

## ORIGINAL RESEARCH

# The effect of freezing on the fracture pattern of adhesive on debonding: an *in-vitro* study

Nadija Murati<sup>1</sup>, Iosif Sifakakis<sup>2</sup>, Theodore Eliades<sup>1,\*</sup>

<sup>1</sup>Clinic of Orthodontics and Pediatric Dentistry, Center for Dental Medicine, University of Zurich, 8032 Zurich, Switzerland

<sup>2</sup>Department of Orthodontics, School of Dentistry, National and Kapodistrian University of Athens, 115 27 Athens, Greece

**\*Correspondence**

theodore-eliades@zzm.uzh.ch  
(Theodore Eliades)

**Abstract**

The purpose of this study was to quantitatively evaluate adhesive remnants on the enamel surface following bracket debonding using a freezing element. Thirty-six sound premolars were used in this study. In each case, a bracket was bonded onto each tooth with conventional light-cured composite resin and de-bonded after one week. Freezing of the underlying composite through the bracket was performed immediately before debonding with a portable cryosurgical system ( $-55^{\circ}\text{C}$ ). Specimens were divided into three groups according to the duration of freezing: a control group without freezing was used as a reference and two interventional groups with different durations of freezing (15 or 40 s). Brackets were removed by using debonding pliers to squeeze the wings of the bracket in an occluso-gingival manner. Adhesive remnants on the tooth were then quantitatively evaluated by stereo-microscopy. Pearson's Chi-squared test was used to investigate the relationship between the proportion of remaining resin and the group of teeth. In the control group, 100% of the composite remained on the enamel surface of all specimens. Significantly less adhesive remnants were found in the intervention groups ( $p = 0.001$  for the 15 s group and  $p = 0.043$  for the 40 s group). There was no significant difference between the two interventions ( $p = 0.165$ ) in terms of the proportion of remaining adhesive remnants. Freezing of the bracket and the underlying adhesive resin prior to bracket debonding may favorably alter the behavioral pattern of composite fracture, thus reducing the extent of adhesive remnants on the enamel. Increasing the freezing time from 15 to 40 s did not exert significant effects on adhesive remnants following debonding. Further research now needs to investigate the effect of freezing on the mechanical properties of the adhesive remnants and its *in-vivo* effect on pulp vitality over both short- and long-terms.

**Keywords**

Adhesive remnants; Debonding; Freezing

## 1. Introduction

Orthodontic fixed appliances should not only provide efficient adhesion to the enamel in order to prevent bonding failures during treatment, but should also be easily removed after treatment without damaging the enamel or leaving any residues [1–3]. The continuous development of dental materials over time has improved composite adhesion, however this has inevitably increased the difficulties encountered during the removal of residual resin following the debonding of brackets. Therefore, research has increasingly focussed on adhesive remnants following the debonding of brackets [1, 4]. Previous research has shown that a clinically adequate but non-excessive bond strength ranges from 6 to 8 MPa [3]. However, if the bond strength exceeds 13 MPa, then a patient may experience pulp pain and cracks and/or fractures in the enamel [5, 6].

It is a significant clinical challenge to restore the surface of a tooth after debonding as closely as possible to its pre-

treatment condition without inducing iatrogenic damage [4, 7, 8]. The loss of the external layer of enamel is clinically relevant since exposure of the enamel prism ends favours demineralisation and increases susceptibility to caries [4, 9, 10]. An intact enamel surface is also of crucial importance for aesthetics. The shape and brightness of the anterior teeth are considered fundamental factors in the determination of dental aesthetics. Increased levels of roughness on the tooth surface are directly associated with reduced gloss and brightness [4, 9, 11]. Furthermore, increased surface roughness has also been associated with the formation of plaque and may lead to tooth discoloration [4].

Many different methods of bracket removal have been discussed in the orthodontic literature, including ultrasonic and electrothermal debonding methods. The application of bracket-removing pliers is the most popular technique due to its low cost and availability [12]. Bracket removal with these pliers leaves most of the resin on the enamel surface

and does not cause any iatrogenic damage to the tooth [13]. Some orthodontists base their choice of debonding technique on their own experience and personal preferences [14].

The composites used in orthodontic bonding contain a matrix and a filler. Each of these components has a different coefficient of thermal expansion and therefore the application of a cooling element will inevitably induce thermal stress [15] that may lead to the formation of microscopic cracks. These cracks render the adhesive more brittle and easier to remove because of their effects on strength, dimensional stability and resistance to fatigue. Previous research has not considered the effect of freeze treatment on the bracket and adhesive during debonding. Therefore, the purpose of this study was to quantitatively evaluate adhesive remnants on the enamel surface after bracket debonding using a freezing element. The null hypothesis was that the amount of adhesive remnants on the teeth would be the same after conventional bracket debonding and debonding after freezing of the bracket/composite.

## 2. Materials and methods

Thirty-six sound upper premolars, extracted for orthodontic reasons within the 40 days prior to testing, were used in this study. Premolar extraction patients who met the following inclusion criteria were selected for analysis: (i) absence of tooth cracks/hypoplasia/caries/fillings, (ii) no exposure to chemical agents (*i.e.*, bleaching). The exclusion criteria were (i) teeth with white spots or fluorosis, (ii) patients with any systemic disease or had used any drug that could have affected salivary composition/flow, and (iii) patients who had previously undergone orthodontic treatment. The crowns of the extracted teeth were examined with a stereomicroscope prior to bonding to exclude those with cracked enamel and eligible teeth were labelled for identification purposes.

Prior to bonding, the surface area of each tooth was cleaned manually with the use of cotton rolls and water. The tooth surface was dried, acid etched for 30 s with a 36% phosphoric acid gel (Blue etch; PPH Cerkamed, Stalowa Wola, Poland), rinsed with air/water spray for 10 s and dried again. A thin uniform layer of conventional adhesive was then applied (Transbond XT; 3M Unitek, Monrovia, CA, USA) and brackets (upper premolars) (Gemini; 3M Unitek) were then bonded with a conventional light-cured composite resin (Transbond XT; 3M Unitek), in accordance with the manufacturer's instructions. A light emitting diode (LED) curing light was used to cure each side of the brackets for 3 s (iLED; Woodpecker, Zhonghai, China). The teeth were then stored in distilled water at 37 °C for a week; then, the brackets were debonded.

First, a pilot study was performed, involving 10 teeth (5 teeth exposed to 15 s of freezing and 5 teeth exposed to 40 s of freezing) to determine the amount of adhesive remnants after debonding. Based on these proportions, the sample size of each group was estimated for a given power of 80% and a 5% level of significance [16]. According to power analysis, 10 teeth were required in each intervention group; however, we decided to include 12 specimens in each group. Thus, 36 tooth specimens were separated into three groups (12 teeth per group): a control group without freezing was used as a reference and two intervention groups depending on the duration of freezing

(15 or 40 s). Freezing of the underlying composite through the bracket was performed immediately prior to debonding with a portable cryosurgical system (Histofreezer; Gima, Gessate, Italy). The 5 mm medium sponge applicators were used to serve as a reservoir for the cryogen; these reached a working temperature of -55 °C. The applicators were pressed on the bonded bracket and were soft and wide enough to cover the whole surface of the upper bracket. A new applicator was used for every bracket. Immediately after freezing, the brackets were removed with a debonding plier (Direct bond removing orthodontic plier; Ormco, Brea, CA, USA); the plier was used to apply compressive forces (in the occluso-gingival direction) on the bracket wings; this was achieved by using the plier to squeeze the wings of the bracket. All bonding and debonding procedures were performed by the same orthodontist.

The mode of fracture was investigated in all specimens by stereomicroscopy (Leica M80; Leica Microsystems, Wetzlar, Hesse, Germany) at 25× original magnification. Images were stored in a personal computer running Leica Application Suite version 3.8 (Leica Microsystems, Heerbrugg, Switzerland). In order to characterize the fracture surfaces in greater detail, the samples were investigated by a multi-focus microscope (Leica DM 400M; Leica Microsystems, Heerbrugg, Switzerland). Images were taken at 50× nominal magnification employing multi focus operation with a 400 μm traveling distance on the z axis; we acquired 35 images with approximately 12 μm between successive images. Final images were reconstructed by Leica Application Suite version 3.8 (Leica Microsystems, Heerbrugg, Switzerland). Percentage values were obtained by dividing the surface area with adhesive remnants by the surface area of the bracket base ("Adhesive Remnant Index (ARI)") [17]. Intra-observer error was calculated by re-examining 10 teeth from the intervention groups after two weeks. The investigator who performed the spectroscopic analysis was blinded to experimental groupings (TE).

Statistical analysis was performed using STATA version 13.1 (Stata; College Station, TX, USA). Pearson's Chi-squared test of independence was applied to identify a potential relationship between groups of teeth and the percentage of tooth resin remaining on each tooth after cooling. This test was selected because both variables were categorical. The percentage of remaining tooth resin was categorized into five categories for the three groups of teeth. A  $p < 0.05$  was considered to be statistically significant.

## 3. Results

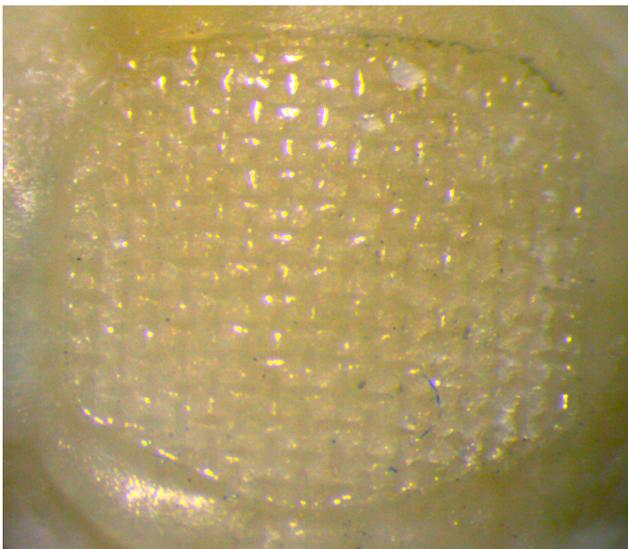
Analysis showed that the intra-observer agreement was almost perfect ( $\kappa = 0.960$ ). The ARI scores are depicted in Table 1. All specimens in the control group retained 100% of the composite on the enamel surface (ARI score = 1) (Fig. 1). In addition, 25% of the teeth in the 15 s group and 58% of the teeth in the 40 s group, also presented with an ARI score of 1 (100% of the composite remained on the enamel).

In the 15 s group, half of the specimens demonstrated an ARI score of 4 (25–49% of composite remaining on the enamel) (Fig. 2), 25% of the teeth had an ARI score of 2 (75–99% of composite remaining on the enamel) and the remaining 25% of the teeth had an ARI score of 1. In the 40 s group, the

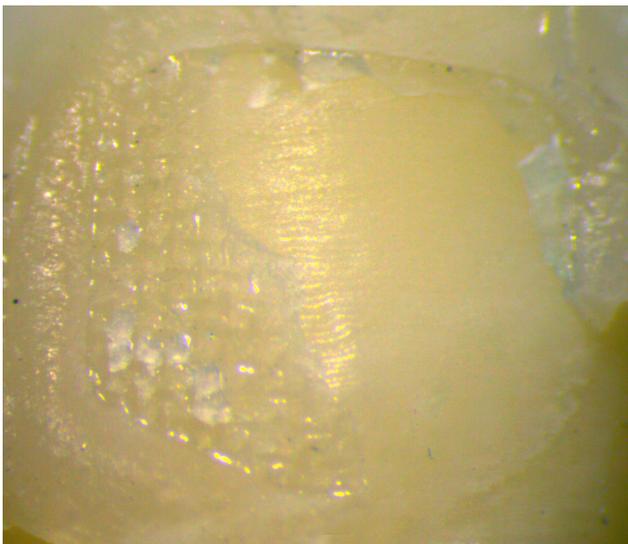
**TABLE 1. Number (and percentage) of teeth by group and ARI scores.**

ARI score (No)	% of remaining tooth adhesive	Control group	Group 1 (15 s cooling)	Group 2 (40 s cooling)
1	100%	12 (100%)	3 (25%)	7 (58%)
	<100%	0 (0%)	9 (75%)	5 (42%)
2	75–99%	0 (0%)	3 (25%)	3 (25%)
3	50–74%	0 (0%)	0 (0%)	0 (0%)
4	25–49%	0 (0%)	6 (50%)	2 (17%)
5	<25%	0 (0%)	0 (0%)	0 (0%)

ARI: Adhesive Remnant Index.



**FIGURE 1. Representative multi-focus image of adhesive remnants (100%) on a typical tooth in the control group.**



**FIGURE 2. Representative multi-focus image of adhesive remnants (25–49%) on a typical tooth in the 15-s cooling time group.**

majority of teeth demonstrated an ARI score of 1; however, in 17% of the teeth in this group, the percentage of remaining adhesive on the enamel was 25–49% (an ARI score of 4); in the remaining 25% of specimens, 75–99% of the composite remained on the enamel (an ARI score of 2). None of the teeth in the three groups had an ARI score of 3 or 5 (50–74% or <25% of adhesive remaining on the enamel, respectively).

A statistically significant difference was detected between the proportion of remaining adhesive between the three groups (Pearson's Chi-squared test with 4 degrees of freedom = 15.6,  $p = 0.004$ ). Further testing was conducted to identify differences between the control group and each of the two intervention groups. Again, statistically significant differences between the control group and the intervention groups were detected (Pearson's Chi-squared test with 2 degrees of freedom = 14.4,  $p = 0.001$  between the control and the first intervention group (15 s cooling time) and Pearson's Chi-squared (2 degrees of freedom) = 6.3,  $p = 0.043$  between the control group and the second intervention group (40 s cooling time)). Pearson's Chi-squared test was also conducted to determine whether the percentage of remaining adhesive was significantly related to intervention group. Analysis found no statistically significant difference between the proportion of remaining adhesive when compared between the two groups (Pearson's Chi-squared (2 degrees of freedom) = 3.6,  $p = 0.165$ ).

#### 4. Discussion

Analysis of the present data led to rejection of the null hypothesis; in other words, fewer adhesive remnants were left on the enamel after freezing of the bracket/composite. Bracket debonding after orthodontic treatment remains a significant clinical challenge because iatrogenic damage may occur, thus leading to a reduction in enamel. Various methods for bracket debonding are described in the orthodontic literature, including the removal of resin and tooth polishing. However, no consensus has been achieved with regards to the most effective and safe method [4, 14]. The required duration of adhesive removal/tooth polishing, as well as the quantity of composite residues, and the loss of hard tissue in teeth, may vary between different methods [18]. Irrespective of the method used, grooves appear on the enamel following bracket debonding and the removal of composite resin [9, 10]. Ideally, as little adhesive residue as possible should be left on the enamel as

this will reduce the removal time and aerosol load required.

In the present study, we quantitatively evaluated adhesive remnants on tooth enamel after conditioning of the bonded bracket/adhesive with a portable cryosurgical system. This is the first time that such an approach has been described in the orthodontic literature. Our results indicate that this type of temporary freezing weakens the resin-enamel-interface and influences the fracture pattern of the adhesive during bracket debonding. At low temperatures, the composite becomes brittle since the phase of semi-crystalline plastics freezes and the molecular chains barely move. This process is referred to as embrittlement. Once the molecules are in the hard-elastic glass state, there are two possibilities: either they may withstand the pressure and remain in the position they are already in, or they can be separated by the pressure and the semi-crystalline plastic breaks apart [19]. Consequently, microscopic cracks are produced and the material becomes brittle. Cracks in the composite can affect strength, dimensional stability and resistance to fatigue resistance, thus facilitating debonding.

In the present study, we used stainless-steel brackets since this is still the most commonly used bracket type by orthodontists. Although ceramic brackets deliver enhanced aesthetic value, their larger dimensions and adjusted heat conductivity contribute to the increased difficulty in their removal from teeth [5]. In the present study, we also used bracket-removing pliers. The most popular combination for bracket/adhesive removal is bracket-removing pliers followed by grinding of the remaining adhesive with a low-speed tungsten carbide bur [10, 20]. Although composite and fiber glass burs can produce a smoother enamel surface when compared to tungsten carbide, the latter remains the most commonly used method due to its increased speed and effectiveness [14].

Other debonding methods can elevate the in-site temperature to deform the adhesive-bracket interface, resulting in the gentler removal of the bracket from the enamel surface. In the electrothermal debonding method (ETD), a heated tip of a battery-driven device is inserted in the slot of the ceramic bracket. The average debonding time with this device has been reported to be 3 s [21, 22]. However, when using this technique, high levels of thermal energy are transferred to the pulp, thus causing vasodilation and increased blood flow. These alterations increase intra-pulpal pressure, deform the cell membranes of free nerve endings, and therefore activate nerve endings in the dentinal tubules. This process is known as the hydrodynamic effect [23, 24]. A higher blood inflow than outflow results in pulpal hyperemia and engorgement; however, this effect can be potentially reversed [21]. An increase of the intra-pulpal temperature by 5.5 °C may cause pulp necrosis in 15% of cases [25]. Consequently, in order to avoid nociceptive stimulation, the maximum temperature increase during debonding/adhesive removal should not exceed 5.5 °C. A reduction of temperature may also provoke hydrodynamic effects. When a freezing element is applied to a tooth, fluid contracts, blood flow decreases and an outwards movement of dentinal fluid is induced. These events may lead to anoxia and the cessation of function in A-type fibers. Furthermore, a sharp and more rapid pain is induced when compared to the pain experienced by patients receiving hot stimulation [23, 26].

Another method that may increase pulp temperature is laser

irradiation. The application of a laser softens the adhesive resin and thus reduces the bond strength. As a result, a laser can facilitate bracket debonding, thus reducing enamel tear-outs, bracket failures and pain [27]. However, existing results regarding the application of laser for the debonding of brackets are controversial, primarily due to the controversy surrounding potential iatrogenic damage. The use of lasers is known to be associated with an increased temperature in the pulp, which may lead to pulp damage. Such damage can be bypassed, at least to a certain extent, by the application of adequate water cooling [1, 28].

Another technique is the application of an ultrasonic scaler (US); this method is occasionally used for adhesive removal, however there is evidence to indicate that this technique is not appropriate [29]. This technique applies high frequency vibrations, which are known to disturb the integrity of the complex interfacial adhesive/bracket structure [1]. A recent study compared the US method for enamel clean-up with a tungsten carbide bur and debonding pliers. The former showed the greatest degree of enamel loss when compared to the control method [30].

However, composite reacts to both hot and cold temperatures. Composites consist of three distinct phases: a polymerizable resin, a viscoelastic material, as well as the inorganic filler and the filler-resin interface. Each phase adopts its own role in dictating material properties [31]. Fillers have a different linear coefficient of thermal expansion than the matrix; this is of great importance, since it influences the dimensions of the adhesive interface [32]. With a declining temperature, the resin matrix shrinks to a greater extent than the filler particles. In contrast, a rise in temperature results in the resin matrix expanding to a greater extent than the filler particles. These differences in thermal expansion can lead to internal stresses [33]. According to the present findings, a longer freezing time of 40 s did not reduce the ARI scores further after bracket debonding when compared with a freezing time of 15 s.

An advantage of freezing the bracket/adhesive immediately before debonding would be the faster and more efficient removal of adhesive. The findings of the present study demonstrated a clear trend in that there was a reduction in the amount of residual resin on the teeth following freeze treatment. This phenomenon may also reduce the amount of time required to remove remnants of adhesive. Furthermore, less aerosols will be produced during this process. Unfortunately, we found that resin residuals still remained on the teeth even after freeze treatment and therefore needed to be polished, a procedure that produces aerosols and affects the surface of the enamel. Further research now needs to investigate the effect of freezing on the mechanical properties of the adhesive remnants and its *in-vivo* effect on pulp vitality over both short- and long terms.

## 5. Limitations

This study was limited by the fact that the orthodontist who performed the bonding and debonding was not blinded to experimental groupings. However, every effort was made to standardize these procedures. Additionally, fluoride pre-treatment before extraction may have affected the fracture pat-

tern of the adhesive during bracket debonding, since it interferes with the acid etching effects on enamel and reduces bond strength.

## 6. Conclusions

Freezing of the bracket and the underlying adhesive resin with a portable cryosurgical system before debonding led to a reduction in the remnants of adhesive on the enamel. Furthermore, increasing the freezing time from 15 to 40 s had no significant effect on ARI scores.

## AVAILABILITY OF DATA AND MATERIALS

The data are contained within this article.

## AUTHOR CONTRIBUTIONS

TE and IS—designed the research study, contributed to editorial changes in the manuscript. NM, IS and TE—performed the research and analyzed the data. NM, IS and TE—wrote the manuscript. All authors read and approved the final manuscript.

## ETHICS APPROVAL AND CONSENT TO PARTICIPATE

The protocol for this study was approved by the Ethics Committee of the School of Dentistry at the National and Kapodistrian University of Athens, Greece (protocol number: 122808/15.02.2022).

## ACKNOWLEDGMENT

We are grateful to Spiros Zinelis (Department of Biomaterials, School of Dentistry, National and Kapodistrian University of Athens, Greece) for providing the equipment for the analysis.

## FUNDING

This research received no external funding.

## CONFLICT OF INTEREST

The authors declare no conflict of interest.

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**How to cite this article:** Nadija Murati, Iosif Sifakakis, Theodore Eliades. The effect of freezing on the fracture pattern of adhesive on debonding: an *in-vitro* study. *Journal of Clinical Pediatric Dentistry*. 2024; 48(3): 31-36. doi: 10.22514/jocpd.2024.056.