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The Influence of Resin Infiltration Pretreatment on Orthodontic Bonding to Demineralized Human Enamel

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Abstract: Prior research reveal that low-viscosity resin is able to significantly penetrate initial caries lesions, which leads to their stabilization. The objective of the present report is to assess the shear bond strength (SBS) of orthodontic brackets bonded with different adhesives to demineralized enamel treated with a low-viscosity resin infiltrant. It also aims to compare the achieved bond strengths to those achieved in relation to sound enamel (SE). A total of 48 newly extracted third molars were collected, distributed in four groups (n=12), covered with a nail varnish, with 4 x 4 mm of uncoated area, immersed in Buskes demineralizing solution (14 days, 37 °C) or remained untreated. Group I: SE + Transbond XT; Group II: demineralized enamel (DE) + ICON + Transbond XT; Group III: DE + ICON + Scotchbond Universal; Group IV: DE + ICON + Assure PLUS. SBS was quantified in megapascals (MPa) and statistically analyzed (ANOVA, $p \leq 0.05$). The mode of failure was assessed microscopically (10 x magnification). The highest SBS detected was in Group IV, and the difference was statistically significant (F = 14.37; $p = 0.000$). Treatment with a resin infiltrant on DE does not impair the shear bond strength when compared to SE, although it does produce a significantly higher strength when combined with Assure PLUS.

Keywords: enamel; demineralization; caries infiltration; ICON; shear bond strength; in vitro

1. Introduction

White-colored alterations may appear as a result of pre- or post-eruptive disorder [1]. The pre-eruptive lesions of importance in this regard result from fluorosis, traumatic hypomineralization and molar–incisor hypomineralization (MIH). Post-eruptive alterations take place when the demineralization process is stronger than the remineralization process and they are usually addressed as white spot lesions (WSLs) [1]. The initial development of WSLs is present as a mineral loss in the bulk of the enamel, while the lesion surface remains mostly intact. Later, the surface layer of the lesion becomes partially porous, which can result in a loss of transparency [2]. The fixed orthodontic appliances may enhance plaque accumulation and lead to the progress of both demineralization and caries lesions, especially in patients with inadequate oral hygiene [3–5]. Further, WSLs can be seen after just four weeks of a fixed orthodontic appliance being fitted [6]. Additionally, the development of WSLs is not only related to

orthodontic treatment, since such changes can also be seen before treatment has started and after it has finished.

The overall prevalence of WSLs ranges from 2–96% depending on the assessment procedure of assessment of the lesions, whether or not any such defects were present before treatment and whether or not fluoride supplements were used during treatment [7]. Moreover, a recent study showed that WSLs were 2.5 times more common in the maxillary arch than in the mandibular arch [4].

The altered enamel structure of teeth with initial caries lesions may reduce the shear bond strength (SBS) between orthodontic brackets and the enamel surface at the beginning of orthodontic treatment, as well as during treatment if the rebonding of the brackets is required [8,9]. In fact, the brackets rebonding is a regular procedure during orthodontic treatment [10] with the most common reasons for this intervention being bond failures on the part of the brackets and the need to reposition the brackets [11]. If bracket bonding is to be performed on demineralized enamel, it is advisable to stabilize and protect the demineralized enamel lesion prior to the reapplication of the brackets [12]. In general, there are two main types of interventions available for the treatment of WSLs. The first type involves non-invasive methods, like the application of topical high-concentration fluorides [13,14] or casein phosphopeptide–amorphous calcium phosphate (CPP–ACP). The second type involves more invasive techniques, such as the hydrochloric acid-pumice microabrasion technique [8] or caries infiltration with resin [15]. Previous reports, demonstrated that low-viscosity resin is significantly able to penetrate initial caries lesions, which leads to their stabilization [16,17]. The optical appearance of WSLs was also found to be improved by resin infiltration, with the achieved concealment being shown to remain stable in one-year interval in vivo [18].

Observing limits of this technique it can be described as sensitive [19] and this can be a problem in vivo. In addition, some cases of WSL treated with the resin infiltrant experience no camouflage effect [20]. The explanation for this outcome can be the depth and activity of the lesion, the infiltration is not complete when the lesion is deeper or inactive and has a thicker surface [20].

The aim of the present study was to assess the SBS of orthodontic brackets bonded with different adhesives to demineralized human enamel that had been treated with a low-viscosity resin infiltrant. It also aimed to compare the achieved bond strengths to those achieved in relation to sound enamel.

The null hypotheses were: 1) treatment with caries infiltrant on demineralized enamel does not impair the SBSs of different adhesives in comparison to the bond strengths seen in relation to sound enamel and 2) the shear bond strengths recorded in demineralized specimens treated with caries infiltrant are not significantly enhanced by substrate pretreatment with a universal adhesive.

2. Materials and Methods

The materials used in this study are displayed in Table 1.

Table 1. Materials used in the present study.

Name	Composition	Manufacturer
ICON	Icon-Etch—hydrochloric acid, pyrogenic silicic acid, surface-active substances Icon-Dry—99% ethanol Icon-Infiltrant—TEG-DMA-based resin matrix, initiators, additives	DMG, Hamburg, Germany
Transbond XT primer	Bis-GMA, TEGDMA, dimethylamino-benzene ethanol, DL-camphorquinone, hydrochinone	3M Unitek, Monrovia, CA, USA
Scotchbond Universal	10-MDP phosphate monomer, dimethacrylate resins, bis-GMA, Viterbond copolymer, filler, ethanol, water, initiators, silane, HEMA	3M ESPE, St. Paul, MN, USA
Assure PLUS	Bis-GMA, ethanol, 10-MDP phosphate monomer	Reliance Orthodontic Products, Itasca, IL, USA
Transbond XT Light Cure Adhesive	Silane-treated quartz, bis-GMA, bis-EMA, silane-treated silica, diphenyliodonium hexa-fluorophosphate	3M Unitek, Monrovia, CA, USA

Bis-GMA: bisphenol-A-glycidyldimethacrylate; TEG-DMA: triethyleneglycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-A-dimethacrylate, HEMA: 2- hydroxyethyl methacrylate.

2.1. Specimen Preparation

A sum of 48 newly extracted third molars was collected, cleansed and set aside in saline solution for no more than three months. The teeth were intact, and they had been extracted for reasons unrelated to the objectives of this study. All the patients provided written informed consent. The teeth were randomly allocated into four groups (n = 12). In Group I (control), enamel demineralization procedure was not carried out. All the teeth in Groups II, III and IV were coated with nail varnish, with a 4x4-mm uncoated area on the buccal surface being left exposed to the demineralization procedure, which was performed according to the recommendations of Buskes et al. [21] (14 days, 37 °C).

2.2. Resin Infiltration Procedure

Following the demineralization procedure, for the teeth in Groups II, III and IV, a resin infiltration procedure (ICON, DMG, Hamburg, Germany) was performed as the manufacturer recommended. The application of resin infiltrant was carried out on the demineralized surface of the enamel. Icon-Etch was applied for 120 s, rinsed with water for 30 s and air-dried. Icon-Dry was then applied for 30 s and again air-dried. In the last step, Icon-Infiltrant was applied for 180 s, light cured for 60 s, re-applied for 60 s and then light-cured for 40 s.

2.3. Bonding Procedure

Stainless steel buccal tubes (3M Victory Series Funneled Second Molar Buccal Tubes, 3M, St. Paul, MN, USA, surface area of 14 mm2) were bonded in the middle of the buccal surface of each tooth so that the long axis of the tube was parallel to that of the tooth. Any excess cement was removed using a probe before the light-curing process. The adhesive resin was polymerized for 20 s from two directions (mesial and occlusal) using a Bluephase light-curing unit (Ivoclar Vivadent, Schaan, Liechtenstein) with a power output of 1100 mW/cm^2. The results were recorded using a Bluephase meter (Ivoclar Vivadent, Schaan, Liechtenstein), which was checked before every bonding. All the specimens were prepared by the same operator.

In Group I (control group), the stainless steel buccal tubes were bonded to intact enamel. The acid etching was performed using 37% phosphoric acid (Ultra-Etch, Ultradent Products, Inc., South Jordan, UT, USA) for 15 s, rinsed for 20 s and then dried for 10 s. The tubes were bonded with Transbond XT Primer (3M Unitek, Monrovia, CA, USA) and Transbond XT Paste (3M Unitek, Monrovia, CA, USA).

In Group II, demineralized enamel that had previously been treated with Icon was treated in the same way and with the same adhesive as the teeth in Group 1.

In Group III, demineralized enamel that had previously been treated with Icon was etched with 37% phosphoric for 15 s, rinsed for 20 s and then dried for 10 s. The tubes were bonded with a universal adhesive, namely Scotchbond Universal (3M ESPE, St. Paul, MN, USA) and Transbond XT Paste.

In Group IV, demineralized enamel that had previously been treated with Icon was etched with 37% phosphoric acid for 15 s, rinsed for 20 s and then dried for 10 s. The tubes were bonded with another universal adhesive, namely Assure PLUS (Reliance Orthodontic Products, Itasca, IL, USA) and Transbond XT Paste.

2.4. Thermocycling

Previous to the SBS testing, the specimens were thermocycled 10,000 times (5–55 °C) with dwell time: 20 s in each bath; transfer time: 10 s).

2.5. Shear Bond Strength Test

The teeth were fixed in cubic plastic molds with cold-curing methacrylate resin (Orthocryl, Dentaurum, Ispringen, Germany). The universal testing machine (Triax Digital 50, Controls S.P.A., Milan, Italy) was used for SBS testing. A shear force was applied through a chisel-shaped loading device at a crosshead speed of 1 mm/min parallel to the specimen's surface. The maximum shear force

necessary to debond each bracket was recorded as a force (N) and then converted into SBS (MPa). This was performed by dividing the force value with the surface area of the bracket base. The surface area of the bracket base was 14.0 mm2, as specified by the manufacturer (3M ESPE).

2.6. Adhesive Remnant Index

The debonded area was examined using a stereomicroscope (Nikon SMZ645, Tokyo, Japan) at 10 x magnification to assess the adhesive remnant index (ARI) [22]. The ARI scores ranged from 0 to 3. Precisely, 0 = no adhesive left on the enamel surface, 1 = less than half of the adhesive, 2 = half or more of the adhesive and 3 = all of the adhesive with the distinct impression of the bracket mesh.

2.7. Statistical Analysis

All the statistical analyses in the present study were carried out by the Statistical Package for the Social Sciences software (IBM SPSS Statistics version 21 for Mac, Chicago, IL, USA). Descriptive statistics, containing the mean, standard deviation and minimum and maximum values of the SBS, were calculated for each group. The normality of distribution was assessed by Kolmogorov–Smirnov test. One-way analysis of variance (ANOVA) and post hoc Tukey's test were used for the between-group comparison. Further, a Kruskal–Wallis test was used to compare the differences in the ARI scores between the groups. The level of significance was set at $P \leq 0.05$.

3. Results

A significantly higher SBS (F = 14.37; $p \leq 0.001$) value was recorded in Group IV, in which the demineralized enamel was pretreated with ICON and the tubes were bonded with Assure PLUS adhesive, when compared to the values in all the other groups (Table 2).

Table 2. Descriptive statistics and shear bond strength (SBS) comparison of four groups.

Group	Number	SBS					SIGNIFICANCE		
								Tukey's Test	
		Mean	Standard Deviation	Minimum	Maximum	ANOVA	Group 2	Group 3	Group 4
I	12	11.33	4.36	3.60	17.15		0.757	0.915	0.000
II	12	12.99	5.51	7.12	24.67	F = 14.37	–	0.369	0.001
III	12	10.23	2.77	5.94	14.36	P = 0.000	–	–	0.000
IV	12	20.28	3.35	16.45	25.62		–	–	–

No statistically significant differences were found among Groups I, II and III. No statistically significant differences were observed among the experimental groups in terms of the ARI values (Table 3).

Table 3. Fracture modes after SBS testing.

Group	Total Number of Cases	ARI				Significance (Kruskal–Wallis Test)
		Score 0 (Number of Cases)	Score 1 (Number of Cases)	Score 2 (Number of Cases)	Score 3 (Number of Cases)	
I	12	6	4	0	2	
II	12	3	7	2	0	P = 0.742
III	12	4	7	1	0	
IV	12	4	4	3	1	

4. Discussion

The aim of this report was to investigate the difference in the SBS to demineralized enamel that had been pretreated with caries infiltrant prior to orthodontic bonding with three adhesives commonly used in relation to fixed orthodontics. The results revealed that the resin infiltration treatment of the demineralized enamel did not impair the SBS comparing it with the SBS on the sound enamel. In fact,

it significantly increased the bond strength of one investigated adhesive (Assure PLUS), while it did not have any impact on the other two investigated adhesives. Therefore, the study's null hypotheses had to be partially rejected (Table 2).

For the scientific purposes of this research, it was necessary to create consistent artificial lesions in order to simulate in vivo caries development. Hence, artificial demineralized lesions were created according to the recommendations of Buskes et al. [21], since it is a widely used and reliable method [6,10,23,24]. The created lesions show uniform dimensions and consistent histological properties [25]. As recommended in other studies, we covered the areas that needed to be preserved from demineralization using an acid-resistant nail varnish [14,25].

The bonding strength of the adhesive system used should be only large enough to resist the forces that arise in the orofacial region [26]. Reynolds [27] suggested the average range of the bond strength to be 5.9–7.8 MPa. The mean SBSs obtained in the present report were all satisfactory for clinical use.

The demineralized samples were treated with an infiltration system prior to one of three different adhesives being used. The etching in the demineralized groups was performed using 15% hydrochloric acid (HCl). The prior studies [28,29] showed that the application of 15% HCl generates erosion of caries lesions nearly completely when compared to 35% orthophosphoric acid, thereby allowing for better resin penetration. The 99% ethanol is applied to eliminate the water that is stored inside the microporosity of the lesion body and, hence, to let the resin to penetrate in the lesion body using capillary forces [30].

A recent study reported that preconditioning using a resin infiltrant increased the SBS of different bonding systems on artificial enamel lesions—probably as a consequence of the deeper penetration of the infiltrant into the body of the lesion when compared to the primer or paste [31]. Monomer formulations containing an increased triethyleneglycol dimethacrylate (TEGDMA), such as ICON, exhibit a high penetration capability [32] and cause the creation of a thick oxygen-inhibited layer [32] and presumably, this leads to the chemical connection of the resin infiltrant to the monomers of the primer [31]. It has been demonstrated that demineralized enamel that had been exposed to a resin infiltrant showed significantly higher debonding forces than an untreated demineralized surface [33]. It is recommended that the bonding of orthodontic elements should be carried out briefly after the resin infiltration [34].

The Transbond XT primer—used in Group 1 and 2—is considered as one of the standard adhesives in orthodontic bonding and has been part of the many studies [12,33–35]. Recent studies have found that the resin infiltrant did not undermine the SBS to demineralized enamel when Transbond XT was used [25,35]. Further, previous studies have shown that the resin infiltrant did not undermine the bonding to sound and demineralized enamel [36] and that it increased the adhesion of the self-etching agent [24]. However, Attin et al. [12] demonstrated that the SBS was found to be significantly reduced on demineralized enamel that had been pretreated with resin infiltrant in comparison to sound enamel, which is opposite to present results. In addition, their results revel higher values than SBS from our study. The explanation of these differences can be in the different methods of the studies as in our study thermocycling was performed in contrary to the above-mentioned study. The regimen used in this report is representing a one year interval [37].

As far as we know, no prior studies have considered the co-application of the Scotchbond and Assure PLUS adhesives to a demineralized resin-infiltrated enamel surface. The MDP monomer (10-Methacryloyloxydecyl dihydrogen phosphate), present in the composition of both Assure PLUS and Scotchbond, enables chemical bonding to the tooth tissues via iconic bonding to the calcium in hydroxyapatite [38]. Moreover, the presence of ethanol enhances the bond to dentin [39]. In the current report, the adherent was a resin base (ICON), meaning that the presence of MDP and ethanol is not considered to be an advantage of Assure PLUS and Scotchbond when compared to Transbond XT, which is reflected in the results showing no significant differences between Groups I, II and III. However, Assure PLUS showed significantly higher SBS values. HEMA (2-Hydroxyethyl methacrylate), the most often used hydrophilic monofunctional monomer, is present in the composition of Scotchbond. HEMA

has high water sorption leading to a reduction of the mechanical properties and the disintegration of the matrix [40]. Considering thermocycling was preformed Scotchbond's composition can be an explanation for the lowest SBS results in all four groups.

Assure PLUS was previously used in a study that compared the SBS to the surface of fluorosed and nonfluorosed teeth and found that, in both groups, significantly higher SBSs were measured when Assure PLUS was used than when Transbond XT was used [23]. These results are in accordance with those recorded in this report. Additionally, Öztoprak et al. [41] demonstrated that Assure PLUS achieved significantly higher SBS values than Transbond XT in blood-contaminated conditions.

The degree of conversion of adhesive could have also affected SBS of the tubes, as it has a direct effect on the chemical, physical and mechanical features of the resin material [42]. A recent study comparing different adhesives used in orthodontics showed no significant difference in the degree of conversion between Scotchbond and Transbond XT [43]. Unfortunately, we found no research investigating the degree of conversion of Assure PLUS, encouraging further investigations in this direction. Further, we found a study that demonstrated that the SBS may not be dependent on the degree of conversion [44].

The results of the ARI score comparisons demonstrated no significant differences between the four tested groups. Most values were zero and one, which indicated a higher frequency of bond failure at the enamel–adhesive interface in all the groups. This agrees with the results of the previously published report [31] and is opposite to the results reported in another research [25].

Our results indicate that the resin infiltration technique undertakes additional clinical investigation in terms of its co-application with different adhesive systems for bracket bonding.

5. Conclusions

Based on our research, there is no evidence that resin infiltrated demineralized enamel surface reduces the SBS of the orthodontic brackets.

Thus, the bonding of the orthodontic elements with the adhesive system on the infiltrated area can be performed without risk of reducing the SBS.

SBS measured in the Assure PLUS group was significantly higher compared to two other groups (Transbond XT and Scotchbond) as well as the control group.

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