10,6; Group III- 23,9  $\pm$  7,4. No statistical differences were found. All groups experienced the three types of failure.

**Conclusion:** There were no differences in bond strength among different groups, so the material used, and techniques were not a significant influence for bond strength, two years after reattachment.

**Keywords:** dental trauma; tooth fragment reattachment; composite resin; ultrasonic vibration; pre-heated composite, dentin bond strength; microtensile bond strength.

## 1.Resumo

**Introdução**: O trauma dentário constitui um problema mundial de saúde pública sendo mais frequente em fases de crescimento e desenvolvimento dos indivíduos. Independentemente da ocasião, a ausência de tratamento pode levar não só a preocupações estéticas, mas também a distúrbios funcionais e emocionais. Ainda são poucas as publicações que nos fornecem informações sobre o procedimento para aplicar os materiais a utilizar na "colagem" dos fragmentos de modo a garantir a longevidade clínica.

**Objetivo:** Comparar a força de união da interface dentina-resina-composta de três diferentes técnicas de reinserção dentária e avaliar os modos de falha após dois anos de envelhecimento com água.

**Materiais e métodos:** Há Dois anos, quinze terceiros molares foram selecionados, preparados e aleatoriamente divididos em três grupos (n = 5). Cada dente foi seccionado transversalmente no nível da coroa para a obtenção de um fragmento da coroa. Posteriormente o protocolo de colagem com resina composta foi aplicado de três formas diferentes de acordo com três grupos: convencionalmente com uma camada de resina composta (grupo I), resina pré-aquecida a 50°C (Grupo II) e resina composta com vibração ultrassónica (Grupo III). Em todos os protocolos foi aplicada uma força controlada de 30 N. Após a reiserção dos fragmentos, os espécimes foram seccionados transversalmente e obtiveram-se bastões com 1 mm<sup>2</sup>. Foram então testados em tensão numa máquina de ensaios universal a 0,5 mm / min. O modo de falha foi avaliado com microscopia ótica. Os dados foram analisados pelo software IBM SPSS e o nível de significância foi estabelecido em  $\alpha = 0,05$ .

**Resultados:** Foram registadas as seguintes forças de adesão à microtração (média em MPa  $\pm$  DP): Grupo I - 21,8  $\pm$  8,7; Grupo II - 20  $\pm$  10,6; Grupo III - 23,9  $\pm$  7,4.

**Conclusão:** Não houve diferenças na resistência de união entre os diferentes grupos, de modo que o material utilizado e as técnicas não foram uma influência significativa para a resistência de união, dois anos após a reinserção.

**Palavras-chave:** traumatismo dentário; reinserção fragmento dentário; resina composta; vibração ultrassónica; compósito pré-aquecido, força de adesão dentinária; resistência de microtração.

# 2.Introduction

Dental trauma is a serious worldwide public health issue more frequent in phases of individuals' growth and development.

The mean ages of children with affected primary and permanent teeth were  $3,35\pm 2,02$  and  $9,09\pm 2,43$  years.<sup>(1)</sup>

Both male and female children in the age group of 13-14 years have experienced highest trauma, when compared with children in the age group of 9-10 and 11-12 years.<sup>(2)</sup>

According to Eltair M *et al.*<sup>(3)</sup> the prevalence of traumatic crown injuries in German adolescents was 6.3% in the 10 and 12-year-old children and 14.0% in 15-year-old children.

Traumatic dental injuries are more prevalent in permanent (58.6%) than in primary dentition (36.8%).<sup>(4,5)</sup> It has been shown that the affected dentition was related to gender. In permanent dentition, boys suffer more dental injuries compared with girls.<sup>(6)</sup> However, in deciduous dentition, that difference was not found.<sup>(7)</sup> Dental trauma mainly involves the upper anterior teeth, affecting more frequently the maxillary central incisors followed by maxillary lateral incisors,<sup>(8)</sup> in both dentitions.<sup>(9)</sup> This was ascribed to their position in the arch and to the eruptive process that causes protrusion, which is more marked in patients who present class II, division 1 Angle malocclusion.<sup>(7,9)</sup>

Traumatic dental injuries occur mainly at home or school. The most frequent causes are accidents, like falling, sports collisions, traffic accidents and physical violence. Regardless the occasion, the absence of treatment may lead not only to aesthetic concerns but also to functional and emotional disturbances.<sup>(6,10)</sup> Sanbuncuoglu and Irmak<sup>(11)</sup> also related that attention deficit hyperactivity disorder (ADHD) may exacerbate such situations, increasing the individual predisposition to dental trauma. ADHD is the most common psychiatric developmental disorder, which affects up to 4 to 12% of all school-age children. De Oliveira Filho PM *et al.* <sup>(12)</sup> concluded that the use of illicit drug among adolescents increases prevalence of dental trauma.

According to the classification of the International Association of Dental Traumatology (IADT), dental trauma can be classified as follows: enamel fissure, crown fracture (involving only enamel, extending to the dentine with or without pulp involvement); fracture that extends from the crown to the root (with and without pulp wrapping); root fracture; alveolar fracture; concussion; subluxation and dislocation (lateral, intrusive and extrusive).<sup>(13,14)</sup>

Crown fracture accounts to 92% of traumatic injuries affecting the permanent dentition.<sup>(15)</sup> Uncomplicated crown fractures, without pulp exposure, are the most common trauma in permanent dentition while subluxations and complete luxation's occur more frequently in the deciduous dentition.<sup>(6)</sup> Several approaches have been developed for the reconstruction of fractured teeth, requiring regular intervention of esthetic dentistry, fixed prosthodontics and/or, on a timely basis, endodontics, periodontics and orthodontics.

Over time improvements in adhesive dentistry have enabled minimally invasive approaches to improve functional and aesthetic aspects, which has become an asset to the patient.<sup>(16,17)</sup> In those cases of crown fractures where the corresponding tooth fragment is present, its bonding is the best form of restoration since it reinstates, not only the patient's function but also esthetics, with the same parameters as in the past, i.e. with the same shape, the same color, translucency and surface texture of the original tooth.<sup>(8,18,19)</sup> The reattachment of a dental fragment is a conservative, simpler, less time-consuming and cost-effective technique compared to composite resin or full coverage restorations.<sup>(20,21)</sup> There is no consensus in the literature about the best technique for bonding dental fragments, however the treatment chosen should be the one that gives better fracture resistance, ideally similar to those found in healthy teeth.<sup>(16,22)</sup> In order to increase fracture resistance of the restored tooth, clinicians may employ different techniques, namely, the use of bevels, chamfers, dentin and enamel grooves, as well as different combinations of adhesive systems and composite resins.<sup>(23–25)</sup>

Reis A *et al.*<sup>(22)</sup> reported that a simple reattachment with no further preparation of the fragment or tooth was able to restore only 37.1% of the intact tooth's fracture resistance. In their study, a buccal chamfer promoted a recovery of 60.6% of fracture resistance. Bonding associated with an over contour or the placement of an internal groove nearly restored the intact tooth fracture strength with retrieving values of 97.2% and 90.5%, respectively.

In the study carried out by Badami *et al.*<sup>(26)</sup> the authors concluded that neither the bevelling nor the material used led to an improvement in the fracture resistance of the tooth. They concluded that the resistance of the fractured segment would be directly proportional to the area of adhesion available.

Cavalleri and Zerman<sup>(27)</sup> compared two types of approaches for the treatment of coronal fractures, namely a direct restoration in composite resin and the bonding of the tooth fragment.

AD Loguercio *et al.*<sup>(28)</sup> evaluated the fracture resistance after reinsertion of the fragment using four techniques (bonded only, chamfer, over-contour; internal dentinal groove) and resin composite build-up. For tooth fragment reattachment an adhesive system with a dual cure resin

cement was used. The over-contour technique and preparation of internal dentinal groove presented the highest fracture strength recovery. The resin composite buildup provided fracture strength recovery similar to intact teeth.

For the reinsertion of fragment in anterior fractured teeth, based on the study of Bhargava et al. it was concluded that the use of nano-composites and the preparation of chamfers in the fracture line will be a useful combination and the chamfer preparation will allow an increase in surface area for application of the material.<sup>(29,30)</sup>

Nevertheless, Chazine M *et al.*<sup>(31)</sup> and Bruschi-Alonso RC *et al.*<sup>(8)</sup> claimed that the materials chosen did not influence the result. Bruschi-Alonso RC *et al.*<sup>(8)</sup> concluded that regardless of the technique or material we individually used, we will never be able to achieve fracture resistance of a healthy tooth.

According to the evidence found in Garcia FCP *et al.*<sup>(32)</sup>'s review simple tooth fragment reattachment was the preferred reattachment technique.

There is still no consensus in the literature about the ideal material that should be used to bond fragment and the fractured tooth.<sup>(32)</sup>

Still, there are variations of this same bonding technique, as in the case of previous or late preparation on the remaining tooth and the use of intermediate materials (flowable resin, conventional resin, resin cements, glass ionomer cements).<sup>(18,21)</sup>

The prognosis of DT will vary according to the type of fracture, the need to perform endodontic treatment or not, the presence or absence of fragments and their adaptation to the remaining tooth, affected dental tissue and the amount lost.<sup>(16,17)</sup>

There are still very few publications that provide us with information about the procedure for applying the fragment "bonding" materials that we should choose to ensure the dentin bond strength. The aim of this study was to compare the microtensile bond strength of three different groups at the dentin-composite interface and evaluate failure modes, after different cementation protocol done two years ago.

The null hypothesis (Ho) was that there are no significant differences in the dentin bond strength after tooth reattachment and two years water aging between different cementation protocols.

## **3.Materials and Methods**

#### **Specimen preparation**

In this experimental study fifteen extracted permanent third molars without structural defects, cracks or incipient lesions were selected. The teeth were disinfected in 0.5% chloramine T and used within 6 months after extraction. All the extracted teeth were cleaned with ultrasonic scaler. The occlusal surfaces were cut perpendicularly to the long axis of the tooth (Accutom 5, Struers, Ballerup, Denmark), under water-cooling, thereby exposing a flat dentin surface.

The prepared occlusal surfaces and corresponding coronal fragment were carefully observed to confirm the absence of residual enamel or other defects in dentin surfaces using a stereomicroscope (Nikon® SMZ 1500, Tokyo, Japan) at 20x.

#### Bonding and restorative procedures

The teeth were randomly divided into three groups, according to the bonding protocols for tooth reattachment. The materials used are depicted in table 1. For all samples both surfaces were etched with 36% phosphoric acid for 15 seconds, then rinsed for 15 seconds and air dried for 5 seconds. Afterwards, a universal adhesive system (Prime&Bond<sup>™</sup> Active, Dentsply DeTrey; LOT: 1702000441; VAL: 01-2019) was applied in a rubbing motion for 20 seconds in both surfaces and gently air blown for 5 seconds. At this time the adhesive was not light cured. Following bonding procedure, the coronal fragment was reattached in accordance to one of the following protocols:

Group I: the nanohybrid composite resin Ceram X® (D3, Dentsply DeTrey; LOT: 0080; VAL: 06-2018), was homogeneously spread over one surface with OptraSculpt Pad (Ivoclar Vivadent), the fragment was reattached, and a controlled 30 N force was applied over the fragment for 20 seconds. Light curing was performed during 15 seconds per surface (SPEC 3<sup>™</sup>, Colténe Whaledent, emitting 3000 mW/cm2), making a total of 75 seconds.

Group II: The same protocol of group I was used but ultrasonic vibration (cm4, (Ultra Fusion®; CVDentus) was applied over the reattached fragment as long as the 30 N force was undertaken.

Group III: The same protocol of group I was used but Ceram X was pre-heated at 50°C using

## EASE-IT (Ronvig®, Akura)

After fragment reattachment, the enamel occlusal surface of each tooth was bonded and restored with two 1,5 mm incremental layers of composite resin. Each layer was light-cured for 10 seconds with a LED light-curing unit (SPEC 3, Colténe, Altstätten, Switzerland), followed by an extra polymerization time of 60 seconds (20 seconds on the occlusal surface and 10 seconds on the other surfaces). The specimens were then stored at 100% humidity at 37°C for seven days (Heraeus BK 6160, Kelvitron<sup>®</sup> Kp, Wehrheim, Germany).

Materials	Manufacturer	Composition		
Prime&Bond Active (P&Ba)	Dentsply DeTrey; Konstanz, Germany	Bisacrylamine 1 (25-50%), 10-MDP (10-25%), bisacrylamide 2 (2,5-10%), 4-(dimethylamino) benzonitrite (0,1-1%), dipentaerythritol pentacrylate phosphate (PENTA), propano-2- ol (10-25%), water (20%)		
Ceram X®	Dentsply DeTrey; Konstanz, Germany	<ul> <li><u>Matrix:</u> Methacrylate modified polysiloxane, dimethacylate resin, fluorescent pigment UV stabilizer, stabilizer, camphorquinone, ethyl-4(dimethylamino) benzoate, iron oxide pigments, titanium oxide pigments, aluminium sulfo silicate pigments.</li> <li><u>Filler</u>: Barium- aluminium -borosilicate glass (1.1-1,5μm).</li> <li>Methacrylate functionalized silicon dioxide nano filter(10nm)</li> </ul>		

Table 1- Materials used in bonding and restorative protocols.

## **Cutting method**

After storage, the specimens were cross-sectioned perpendicularly to the adhesive interface with a low-speed cutting saw (Accutom 5, Struers, Ballerup, Denmark), under water cooling at 100 rpm and 0,300 mm/s, to produce dentin sticks with a sectional square area of approximately 1,00 mm<sup>2</sup>. After the first cut in x-axis direction, the free residual space between the slices was filled with light-bodied silicone Aquasil Ultra XLV (Dentsply, DeTrey, Konstanz, Germany). Then a second set of cuts were made in y-axis. Finally, the roots were cut from the crown

approximately 4 mm below the cementoenamel junction releasing the sticks which were then checked under an optical microscope (M300, Leica, Switzerland) at 40-fold magnification in order to exclude samples with defects. All sticks were carefully identified and were then stored in water at 37°C for two years. According to ISO/TS 11405:2015, the medium was replaced every seven days to avoid contamination. <sup>(33)</sup>

#### Microtensile bond strength testing

Two years later, each stick was bonded to a microtensile sample holder with cyanoacrylate adhesive (CE10Flex<sup>®</sup>, Ce Chem Limited, Derbyshire, UK) and then fixed on the microtensile device (Od04-Plus; Odeme Dental Research, Luzerna, Brasil). Specimens were fractured in tensile mode in a universal testing machine (Model AG-I, Shimadzu Corporation, Kyoto, Japan) at a 0,5 mm/min speed and the maximum load was recorded in Newtons and microtensile bond strength was calculated according to the following equation:  $\mu$ TBS = F/A=N/mm<sup>2</sup> = MPa, where F is the load at fracture (N) and A is the bonded area (mm<sup>2</sup>).

The failure mode was analysed under an optical microscope (Leica CLS 150 MR, Switzerland) with a x40 magnification. The fracture pattern was classified as follow: (A) adhesive at the bonding interface; (C) cohesive, if complete failure occurred in dentin and (M) mixed, when failure occurred at both the adhesive interface and cohesively in dentin.

#### **Statistical Analysis**

Statistical analysis was performed with IBM SPSS Statistics 20.0 (SPSS; Chicago, IL, USA). Kruskal-Wallis was used to compare means of microtensile bond strength data between the groups. The chi-square test was used to compare failure modes between the groups. For all analysis the significance level was set at  $\alpha = 0.05$ .

# 4.Results

A total of one hundred and forty-eight (148) specimens were available for microtensile testing. Descriptive statistics and the number of tested specimens is described in Table 2. No statistically significant differences were found among groups (figure 1).

Chi-square test indicated that there were no significant differences in the proportion of failure between the three groups (p=0,149). Distribution of the failure mode is summarized in Table 3. Cohesive and adhesive failures occurred more frequently in all groups.

Table 2: Descriptive statistics of dentin  $\mu$ TBS as mean ± standard deviation and 95% confidence interval (CI) of experimental groups.

Group	Ν	Mean ± SD(MPa)	Min (MPa)	Max (MPa)	95% CI
I	62	21,75±8,66	0,46	34,55	[19,55 ; 23,95]
II	48	20,04 ± 10,59	0,27	33,91	[16,96 ; 23,11]
111	38	23,90±7,36	5,31	33,15	[21,48 ; 26,31]

n: number of specimens; Min: lowest bond strength value; Max: highest bond strength value; 95 % CI: 95% confidence interval

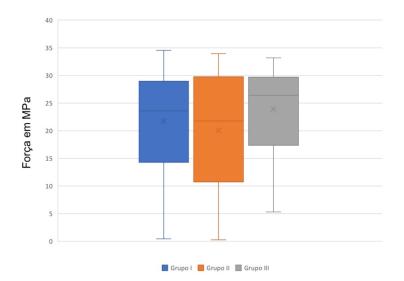


Figure 1: Box-plot (min-[lower quartile-median-upper quartile]-max) of µTBS (in MPa) of the three groups.

Failure Mode	Groups			Total
	I	II	Ш	
Adhesive	16	20	14	50
%	25,80%	41,70%	36,80%	33,80%
Cohesive	38	21	15	74
%	61,30%	43,80%	39,50%	50,00%
Mixed	8	7	9	24
%	12,90%	14,60%	23,70%	16,20%
Total	62	48	38	148

Table 3: Distribution of the failure mode for each group.

## 5. Discussion

Fragment reattachment can be considered a valid treatment option in coronal fractures of anterior teeth. This seems to be a valuable alternative to incremental composite resin restorations in the managing of fractured teeth, as this method is simple, conservative, ensures complete restitution of the tooth's integrity and also provides aesthetics and satisfactory fragment retention.<sup>(18)</sup> Furthermore, conventional composite resin restorations tend to undergo aesthetic loss over time due to discoloration and abrasion process that may occur when exposed to the oral cavity.<sup>(22)</sup>

Bond strength can be measured using macro and micro test set-ups, basically depending upon the bonded size area.<sup>(34)</sup> The characteristic of the bonding substrate plays a major role on the quality of adhesion.<sup>(35)</sup> Microtensile is the most valuable and recognized test set up for dentin bond strength evaluation and has been used for all clinically relevant substrates including caries-affected, caries-infected, sclerotic, deep and bur cut dentin.<sup>(36)</sup> This method allows the measurement of the tensile strength on very small surfaces, with an area around 1 mm<sup>2</sup> and was used in this study.<sup>(37–39)</sup> This allows for a more uniform stress distribution in the prepared samples of each tooth by measuring the bond strength in critical areas with a more reliable correlation with the loss of clinical retention. Nevertheless, until reaching the adhesive interface the load force applied in this test has to cross the tooth substrate and the composite resin. <sup>(40)</sup> This generates stress concentration in these areas, which can justify the frequent cohesive failure observed in the tooth substrate, which can mask the true bond strength.<sup>(41)</sup>

Microtensile bond strength results may be influenced by factors such as storage of bonded specimens in water,<sup>(42)</sup> trimming, diameter of the stick and also the type of sample holder.<sup>(43)</sup> In order to minimize influencing factors for this type of test, in the present study, square non-trimmed samples of 1mm<sup>2</sup>,<sup>(43)</sup> a notched sample holder<sup>(43)</sup> and water storage according to ISO standard indications were preconized.<sup>(33)</sup>

In different studies, intentional fractures were performed on teeth by sectioning them with disks, surgical blades or saws. It is important to note that structural loss and the contact surface can change according to technique and this can lead to an incompletely fit of fragment due to the loss of tooth structure.<sup>(10,22,28,44)</sup> Otherwise, when trauma occurs, fracture occurs following enamel prisms orientation and fragments fit more easily into the remnant tooth tissue.<sup>(22,28,30,31,45)</sup> Reis *et al.*<sup>(30)</sup> established a protocol in order to promote fracture of teeth. They preconize the application of an axial load (at a speed of 0,6mm/min) in a buccal-to-lingual direction after

dividing buccal surface into transversal and longitudinal thirds to standardize the area (point) for the load application. They made perpendicular load application by means of a small steel ball (2mm<sup>2</sup>) introduced at the end of a pin held in the crosshead of the universal testing machine where roots of teeth were confined in a special device. However, using the sectioning technique we can improve a more accurate reproduction in all teeth, simplifying standardization.<sup>(17,46)</sup>

Reis *et al.*<sup>(22)</sup> compared the fracture strength of two different techniques (bonded only and with buccal chamfer) and different material combinations used to reattach tooth fragments. The control group comprises a resin composite build-up. According to the material used, for both study 5 subgroups were considered: adhesive system, light cured luting agent; dual cured luting agent; flowable resin or hybrid resin. After reattachment, teeth were submitted to the same load in order to evaluate fracture strength. All fractures occurred in the bonded interface in all specimens. It was demonstrated that a buccal chamfer preparation achieves higher fracture strength values than those obtained with only reattachment with no further preparation. However, both techniques showed lower values than control group, in which a 45° bevel extending 1mm on the buccal surface was made and after that a composite resin build-up. Between groups there was no statistical differences regarding to the material that was used and so, they concluded that material was less important and didn't influence significantly fracture strength. Other authors showed similar results reporting that material combinations used for reattachment did not influence the bond strength of the fracture tooth.<sup>(31,47)</sup>

Some studies have considered that prime factors for establishing the bond strength between the fragment and tooth remnant are both the material and the technique used in the reattachment process. Pusman *et al.*<sup>(45)</sup> aimed to evaluate and compare the bond strengths of experimentally fractured tooth fragments reattached with different adhesive materials and retentive techniques. Sound teeth were subjected to a fracturing protocol previously described by Reis *et al.*<sup>(30)</sup> Then, fractured teeth were randomly assigned into one of three reattachment protocols: simple reattachment, over contour preparation (2.5-mm coronally and apically to the fracture line) and internal dentin groove (1-mm deep and 1-mm wide). Teeth were subdivided within each group and in each subgroup were used one of the five different adhesive systems in bonding protocol with or without a hybrid resin composite (Z250). Afterwards, restored teeth were subjected to thermal cycling, and subsequently to the same loading protocol used for fracturing intact teeth in the first step. Fracture line corresponded to the adhesive interface. Both the reattachment techniques and the adhesive systems significantly affected the extent of bond strength. Internal dentinal groove showed highest bond strength followed by over

contour technique. Preconizing internal dentinal groove, the adhesives Clearfil S<sup>3</sup> Bond and Prime&Bond NT both associated with an intermediate layer of composite resin Filtek Z250 achieved the highest bond strengths. Regardless of the adhesive employed, simple reattachment of fragments with an intermediate resin composite layer significantly improved bond strength. Other authors found similar results.<sup>(48,49)</sup> Demarco *et al.*<sup>(49)</sup> evaluated two reattachment techniques (bevel preparation and no bevel preparation) reattached with (a dual-cured resin cement RelyX ARC; a chemically cured composite Bisfil 2B, a light-cured composite Z250 and a one-bottle adhesive Single Bond. Highest bond values were obtained with the chemically cured composite Bisfil 2B group and then with the light-cured composite Z250 with either bevel or non-bevel. Demarco *et al.*<sup>(49)</sup> study like both studies of Rajput *et al.*<sup>(48)</sup> and Pusman *et al.*<sup>(45)</sup> disclosed significant influence for materials and cavity designs.

To the best of the author's knowledge there are no studies about tooth fragment reattachment techniques using pre-heated composite or applying ultrasonic vibration, which make difficult the comparison of our data. In the present study, pre-heating of resin composite to 50°C and ultrasonic vibration during cementation protocol did not have any significant effect on the microtensile bond strength after two years water aging. Therefore, the null hypothesis was not rejected.

It has been considered that pre-heating resin composites enhance dentin bond strength. Davari *et al.*<sup>(50)</sup> and Pappacchini *et al.*<sup>(51)</sup> evaluated the effect of pre-heating composites at 4°C, 23°C or 37°C temperatures on the microtensile dentin bond strength. Both studies concluded that pre-heating composites at higher temperature raised dentin bond strength. Davari *et al.*<sup>(50)</sup> demonstrated that Filtek P60 achieved highest bond values when pre-heated due to optimal viscosity and flowability resulting in deeper penetration of resin into the micro-retentive areas. Otherwise, Pappacchini *et al.*<sup>(51)</sup> used a preheated flowable composite as an intermediate agent record the highest microtensile bond strength. Also, interfacial quality was improved by raising the resin temperature. Cantoro *et al.*<sup>(52)</sup> aimed to assess whether the pre-cure temperatures of resin cements influenced significantly the bonding to dentin. Different pre-curing temperatures were evaluated 4°C, 24°C, 37°C or 60°C. They concluded that temperatures over 50°C (60°C) achieved highest dentin bond strength.

Moss *et al.*<sup>(53)</sup> showed that elapsing two minutes after removing the heated resin at 60° C from the heating device the temperature drops about 50%. Therefore, it is advisable to clinically manage pre-heated composites quickly.

17

In our study, although without statistical differences between groups, the pre-heated composite resin group demonstrated the higher dentin bond strength. Daronch and Rueggeberg<sup>(54)</sup> and Wagner *et al.*<sup>(55)</sup> also reported that higher molecular mobility and reduced viscosity due to increased temperature are correlated with the decrease in film thickness of composite resin. It may be clinically advantageous because it can help to achieve a better adaptation to the cavity wall thereby reducing the risks of incorporating interfacial voids within the restorations and also decreasing microleakage. Therefore, pre-heated resin composites may be recommended in order to decrease film thickness, at least.

An important advantage of sonic application in dental practice is that it does not require any calibration procedure and it is probably less sensitive to operator's experience. This alternative is also easy to implement in clinical procedures and does not add more clinical steps to the bonding protocol.<sup>(56)</sup> The purpose of using the ultrasound application is also to decrease cement materials thickness.

In dental practice the frequency of the sonic oscillating instruments ranges from 1000 to 6000 Hz while ultrasonic frequencies ranges from 20,000 to 40,000 Hz.<sup>(56)</sup> Serrano *et al.*<sup>(56)</sup>'s study aimed to evaluate the effect of different sonic application modes on the immediate and 6-month resin–dentin bond strength. Three one-step self-etch adhesives (One Coat 7.0 ; Clearfil S<sup>3</sup> Bond; FuturaBond NR) were applied on a flat superficial dentin surface under manual mode (using micro brush adhesive application performed as per manufacturers' directions) or sonic vibration ( where the micro brush was firstly attached to the prototype of the sonic device Smart (FGM) at a frequency of 170 Hz). FGM has five different oscillating frequencies (144.5, 150, 170, 223.5, and 167.5 Hz) and the middle frequency was used. Crowns were after restored with composite resin build up. They concluded that the use of a sonic device with an oscillating frequency of 170 Hz improved the immediate resin-dentin microtensile bond strength and delayed the degradation of one one-step self-etching adhesive.

Other studies reported that ultrasonic application mode affected bond strength of self-etching adhesives. Bagis B *et al.*<sup>(57)</sup> studied the effect of high frequency ultrasonic agitation (1 MHz) on the microtensile bond strengths of different self-etching adhesives, Clearfil S<sup>3</sup> Bond, G-Bond, and Futurabond NR. They noticed that ultrasonic agitation effect (through an ultrasonic scaler) positively influenced bond strength. Cantoro *et al.*<sup>(58)</sup> study aimed to evaluate the influence of the cement manipulation and ultrasonic application on the bonding potential of self-adhesive resin cements to dentin by microtensile bond strength testing. Class II inlay were prepared using the nanohybrid resin composite Gradia Forte and the luting agents were the following self-adhesive

resin cements: G-Cem (Automix and Capsule), RelyX Unicem (Cliker and Aplicap). Luting process was made under a static seating pressure (1kg by means of a plunger loaded with a box of lead pellets) or under vibration (with an ultrasonic tip provided with a rubber cap mounted on an ultrasonic handpiece (SONICflex, KaVO, Biberach, Germany) at medium power according to the manufacturer's recommendation). All the specimens failed adhesively at the cement–dentin interface. They concluded, that dentin bond strength was increased following ultrasonic vibration of the inlay.

Serrano *et al.*<sup>(59)</sup> study also compared manual and sonic vibration application modes in terms of microtensile bond strength of a self-etching All Bond SE (Bisco) applied as a one- or a twostep adhesive. However, contrarily to previous results<sup>(56)</sup>, the authors have shown that sonic application was not able to increase the microtensile bond strength values.

The ultrasonic application in the reattachment tooth fragments may require the use of a stabilized tooth fragment guide due to the fact that the tooth fragment with vibration may lose references and position and consequently have a harmful effect on bonding protocol.

# 6. Conclusion

Within the limitations of this in vitro study, it can be concluded that:

All bonding and cementation techniques studied can be used for tooth fragment reattachment. After two years of direct water aging no significant differences could be found between corresponding microtensile values.

Preheating composite resin for luting procedures may not improve microtensile bond strength but could be advantageous since it diminishes material viscosity possibly improving restoration setting and film thickness.

# 7.Acknowledgements

Thanks to my advisor, Doctor Alexandra Vinagre and to my co-advisor, Prof. Dr João Carlos Ramos, for all the support and sharing of knowledge, not only during the elaboration of this thesis, but throughout my entire academic career, having always been, a reference for me as teachers and health professionals.

To Doctor Ana Messias for the precious help in the statistical data treatment.

To Doctor Rui Isidro Falacho, for support and sharing of knowledge during this work and all academic career.

A special thanks to my parents and my sister for all support and love my entire life and for all understanding and help on hard times.

To all my friends for all motivation and help.

This work was carried out using the means provided by the Laboratory of mechanical tests and preparation of samples of Dental Medicine area.

# 8.References

- de Paula Barros JN, de Araújo TAA, Soares TRC, Lenzi MM, de Andrade Risso P, Fidalgo TK da S, et al. Profiles of Trauma in Primary and Permanent Teeth of Children and Adolescents. J Clin Pediatr Dent. 2018;
- Hegde R, Agrawal G. Prevalence of Traumatic Dental Injuries to the Permanent Anterior Teeth among 9- to 14-year-old Schoolchildren of Navi Mumbai (Kharghar-Belapur Region), India. Int J Clin Pediatr Dent. 2017;
- 3. Eltair M, Pitchika V, Standl M, Lang T, Krämer N, Hickel R, et al. Prevalence of traumatic crown injuries in German adolescents. 2019;
- Marcenes W, Al Beiruti N, Tayfour O, Issa S. Epidemiology of traumatic injuries to the permanent incisors of 9-12-year-old schoolchildren in Damascus, Syria. East Mediterr Heal J. 2001;
- 5. Granville-Garcia AF, De Menezes VA, De Lira PIC. Dental trauma and associated factors in Brazilian preschoolers. Dent Traumatol. 2006;
- 6. Bastone EB, Freer TJ, McNamara JR. Epidemiology of dental trauma: A review of the literature. Australian Dental Journal. 2000.
- Vijayaprabha K, Marwah N, Dutta S. A biological approach to crown fracture: Fracture reattachment - A report of two cases. Contemp Clin Dent. 2012;
- Bruschi-Alonso RC, Alonso RCB, Correr GM, Alves MC, Lewgoy HR, Sinhoreti MAC, et al. Reattachment of anterior fractured teeth: Effect of materials and techniques on impact strength. Dent Traumatol. 2010;
- 9. Rappelli G, Massaccesi C, Putignano A. Clinical procedures for the immediate reattachment of a tooth fragment. Dent Traumatol. 2002;
- 10. Ferreira JMS, Fernandes De Andrade EM, Katz CRT, Rosenblatt A. Prevalence of dental trauma in deciduous teeth of Brazilian children. Dent Traumatol. 2009;
- Sabuncuoglu O, Irmak MY. The attention-deficit/hyperactivity disorder model for traumatic dental injuries: a critical review and update of the last 10 years. Dent Traumatol. 2017;33(2):71–6.
- De Oliveira Filho PM, Jorge KO, Paiva PCP, Ferreira EF e., Ramos-Jorge ML, Zarzar PM. The prevalence of dental trauma and its association with illicit drug use among adolescents. Dent Traumatol. 2014;
- 13. Szmidt M, Górski M, Barczak K, Buczkowska-Radlińska J. Direct Resin Composite Restoration of Maxillary Central Incisors with Fractured Tooth Fragment Reattachment:

Case Report. Int J Periodontics Restorative Dent. 2017;

- Andersson L. IADT guidelines for treatment of traumatic dental injuries. Dent Traumatol. 2012;
- Pagliarini A, Rubini D, Rea DMDM, Campese M. Crown fractures: Effectiveness of current Enamel-Dentin adhesives in reattachment of Fractured Fragments. Quintisscence Int. 2000;
- 16. Olsburgh S, Jacoby T, Krejci I. Crown fractures in the permanent dentition: Pulpal and restorative considerations. Dental Traumatology. 2002.
- DiAngelis AJ, Andreasen JO, Ebeleseder KA, Kenny DJ, Trope M, Sigurdsson A, et al. International Association of Dental Traumatology guidelines for the management of traumatic dental injuries: 1. Fractures and luxations of permanent teeth. Dent Traumatol. 2012;
- Andreasen FM, Norèn JG, Andreasen JO, Engelhardtsen S, Lindh-Strömberg U. Longterm survival of fragment bonding in the treatment of fractured crowns : A multicenter clinical study. Pediatr Dent. 1995;
- 19. Murchison DF, Burke FJT, Worthington RB. Incisal edge reattachment: Indications for use and clinical technique. Br Dent J. 1999;
- Author C. Influence of Different Bonding Agents and Composite Resins on Fracture Resistance of Reattached Incisal Tooth Fragment. J Dent Shiraz Univ Med Sci J Dent Shiraz Univ Med Sci. 2014;
- 21. Poubel DLN, Almeida JCF, Dias Ribeiro AP, Maia GB, Martinez JMG, Garcia FCP. Effect of dehydration and rehydration intervals on fracture resistance of reattached tooth fragments using a multimode adhesive. Dent Traumatol. 2017;
- 22. Reis A, Kraul A, Francci C, de Assis TGR, Crivelli DD, Oda M, et al. Re-attachment of anterior fractured teeth: fracture strength using different materials. Oper Dent. 2002;
- 23. Farik B, Munksgaard EC, Andreasen JO. Impact strength of teeth restored by fragmentbonding. Dent Traumatol. 2000;
- 24. MacEdo G V., Diaz PI, Carlos CA, Ritter A V. Reattachment of anterior teeth fragments: A conservative approach. J Esthet Restor Dent. 2008;
- 25. Abdulkhayum A, Munjal S, Babaji P, Chaurasia VR, Munjal S, Lau H, et al. In-vitro evaluation of fracture strength recovery of reattached anterior fractured tooth fragment using different re-attachment techniques. J Clin Diagnostic Res. 2014;
- 26. Badami AA, Dunne SM, Scheer B. An in vitro investigation into the shear bond strengths of two dentine-bonding agents used in the reattachment of incisal edge fragments. Dent

Traumatol. 1995;

- 27. Cavalleri G, Zerman N. Traumatic crown fractures in permanent incisors with immature roots: a follow-up study. Dent Traumatol. 1995;
- 28. Loguercio AD, Mengarda J, Amaral R, Kraul A, Reis A. Effect of fractured or sectioned fragments on the fracture strength of different reattachment techniques. Oper Dent. 2004;
- 29. Bhargava M, Pandit IK, Srivastava N, Gugnani N, Gupta M. An evaluation of various materials and tooth preparation designs used for reattachment of fractured incisors. Dent Traumatol. 2010;
- 30. Reis A, Loguercio AD, Kraul A, Matson E. Reattachment of fractured teeth: A review of literature regarding techniques and materials. Oper Dent. 2004;
- Chazine M, Sedda M, Ounsi HF, Paragliola R, Ferrari M, Grandini S. Evaluation of the fracture resistance of reattached incisal fragments using different materials and techniques. Dent Traumatol. 2011;
- Garcia FCP, Poubel DLN, Almeida JCF, Toledo IP, Poi WR, Guerra ENS, et al. Tooth fragment reattachment techniques-A systematic review. Dent Traumatol. 2018;(December 2017):135–43.
- 33. Iso 11405.
- Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, et al.
   Relationship between bond-strength tests and clinical outcomes. Dental Materials. 2010.
- 35. P. S, Q. Y, J. P, E.M. T, A. M, O. M, et al. Adhesive/Dentin interface: the weak link in the composite restoration. Ann Biomed Eng. 2010;
- 36. Carvalho RM, Manso AP, Geraldeli S, Tay FR, Pashley DH. Durability of bonds and clinical success of adhesive restorations. Dental Materials. 2012.
- 37. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: A review. Dental Materials. 1995.
- Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength - Evaluation of a microtensile bond test. Dent Mater. 1994;
- 39. Yoshida BVMJDMY, Vijay SIMVP, Vanherle KVLPLG, Buonocore M. Adhesion to Enamel and Dentin : Current Status and Future Challenges. Oper Dent. 2003;
- 40. El Zohairy AA, Saber MH, Abdalla AI, Feilzer AJ. Efficacy of microtensile versus microshear bond testing for evaluation of bond strength of dental adhesive systems to enamel. Dent Mater. 2010;
- 41. Heintze SD, Zimmerli B. Relevance of in vitro tests of adhesive and composite dental

materials. A review in 3 parts. Part 3: in vitro tests of adhesive systems. Schweizer Monatsschrift fur Zahnmedizin = Rev Mens suisse d'odonto-stomatologie = Riv Mens Svizz di Odontol e Stomatol. 2011;

- 42. Heintze SD. Clinical relevance of tests on bond strength, microleakage and marginal adaptation. Dent Mater. 2013;
- Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, et al. Critical analysis of the influence of different parameters on the microtensile bond strength of adhesives to dentin. J Adhes Dent. 2008;
- 44. Andreasen JQ, Ravn JJ. Epidemiology of traumatic dental injuries to primary and permanent teeth in a Danish population sample. Int J Oral Surg. 1972;
- 45. Pusman E, Cehreli ZC, Altay N, Unver B, Saracbasi O, Ozgun G. Fracture resistance of tooth fragment reattachment: Effects of different preparation techniques and adhesive materials. Dent Traumatol. 2010;26(1):9–15.
- 46. Andreasen JO AF. Crown fractures. In: Essentials of traumatic injuries to the teeth (ebook). In 1992. p. 21–45.
- 47. Munksgaard EC, Jorgensen EHW, Andreasen JO, Andreasen FM. Enamel-dentin crown fractures bonded with various bonding agents. Dent Traumatol. 1991;
- 48. Rajput A, Ataide I, Lambor R, Monteiro J, Tar M, Wadhawan N. In vitro study comparing fracture strength recovery of teeth restored with three esthetic bonding materials using different techniques. Eur J Esthet Dent. 2010;
- Demarco FF, Fay RM, Pinzon LM, Powers JM. Fracture resistance of re-attached coronal fragments - Influence of different adhesive materials and bevel preparation. Dent Traumatol. 2004;
- Davari A, Daneshkazemi A, Behniafar B, Sheshmani M. Effect of Pre-heating on Microtensile Bond Strength of Composite Resin to Dentin. J Dent (Tehran) [Internet].
   2014;11(5):569–75. Available from: http://www.ncbi.nlm.nih.gov/pubmed/25628684%0Ahttp://www.pubmedcentral.nih.gov/arti clerender.fcgi?artid=PMC4290777
- Papacchini F, Magni E, Radovic I, Mazzitelli C, Monticelli F, Goracci C, et al. Effect of Intermediate Agents and Pre-heating of Repairing Resin on Composite-repair Bonds. Oper Dent. 2008;32(4):363–71.
- Cantoro A, Goracci C, Papacchini F, Mazzitelli C, Fadda GM, Ferrari M. Effect of pre-cure temperature on the bonding potential of self-etch and self-adhesive resin cements. Dent Mater. 2008;

- 53. Moss L, Goes MFDE, Daronch M, Rueggeberg F a, de Goes MF. Clinically relevant issues related to preheating composites. J Esthet Restor Dent. 2006;
- 54. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. J Dent Res. 2005;
- 55. Wagner WC, Aksu MN, Neme AL, Linger JB, Pink FE, Walker S. Effect of Pre-heating Resin Composite on Restoration Microleakage. Oper Dent. 2008;
- 56. Mena-Serrano A, Garcia EJ, Loguercio AD, Reis A. Effect of sonic application mode on the resin-dentin bond strength and nanoleakage of simplified self-etch adhesive. Clin Oral Investig. 2014;
- Bagis B, Turkarslan S, Tezvergil-Mutluay A, Uctasli S, Vallittu PK, Lassila LVJ. Effect of ultrasonic agitation on bond strength of self-etching adhesives to dentin. J Adhes Dent. 2008;
- 58. Cantoro A, Goracci C, Coniglio I, Magni E, Polimeni A, Ferrari M. Influence of ultrasound application on inlays luting with self-adhesive resin cements. Clin Oral Investig. 2011;
- 59. Mena-Serrano A, Costa TRF da, Patzlaff RT, Loguercio AD, Reis A. Effect of sonic application mode on the resin-dentin bond strength and dentin permeability of self-etching systems. J Adhes Dent. 2014;