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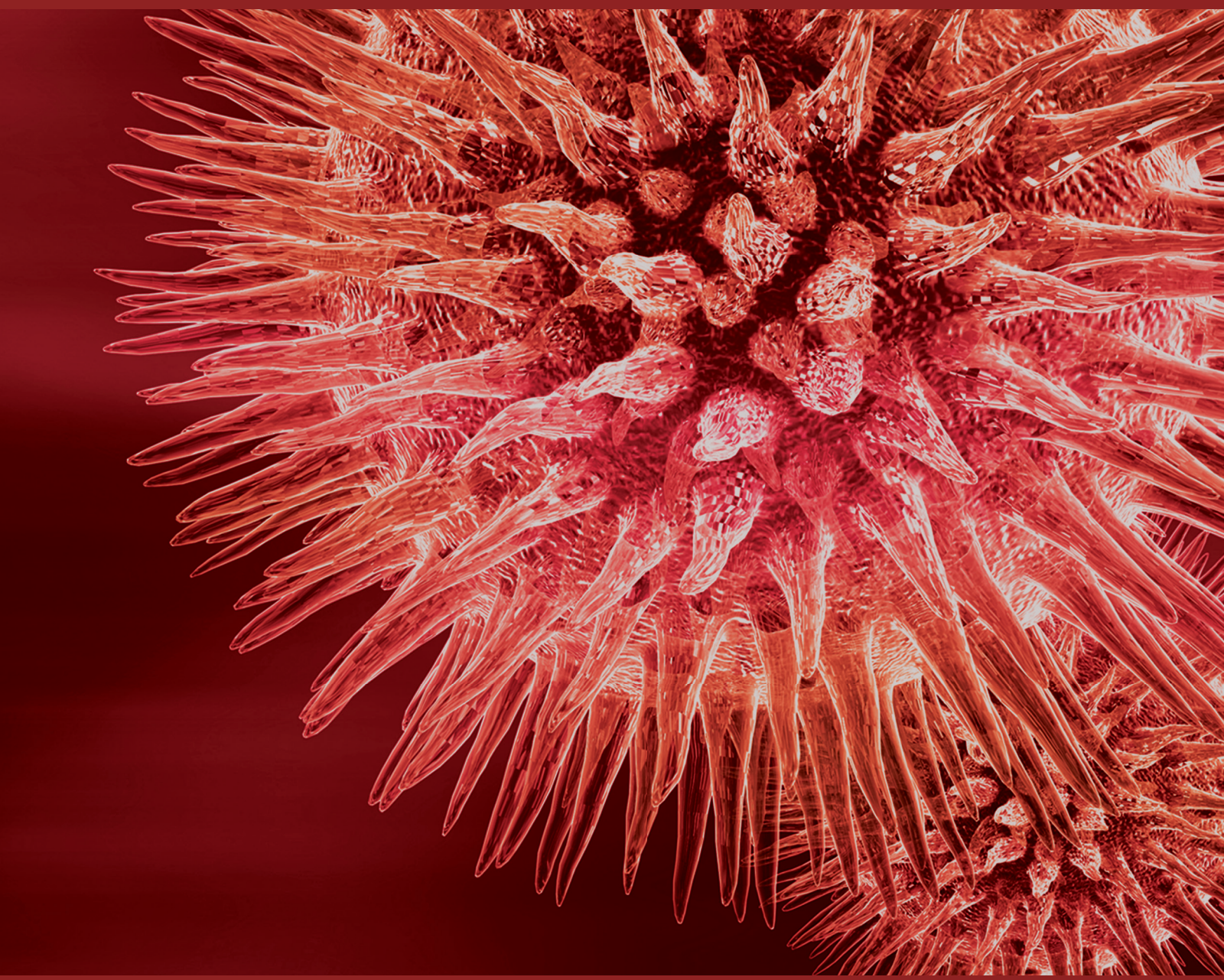
Guest Editors: Andrea Scribante, Rosalia Contreras-Bulnes, Mona A. Montasser, and Pekka K. Vallittu



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Editorial

Orthodontics: Bracket Materials, Adhesives Systems, and Their Bond Strength

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Adhesive interfaces influence greatly clinical success of modern dentistry. Durability of the interface can be determined by using several *in vitro* testing methods. Shear bond strength tests are widely used in dentistry and they are well suitable for testing orthodontic materials bonded to teeth. The first study that analyzed shear bond strength of orthodontic appliances appeared in international literature in the late 1970s [1]. Nowadays, more than one thousand reports have been conducted in order to analyze various factors influencing shear bond strength of orthodontic brackets. Precise interpretation of the shear bond strength test results should, however, take into account other types of stress which are occurring at the interface during testing.

Previous studies that evaluated bond strength analyzed different variables related to adhesive system (composite or resin-modified glass ionomer), bonding surface (enamel, ceramic, or metal), antibacterial agents (added to adhesive system), bracket material (steel, ceramic, or plastic), bracket type (conventional, self-ligating, or lingual), attachment base (with various mesh sizes and shapes), brace mesh or surface pretreatment (such as sandblasting) [2], bracket placement force, enamel conditioning (with etchants or lasers), enamel pretreatment (with protecting or bleaching agents), and enamel contaminants (such as blood or saliva). The effect of any of these factors may differ when rebonding orthodontic brackets [2–10]. Moreover, bonding studies have been applied to test not only orthodontic brackets but also other materials bonded to tooth structure during active or passive orthodontic treatment (such as customized CAD CAM bases, disinclusion buttons, and fiber reinforced composites bars and nets) [11].

During over 35 years of orthodontic bonding studies, a standardized technique has been reached, but many differences in methods among different studies still remain [12]. Due to increased ethical requirements, the human teeth used are usually wisdom teeth or first premolars (extracted for orthodontic reasons). Bovine teeth are collected in slaughterhouses in deciduous or permanent dentition. Tooth selection includes intact buccal enamel and no cracks due to extraction procedure. After extraction, teeth are stored in thymol, water, or artificial saliva, whereas formalin and alcohol are no more used in order to avoid adverse effects on bond strength measurement.

Brackets or jigs are bonded to teeth with an adhesive system and subsequently, or after artificial ageing specimens, are placed in a testing machine with the adhesion surface parallel to shearing force.

Predominantly, a shear force is applied with a steel tip with standardized crosshead speed until adhesive failure. Debonding force is recorded in newtons and then often converted into megapascals, which is the unit of stress at the interface. Special attention needs to be paid to ensure the geometry of the bonding site of the bracket allows calculation of stress. In the case of complex form of the bonding site, it is

correct to report the bonding properties as debonding load. Moreover, enamel and appliance surfaces are analyzed under optic magnification and an Adhesive Remnant Index (ARI) is assigned to give information of the location of the adhesive failure [13]. ARI score is calculated evaluating the amount of adhesive left on tooth and appliance surfaces after debonding. ARI scale usually ranges from 0 to 3 (0: no resin remaining on tooth; 1: less than 50% resin remaining on tooth; 2: more than 50% resin remaining on tooth; 3: 100% resin remaining on tooth).

As it is a standard procedure in biomedical research, statistical analyses are performed with a high enough number of test specimens (i.e., teeth). Descriptive statistics (mean, standard deviation, minimum, median, and maximum values) are calculated for the groups which are compared. The normality of the data can be calculated (e.g., using the Kolmogorov-Smirnov test). Parametric (e.g., ANOVA) or nonparametric (e.g., Kruskal-Wallis) tests are then applied and parametric (e.g., Tukey) or nonparametric (e.g., Mann–Whitney) post hoc tests are used to show differences among various groups. On the other hand, for ARI scores a Chi Squared test is often applied. Significance for all statistical tests is almost always predetermined at $P < 0.05$.

In the literature, there are not clear guidelines about shear force limits, but in fact a good orthodontic biomaterial should allow good adhesion in order to sustain masticatory forces (with a minimum bond strength of 5–10 MPa) [14]. On the other hand, adhesion forces should not be too strong in order to avoid enamel loss after debonding (40–50 MPa) [15]. Therefore, the ideal orthodontic biomaterial should have bonding forces included in the interval of 5–50 MPa, even if these limits are mostly theoretical.

When considering ARI index, even if methods of measurement could influence score assignment results [16], ARI score is nowadays widely used in bonding studies to assess and discuss adhesive left on tooth surface after debonding. Generally, a score of "0" is often related to lower shear bond strength values and is often related to contaminants over enamel that can reduce bond strength. On the other hand, an ARI score of "3" means less risk of enamel fracture after bracket debonding but polishing procedures are longer as more adhesive remains on tooth surface [9]. Therefore, an orthodontic biomaterial should aspire to a mixed adhesion modality (ARI "1" and "2").

In conclusion, bonding studies represent one of the first steps of materials testing and should be followed by *in vivo* clinical studies in order to confirm the *in vitro* results. Therefore, although some criticisms have been stated against bonding studies in orthodontics, bonding tests are still a valid instrument to test new brackets, adhesives, jigs, pad, and other biomaterials bonded to tooth surface.

On the basis of these considerations, the present special issue has been proposed to explore new variables of bonding studies. These new topics have been about the Er:YAG laser-recycled ceramic orthodontic brackets, the transmission of curing light through treated dental tissues, the effect of removal of enamel on rebonding strength of resin composite, the bond strength of different bonding systems on enamel and restorative materials, and the bonding of metal attachments to sandblasted porcelain and zirconia.

The Guest Editors do hope that the present special issue would be interesting for the readers of the journal and wish that the present work could encourage other researchers for future, original, interesting bond strength studies.

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Mona A. Montasser
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Research Article

Bonding of Metal Orthodontic Attachments to Sandblasted Porcelain and Zirconia Surfaces

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This study evaluates tensile bond strength (TBS) of metal orthodontic attachments to sandblasted feldspathic porcelain and zirconia with various bonding protocols. Thirty-six (36) feldspathic and 36 zirconia disc samples were prepared, glazed, embedded in acrylic blocks and sandblasted, and divided into three groups according to one or more of the following treatments: hydrofluoric acid 4% (HF), Porcelain Conditioner silane primer, Reliance Assure® primer, Reliance Assure plus® primer, and Z Prime™ plus zirconia primer. A round traction hook was bonded to each sample. Static tensile bond strength tests were performed in a universal testing machine and adhesive remnant index (ARI) scoring was done using a digital camera. One-way ANOVA and Pearson chi-square tests were used to analyze TBS (MPa) and ARI scores. No statistically significant mean differences were found in TBS among the different bonding protocols for feldspathic and zirconia, p values = 0.369 and 0.944, respectively. No statistically significant distribution of ARI scores was found among the levels of feldspathic, p value = 0.569. However, statistically significant distribution of ARI scores was found among the levels of zirconia, p value = 0.026. The study concluded that silanization following sandblasting resulted in tensile bond strengths comparable to other bonding protocols for feldspathic and zirconia surface.

1. Introduction

Advancements in cosmetic dentistry and orthodontic treatment needs in the United States have led to increasing number of adult patients seeking orthodontic care [1]. Orthodontists must often bond attachments to various dental restorations including those fabricated with porcelain. Feldspathic, leucite, and lithium disilicate are the silica based porcelains while zirconia is a nonsilica based ceramic. Feldspathic porcelain is the most esthetic porcelain due to its high translucency. Zirconia is one of the most popular types of all ceramic restorations today [2]. For the different types of porcelains, literature suggests a wide range of surface treatments that include sandblasting, hydrofluoric acid treatment, silane application, or a combination of them. For feldspathic and lithium disilicate-based porcelain the combination of sandblasting and silane has shown highest shear bond strength values in comparison to sandblasted only samples [3, 4]. For surface treatment of zirconia, application of primer containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) after air abrasion has been recommended [5–7]. The use of ceramic primer along with self-adhesive resin composite cement has shown a positive effect on shear bond strength to zirconia and is recommended during crown cementation [8]. Manufacturers have introduced different primers for feldspathic and zirconia crowns. However, when orthodontic providers wish to bond an attachment to an esthetic crown, it is difficult to differentiate clinically a feldspathic porcelain crown from a zirconia crown. Recently, products such as one-step universal primer for silica based and nonsilica based porcelains have been introduced in the market. One example is Reliance Assure plus (RA plus) (Reliance Orthodontic Products, Itasca, IL), with a chemical composition of Bis-GMA, bisphenol A-glycidyl methacrylate, and ethanol. It is imperative for clinicians to know the differences in bond strengths obtained with various available bonding protocols.

Orthodontic bonding primarily involves three steps: etching, primer application, and bonding [9]. To minimize the number of clinical steps, self-etching primers and one-step universal primers were introduced. These primers are composed of unfilled resin and play an indispensable role in the bonding process. With the introduction of self-etch adhesives the number of clinical steps was reduced. Self-etch adhesives do not require rinsing the enamel surface with water. The treated smear layer and demineralized products are incorporated into the resin [10]. Zachrisson et al., Kocadereli et al., and Abu Alhaija et al. all suggested sandblasting to increase the bond strength to porcelain surface [11–13]. However, it has been reported that mechanically roughening the surface of porcelain also entails a higher incidence of porcelain surface fracture [14]. Hydrofluoric acid (HF) has been advocated as a chemical treatment of the porcelain surface to increase the bond strength [11, 12]. Hydrofluoric acid attacks the glass phase of ceramics, inducing microporosities leading to micromechanical bonding with composite resin. However, Hayakawa et al. reported the corrosive nature of hydrofluoric acid causing damage to oral tissues [15]. Bach et al. conducted a systematic review on orthodontic bonding to porcelain; they discussed that application of silane increases the bond strength of brackets to porcelain surfaces [16].

Bonding orthodontic attachments to zirconia surfaces differs in bonding protocol from that of the feldspathic crown surfaces. In contrast to feldspathic porcelain, zirconia does not contain a glass phase and etching zirconia surface with hydrofluoric acid does not enhance bond strength [17, 18]. A systematic review concluded that increased adhesion to zirconia is expected after physicochemical conditioning that involves combination of air abrasion and adhesive promoters such as primers or silanes [19]. Chen et al. concluded that the bond strength of resin cement to zirconia is increased by silanization of the zirconia surface [20]. Another study concluded that incorporation of BisGMA into silane containing zirconia primers does not affect their efficacy while it has shown to affect the bonding ability of silane containing primers [21].

This study aims to investigate different bonding procedures for metal orthodontic attachments to feldspathic porcelain and zirconia surfaces.

2. Materials and Methods

2.1. Sample Size and Preparation. A total of 36 zirconia specimens, 97% zirconium dioxide stabilized with a 3% Yttria-Lava Frame (Sagemax Bioceramics, WA), was obtained. The blocks were sectioned with diamond blade (Allied High Tech Productions Inc., Compton, CA) to obtain 2.5 mm thick and 8.5 mm in diameter disc specimens. The samples were polished using 600-grit silicon carbide paper and subsequently water-cooled. The blocks were then ultrasonically cleansed and glazed in a Dekema oven (Freilassing, Germany) at 1500°C for one minute.

A total of 36 glazed feldspathic porcelain specimens measuring 2 mm thick and 8.5 mm in diameter were prepared. Feldspathic porcelain powder (GC America, Alsip, Illinois) was stacked onto degassed metal disks and fired in a Dekema oven (Freilassing, Germany) at a temperature of 895°C. The sintered specimens were ultrasonically cleaned and glazed.

Table 1: Surface treatment for the feldspathic porcelain group.

Bonding protocols	Surface treatment
Group 1	Sandblasting + HF + Porcelain Conditioner (silane) + RA (primer)
Group 2	Sandblasting + Porcelain Conditioner (silane) + RA plus (primer)
Control group	Sandblasting + Porcelain Conditioner (silane)

Table 2: Surface treatment for the zirconia group.

Bonding protocols	Surface treatment
Group 1	Sandblasting + Z Prime plus (primer)
Group 2	Sandblasting + RA plus (primer)
Control group	Sandblasting + Porcelain Conditioner (silane)

2.2. Surface Treatment. All the samples were embedded in acrylic (Orthodontic Resin, Item # 040–013, Great Lakes Orthodontics Ltd., Tonawanda, NY), placed in mounting blocks, and sandblasted with 50 μm aluminium oxide particles at 30 psi for 4 seconds in a Basic Meter Sandblaster (Renfert Corp, Hilzingen, Germany). The ideal protocol for air abrasion is sandblasting with aluminium oxide particles for 4 sec at 2.5-bar pressure (36.5 psi) [16].

Each material was divided into three groups (Tables 1 and 2) according to one or more of the following surface treatments: hydrofluoric acid 4% (HF), Porcelain Conditioner (silane) (Reliance Orthodontics, Itasca, IL) (trimethoxysilyl-propyl-2-methyl-2-propenoic acid and acetone), Reliance Assure (RA) (Reliance Orthodontics) primer (biphenyl dimethacrylate, hydroxymethyl acrylate), Reliance Assure plus (RA plus) (Reliance Orthodontics) primer (bisphenol A-glycidyl methacrylate and ethanol), and Z Prime plus (Bisco, Inc., Schaumburg, IL) zirconia primer (biphenyl dimethacrylate, methacryloyloxydecyl dihydrogen phosphate, and ethanol).

2.3. Bonding Adhesives. Four different kinds of bonding adhesives (Table 3) were used in the study. All four have different chemical properties and application techniques.

(1) Reliance Assure (RA) primer: the bonding protocol following the manufacturer instructions is to sandblast the porcelain surface with aluminium oxide particles for 4 sec, followed by surface treatment with 4% hydrofluoric acid for 60 sec, rinsing with water and light air drying, applying a single coat of silane, and light air drying for 3–5 sec. Lastly a single coat of the RA primer is applied before using the adhesive for bonding.

(2) Reliance Assure plus (RA plus) primer: manufacturers suggest sandblasting with aluminium oxide particles for 4 sec, followed by application of a single coat of the RA plus primer and light air drying

Table 3: Composition of bonding adhesives.

Bonding adhesive	Chemical composition
Reliance Assure (RA) primer	2-Hydroxyethyl methacrylate (10–30%) and acetone (50–75%)
Reliance Assure plus (RA plus) primer	Bisphenol A-glycidyl methacrylate (10–30%) and ethanol (50–75%)
Z Prime plus (Bisco) zirconia primer	Biphenyl dimethacrylate, methacryloyloxydecyl dihydrogen phosphate (1–5%), and ethanol (70–90%)
Porcelain Conditioner silane primer	Trimethoxysilyl-propyl-2-methyl-2-propenoic acid and acetone

for 3–5 sec before bonding to porcelain and zirconia surfaces. Per the manufacturer, this bonding protocol has reduced number of clinical steps involved in surface preparation for bonding to porcelain and zirconia surfaces.

(3) Z Prime plus (Bisco) primer: the bonding protocol following the manufacturer instructions includes sandblasting with aluminium oxide particles for 4 sec, followed by a single coat application of Z Prime plus primer and light air drying for 3–5 sec before bonding to zirconia.

(4) Porcelain Conditioner silane primer: it was suggested by the manufacturer to be used as one of the preliminary bonding protocol steps to condition feldspathic porcelain. In this study it is a control to the other bonding protocols.

2.4. Bonding Attachments. Following the surface intervention, a round traction hook with a laminated mesh bonding pad, Product # 224–011 from TP Orthodontics (La Porte, Indiana), was bonded using Pad Lock™ light cure composite (Reliance Orthodontics, Itasca, IL). The bonding surface of the traction hook measured 9.95 mm^2. For consistency, all the attachments were bonded to the samples by the same clinician. Each sample was light cured for 20 seconds from the center of the sample to its rim with Ortholux light at 900–1,100 mW/cm^2 (3 M Unitek, Monrovia, CA). Light intensity was measured using a curing light intensity meter, Model #8000 (EFOS Inc., Mississauga, Ontario, Canada) for standardization.

2.5. Tensile Bond Strength Testing. Feldspathic porcelain and zirconia samples were stabilized in the acrylic blocks and ensured for parallelism using stainless steel ruler. Using a 12 V cordless drill (Black and Decker, Towson, Maryland), holes measuring 5/32 inches were drilled into the acrylic units. All the samples were labeled with material group (feldspathic or zirconia) and bonding agent group (RA, RA plus, Z Prime plus, and Porcelain Conditioner). Samples were randomized into three batches constituting four samples from each bonding protocol. Each batch was prepared according to the respective bonding protocol and tested for tensile bond strength measurement immediately. This helped minimize bias with environmental conditions and clinician efficiency.

A round 0.012-inch stainless steel wire was used to loop around the traction hook. A perpendicular tensile force was ensured by attaching this wire to the upper unit of the Instron testing machine Model 1125 (Instron Corp., Canton, MA). The acrylic units were secured via a rod to the lower unit of the Instron machine. The Instron machine was directed to provide tensile stress to the bracket unit until bond failure occurred at a cross head speed of 1.0 mm/minute.

Machine calibration was performed each time a batch was tested and the load balance was set at zero to ensure uniformity. The size load cell for the testing was set to be at 500 kg. Once the sample to be tested was engaged with the stainless wire looping around the traction hook and attached to the upper unit the test was run. The data collection was done using TestWorks® software (MTS Corp., Eden Prairie, MN). The bond strength measured was calculated using the formula $R = F/A$, where "R" is the strength (MPa), "F" is the load required for rupture of the specimen (N), and "A" is the interface area of the specimen (mm square). A digital caliper was used to measure the interface surface area of the specimen. The surface area of the bondable surface of the traction hook measured to be 9.95 mm^2.

2.6. Adhesive Remnant Index Scoring. To determine the adhesive remnant index (Artun and Bergland, 1984), a digital camera Model GT800 (Belmont, MA) was used to examine the debonded surface and the bracket mesh. The scores are as follows:

Score 0: no adhesive left on the porcelain or zirconia surface.

Score 1: less than half of the adhesive left on the porcelain or zirconia surface.

Score 2: more than half of the adhesive left on the porcelain or zirconia surface.

Score 3: almost all adhesive left on the porcelain or zirconia surface, with distinct impression on the attachment mesh.

The digital camera Model GT800 used in this study has a magnification up to 700x (digitally). An optical magnification of 230x was deemed sufficient for this study's purpose. To minimize bias all images were randomly selected and scored for ARI indices by three calibrated examiners. In case the individual scores differed amongst the examiners the majority score was chosen to be the final score.

2.7. Statistical Analysis. The Shapiro-Wilk test was used to check the distribution of raw data and descriptive statistics

Table 4: Descriptive statistics of tensile bond strengths for bonding agents in feldspathic group.

Bonding agents	N	Mean (MPa)	Std. deviation	Std. error	95% confidence interval for mean	
					Lower bound	Upper bound
Reliance Assure plus	12	4.657	0.6020	0.1738	4.275	5.040
Reliance Assure	12	4.724	0.7466	0.2155	4.250	5.199
Porcelain Conditioner	12	4.339	0.7494	0.2163	3.863	4.815
Total	36	4.574	0.7033	0.1172	4.336	4.812

Table 5: Descriptive statistics of tensile bond strengths for bonding agents in zirconia group.

Bonding agents	N	Mean (MPa)	Std. deviation	Std. error	95% confidence interval for mean	
					Lower bound	Upper bound
Reliance Assure plus	12	5.323	0.5249	0.1515	4.990	5.657
Porcelain Conditioner	12	5.290	0.7287	0.2104	4.827	5.753
Z Prime plus	12	5.245	0.3900	0.1126	4.997	5.493
Total	36	5.286	0.5499	0.0916	5.100	5.472

were calculated. To determine the differences in mean tensile bond strengths amongst the different bonding agents for feldspathic and zirconia samples, a one-way ANOVA was used. ARI scores were reported for each group. A cross-tabulation followed by Pearson chi-square test was done for ARI scores among the bonding agents for feldspathic and zirconia. Statistical significance was set at 0.05. Data analysis was performed using IBM SPSS Statistics for Windows (version 22.0 IBM Corp., Armonk, NY).

3. Results and Discussion

All study data showed normal distribution. Descriptive statistics were presented for feldspathic samples in Table 4 and zirconia samples in Table 5. The results of one-way ANOVA indicated no statistical significant mean difference on the variable tensile bond strength (MPa) amongst bonding agents on feldspathic and zirconia samples used in the study: feldspathic, $F(2, 33) = 1.029$, p value = 0.369, and zirconia, $F(2, 33) = 0.058$, p value = 0.944.

The literature on minimum bond strengths is not consistent. Some authors reported the minimum shear bond strength for orthodontic bonding purpose to be 13 to 21.3 MPa [22], while some authors reported the minimum tensile bond strength to be between 6 and 8 MPa [23, 24]. Bond strengths as low as 3–5 MPa have been reported as well [25]. The reason for the differences between the literature and this study may be explained on the basis of (1) the method of bond strength measurement, (2) the time duration from bonding the brackets to the time the actual testing was performed, and (3) the protocol for curing the bracket adhesive. The literature search suggests shear bond strength testing to produce higher values than tensile bond strength tests [26]. In this study 12 samples were used for each intervention group. A minimum of 10 specimens is recommended to perform the shear bond strength testing [27]. Previous studies had specimens thermocycled to induce mechanical fatigue. However, from a clinical standpoint, immediate bond strength is important since arch wires exerting a force are engaged into the brackets within minutes of the bonding procedure.

Feldspathic porcelain (silica based) undergoes surface roughness after sandblasting and/or exposure to hydrofluoric acid, exposing silica oxides for chemically bonding with silane coupling agent and resin. The silanol (Si-OH) group of the primer and the OH group of the ceramic combine to liberate a water molecule and in the process form a stable siloxane (Si-O-Si) bond [28, 29]. It has been reported that sandblasting increases the surface roughness of zirconia [30]. The importance of sandblasting in increasing the bond strength between the adhesive resin and the sample is supported by the study conducted by Ourahmoune et al. [31]. They observed that sandblasting increases surface roughness and the wettability behavior for all materials is significantly influenced by their surface morphology. The contact angle increased with average particle size of the aluminium oxide particles, increasing the mechanical interlocking and hence improving the bond strength. It can be interpreted that sandblasting leads to an increased surface roughness and an increased contact angle of wettability, following which any of the bonding protocols included in this study can provide tensile bond strength values that are not statistically different.

While debonding brackets from enamel, it is important to prevent enamel damage but at the same time have minimal adhesive left on the tooth surface. Likewise, for ceramics, the aim is a debond site that has minimal cohesive damage to porcelain or zirconia but at the same time has minimal residual composite left to remove.

The ARI scores for various bonding agents on feldspathic group and in zirconia group are shown in Tables 6 and 7. For feldspathic group the Pearson chi-square results indicated that Reliance Assure plus, Reliance Assure, and Porcelain Conditioner are not significantly different on whether they were scored as 1, 2, or 3, $\chi^2 = 2.934$, df = 4, $N = 36$, p value = 0.569. For zirconia group the Pearson chi-square results indicate that Reliance Assure plus, Z Prime, and Porcelain Conditioner are significantly different on whether they were

Table 6: Adhesive remnant index scores cross-tabulations for bonding agents in feldspathic group.

Bonding agents	ARI scores			Total
	Score 1	Score 2	Score 3	
Reliance Assure plus	1	3	8	12
Reliance Assure	1	6	5	12
Porcelain Conditioner	0	4	8	12
Total	2	13	21	36

Table 7: Adhesive remnant index scores cross-tabulations for bonding agents in zirconia group.

Bonding agents	ARI scores			Total
	Score 1	Score 2	Score 3	
Reliance Assure plus	0	4	8	12
Porcelain Conditioner	1	3	8	12
Z Prime plus	0	10	2	12
Total	1	17	18	36

scored as 1, 2, or 3, $\chi^2 = 11.059$, df = 4, $N = 36$, p value = 0.026.

This type of failure mode signifies that the physiochemical bond that was formed between the various bonding agents and the sandblasted feldspathic and zirconia samples in this study was higher than the micromechanical retention between the adhesive and the traction hook base. It can be interpreted that the traction hook base was not retentive enough for the adhesive and before the bond strength limit could be reached for any of the samples the traction hook debonded. This may also be purely intentional on the part of the manufacturer because bond strength that is significantly high may pose a risk to harming tooth enamel; thus, by design, the bracket is supposed to debond at the bracket-adhesive interface. Therefore, with the current study design it cannot be determined if there was no difference in tensile bond strength of orthodontic attachments to sandblasted feldspathic and zirconia samples with the included bonding protocols.

The limitation of this study is no inclusion of a material group that was not sandblasted. It is recommended that future studies include a control group where a nonsandblasted surface is tested for application of various bonding protocols.

4. Conclusion

From the results obtained in this study it can be concluded that bonding metal orthodontic attachments to sandblasted surfaces of feldspathic porcelain and zirconia with Porcelain Conditioner, Reliance Assure (RA), Reliance Assure plus (RA plus), and Z Prime plus resulted in tensile bond strengths that were higher than the micromechanical retention between the adhesive and the orthodontic attachment hook base.

Competing Interests

The authors declare that they have no competing interests.

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Research Article

Shear Bond Strength of Three Orthodontic Bonding Systems on Enamel and Restorative Materials

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Objective. The aim of this in vitro study was to determine the shear bond strength (SBS) and adhesive remnant index (ARI) score of two self-etching no-mix adhesives (iBond™ and Scotchbond™) on different prosthetic surfaces and enamel, in comparison with the commonly used total etch system Transbond XT™. *Materials and Methods.* A total of 270 surfaces (1 enamel and 8 restorative surfaces, $n = 30$) were randomly divided into three adhesive groups. In group 1 (control) brackets were bonded with Transbond XT primer. In the experimental groups iBond adhesive (group 2) and Scotchbond Universal adhesive (group 3) were used. The SBS was measured using a Zwicki 1120™ testing machine. The ARI and SBS were compared statistically using the Kruskal–Wallis test ($P \leq 0.05$). *Results.* Significant differences in SBS and ARI were found between the control group and experimental groups. *Conclusions.* Transbond XT showed the highest SBS on human enamel. Scotchbond Universal on average provides the best bonding on all other types of surface (metal, composite, and porcelain), with no need for additional primers. It might therefore be helpful for simplifying bonding in orthodontic procedures on restorative materials in patients. If metal brackets have to be bonded to a metal surface, the use of a dual-curing resin is recommended.

1. Introduction

Increasing numbers of adults have been receiving orthodontic treatment in recent years. In this situation, bonding of fixed orthodontic appliances or orthodontic attachments often has to be conducted on different prosthetic surfaces, such as crowns or cavity fillings made of metal, ceramic, or composite [1]. For an efficient workflow, it is important to establish treatment procedures that are as effective as possible, time-saving, and not subject to error. One-step adhesives were developed in the prosthetic area of dentistry. The use of these materials could be helpful in reducing the cost and effort involved in equipment in orthodontics, as a result of requiring fewer substances to achieve adequate bonding strength. In these adhesives, special primers are used to establish a durable bond following pretreatment of the bonding surface. A specific advantage of the one-step adhesives iBond and Scotchbond Universal is the fact that they contain the monomer MDP, which bonds to other materials than enamel, such as metal and ceramic surfaces [2]. When Scotchbond Universal is used, no other preliminary treatments are necessary apart from macroscopic roughening and cleaning. When iBond is used on silicate ceramic surfaces, an additional ceramic primer is needed. One-step adhesives might be particularly helpful for reducing material costs in orthodontics, with less chair-side time and the ability to avoid hydrofluoric acid.

Bonding between the adhesive and the dental enamel or prosthetic surface is decisive for treatment with a multibracket appliance. According to Brantley and Eliades, the bond strength values for conventional adhesive systems on enamel lie between 8 and 30 MPa [3]. The bond has to withstand the forces that occur in the moist oral environment and at the end of the treatment must be capable of being removed without residue and without causing damage to the enamel or prosthetic crowns, such as cracks or chipping

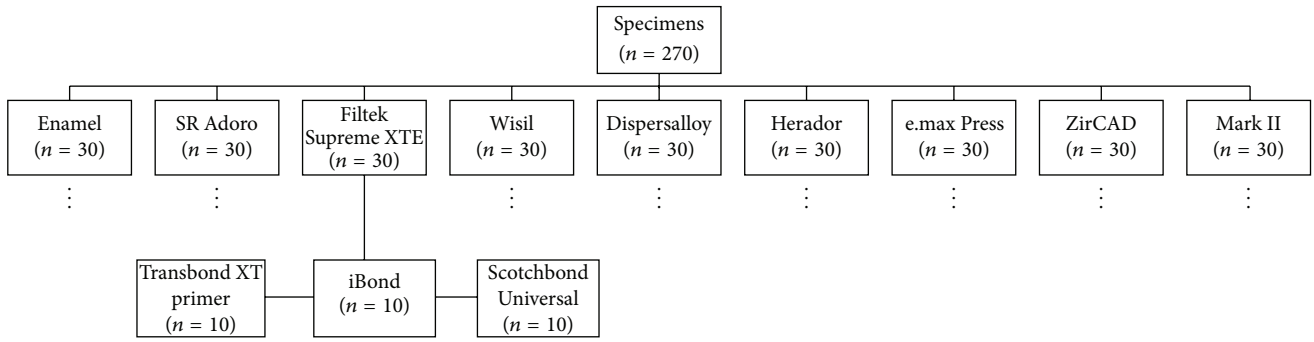


Figure 1: Distribution of the specimens ($n = 270$) across the different surface groups ($n = 30$) and adhesive subgroups ($n = 10$) used.

[4]. Although shear bond strengths (SBS) in the range of 4–10 MPa have been required for bonding to enamel [5], no recommendations are currently available for bonding to different restorative materials. From the clinical point of view, the SBS on restorative materials should be at least as high as on enamel, in order to prevent high rates of bracket loss.

The classic bond to enamel is created using the acid etching technique. Preliminary treatment of the enamel with 37% phosphoric acid leads to micromechanical retention on the enamel surface [6]. Using an adhesive makes it possible to establish a bond from the enamel to the composite.

Self-etching adhesive systems were introduced as an alternative to the conventional adhesive technique. These systems simplified the technique, as etching and application of a bonding agent were now combined into a single step. Bonding to prosthetic surfaces is also simplified, since no additional primers are usually needed.

The advantages and disadvantages of these one-step adhesives have been debated in detail in the literature ever since they were first introduced. Potential advantages include reducing the time required for procedures and possibly minimizing potential errors in application [7]. In addition, self-etching adhesives appear to have advantages for use in a moist environment, due to the aqueous components contained in the primer [8].

Adequate bonding is decisive for complication-free treatment with a multibracket appliance, even on prosthetic surfaces. The aim of the present study was therefore to investigate the shear bond strength (SBS) of two one-step adhesive systems (iBond and Scotchbond Universal) in comparison with the conventional enamel etching system (Transbond XT primer) on prosthetic surfaces. Human incisors served as controls.

2. Materials and Methods

Bonding was conducted on 270 surfaces, 30 human incisors, and 240 prosthetic surfaces (Figure 1). All specimens were randomly divided into nine groups (with human enamel as the control and eight restorative surfaces as the testing groups, $n = 30$). In each group, all of the surfaces were divided into three subgroups with different adhesives ($n = 10$). The shearing tests were carried out on the basis of the DIN 13390-1 and DIN 13990-2 standards [10]. There are numerous test parameters that can influence bonding values. To ensure good comparison, all of the parameters were standardized in the present study, except for the adhesive type. Bonding to the enamel was used as the control group. Human dental enamel is the most appropriate material for testing bond strength on teeth [11]. The teeth used were extracted for general dental reasons and were obtained from dental and orthodontic practices. In relation to ethical guidelines, this represents residual biological material. The enamel surfaces were free of caries, had not been subjected to any dental treatment, and showed no enamel fractures. According to the DIN 13390-1 and DIN 13990-2 standards all extracted teeth were kept in a 0.5% tosylchloramide solution at room temperature. The storage period up to the time of testing was less than 6 months. The other 240 specimens all consisted of restorative materials used in prosthetic and conservative dentistry and were also included as bonding substrates. The specimens had a minimum size of $8 \times 6 \times 1$ mm.

SR Adoro™ Deep Dentin A2 (Ivoclar Vivadent, Schaan, Liechtenstein) was used as a composite, and the bulk fill composite Filtek™ Supreme XTE (3M ESPE Dental Products, St. Paul, Minnesota, USA) was used as a composite resin.

In the alloy group, Herador™ MP (Heraeus Kulzer, Hanau, Germany) was analyzed for the gold alloy group; Wisil™ (Elephant Dental BV, Hoorn, Netherlands) for chrome cobalt alloys; and Dispersalloy™ (Dentsply, Milford, Delaware, USA) for the amalgam group.

Three materials were also used as ceramics: for glass-ceramic veneering, IPS e.max™ Press (Ivoclar Vivadent, Schaan, Liechtenstein) was used; IPS e.max ZirCAD for inLab™ (Ivoclar Vivadent, Schaan, Liechtenstein) was used as high-strength zirconia; and VITAblocs™ Mark II, C2 I14 for CEREC™/inLab (VITA Zahnfabrik, Bad Säckingen, Germany) was used as a monochromatic feldspar ceramic.

A total of 270 samples were thus available for debonding. All of the materials were used in accordance with the manufacturers' instructions.

2.1. Sample Preparation. The roots were cut from the teeth using a diamond saw. The enamel surfaces being tested were

Table 1: Specific information about the components of the adhesives investigated. Etching: Ormco® Gel Etch phosphoric acid 5 mL (Ormco Corporation, Glendora, California, USA).

Adhesive	Pack contents and batch identifier
Transbond XT	Light cure adhesive primer, batch number 8FB/712-034 Light cure adhesive paste, batch number 8CU
iBond	1a iBond ceramic primer, batch code 010089 1b iBond Universal, batch code 010020
Scotchbond Universal blister	1a MDP phosphate monomer, dimethacrylate 1b HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, and silane

at least twice the size of the adhesive surface of the brackets used.

The specimens of SR Adoro Deep Dentin A2 and Filtek Supreme XTE were created by layering the composite into a mold of addition-type silicone (8 × 8 × 2 mm). The light curing with the Elipar™ FreeLight 2 LED lamp in 400–515 nm wavelength range (3M ESPE, Seefeld, Germany) occurs to the manufacturer's instructions (time of light curing: Filtek Supreme XTE 10 s and SR Adoro 20 s). No polishing was done.

The Herador MP gold alloy was available from the manufacturer in the form of plates (8 × 8 × 1 mm) not requiring further processing. The Dispersalloy samples were stuffed into molds in a silicone form and polished to a high gloss in the laboratory after 1 day.

The chrome cobalt alloy Wisil was provided in ingots and required laboratory processing. First of all, wax probes were made in the silicone form. The probes were then placed in an embedding compound, and the Wisil probes were made in accordance with the manufacturer's instructions. After the probes had been cleaned of the embedding compound, no polishing was done.

The IPS e.max Press glass-ceramic material was also provided in ingots, so that laboratory processing was also needed, as in the Wisil group.

The zirconia IPS e.max ZirCAD for inLab samples were cut into probes 8 × 8 × 2 mm from the blocks using a diamond saw (HORICO DENTAL Hopf, Ringleb & Co. GmbH & Cie., Berlin, Germany). In accordance with the manufacturer's instruction all probes needed a laboratory process and were compacted in a sintering furnace. Consecutively there was no polishing done.

The monochromatic feldspar ceramic Mark II was also supplied in the form of blanks, which were cut in a CEREC™ machine.

All of the probes were roughened with 50 μm aluminum oxide particles using an intraoral sandblaster (MicroEtcher; Danville Materials, San Ramon, California, USA) applied from a distance of approximately 50 mm for 5 s, followed by rinsing with a water spray for 10 s and drying with oil-free compressed air. All of the surfaces of the restorative materials were degreased with alcohol.

The clinical crowns and prosthetic specimens were embedded in Palavit G™ (Heraeus Kulzer, Wehrheim, Germany) as required by DIN 13990-2 [10]. The surfaces were oriented with their vestibular surfaces parallel to the upper end of the test tube.

The specimens were randomly divided by an external operator into three subgroups ($n = 10$) with different bonding adhesive systems:

(i) Transbond XT primer (3M Unitek, Monrovia, California, USA);

(ii) iBond (Heraeus Kulzer, Hanau, Germany);

(iii) Scotchbond Universal (3M Unitek, Monrovia, California, USA).

Table 1 lists specific information about the components of the adhesives investigated.

2.2. Bonding Procedure. All of the specimens were polished with Zircate™ Prophy Paste (Dentsply DeTrey, Constance, Germany), rinsed with water, and air dried. For light polymerization, only the Elipar FreeLight 2 LED lamp (3M ESPE, Seefeld, Germany; light irradiance: 1200 mW/cm^2, curing mode: standard, light guide: max Ø 13 mm) was used. Light curing was done parallel to the surface at a minimum distance in the 400–515 nm wavelength range, which meets the DIN 13900-2 standard for the light source.

(i) In group 1 (the adhesive control group), the conventional acid etching technique was conducted for enamel. The dental enamel surfaces were conditioned with 37% phosphoric acid for 20 s and then rinsed and air dried. In the composite group (SR Adoro/Filtek Supreme XTE), a plastic conditioner (Reliance Orthodontic Products, Inc., Itasca, Illinois, USA) was applied. In the base metal group (Wisil/Dispersalloy/Herador), a metal primer (Reliance Orthodontic Products) was used, and in the ceramics group (e.max Press/ZirCAD™/Mark II™), a porcelain conditioner (Reliance Orthodontic Products) was used. The Transbond XT Primer™ was applied afterwards using a foam pellet, thinly dispersed with air. All of the samples were light cured with the Elipar FreeLight 2 LED lamp for 10 s parallel to the surface at a minimum distance.

(ii) In the second group, the self-etching and light curing adhesive iBond (Heraeus Kulzer, Hanau, Germany) was applied to the unconditioned enamel/prosthetic surface. It was applied to the dry enamel/prosthetic surface and rubbed in for 20 s with a single-use applicator. The liquid was then subjected to a gentle

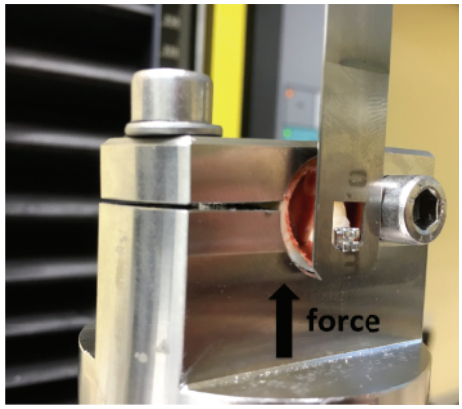


Figure 2: The Zwicki hydraulic testing machine with a specimen in place from lateral view.

airstream for 5 s and light cured in the same way. An additional ceramic primer (Heraeus Kulzer, Hanau, Germany) was needed for 20 seconds on only two ceramic surfaces (e.max Press/Mark II).

(iii) In the last group, the one-step adhesive Scotchbond Universal (3M Unitek, Monrovia, California, USA) was used. Following manual activation of the adhesive in the blister pack, it was applied to the unconditioned enamel/prosthetic surface, rubbed in with the single-use applicator for 20 s, and then air dried and also light cured for 10 s in the same way. No additional primers were needed.

After the use of the different adhesives on the different surfaces, the adhesive paste Transbond XT Light Cure Adhesive (3M Unitek, Monrovia, California, USA) was applied to the bracket base. To allow better comparability, only Discovery™ upper incisor (21) steel brackets (Dentaurum, Ispringen, Germany) were used in this study. The average contact area on the bracket base was 10.95 mm^2. Curing was then carried out again for 20 s (10 s mesial and 10 s distal) with the same light source.

Before polymerization, the brackets were applied at a pressure of 3 N with the help of a Correx™ gauge (Haag-Streit, Berne, Switzerland), following the procedure described by Bishara et al. [12]. All of the test pieces were prepared by one person (J. E.) on 1 day. Before the shear bond testing, the specimens were stored in deionized water at 37°C for 24 ± 4 h.

The shear bond testing was carried out with a standardized, computer-controlled hydraulic testing machine, the Zwicki™ 1120.25 (Zwick Ltd., Ulm, Germany) (Figure 2). The velocity of the force introduced was 1 mm/min, and the shearing force was measured in newtons (N). The clamping yoke had a square opening of 6 mm in diameter and 0.5 mm in thickness. The residual adhesive left on the base of the bracket and on the tooth surface after shearing-off was assessed using the adhesive remnant index (ARI) [13]. This allows bonding failure to be assessed (adhesive rupture versus cohesive rupture). The rupture surfaces were examined under a Leica™ M420 microscope (Leitz, Wetzlar, Germany) at tenfold magnification.

(i) An ARI of 0 corresponds to 0% adhesive on the tooth and 100% adhesive on the bracket.

(ii) An ARI of 1 corresponds to less than 50% of the adhesive on the tooth and more than 50% of it on the bracket.

(iii) An ARI of 2 corresponds to more than 50% of the adhesive on the tooth and less than 50% of it on the bracket.

(iv) An ARI of 3 corresponds to 100% of the adhesive on the tooth and 0% on the bracket.

(v) An ARI of 4 means a surface fracture.

For purposes of better comparability, the resulting forces were converted into MPa in accordance with the following formula:

$$R\ \left(\mathrm{N/mm^2}\right) = \frac{F\ (\mathrm{N})}{A\ \left(\mathrm{mm^2}\right)}, \quad (1)$$

where R is cohesive bond strength, F is force, and A is the cross-sectional surface of the adhesive test piece. The relative value calculated allows comparisons with other studies.

Statistical analysis was carried out using IBM SPSS Statistics™ for Macintosh, version 21.0 (IBM Corporation, Armonk, New York, USA). Normal distribution was tested using the Shapiro–Wilk test. Testing with the Shapiro–Wilk test showed that the values were not normally distributed. Nonparametric tests were therefore used. Statistical differences were analyzed using the Kruskal–Wallis test. The Kaplan–Meier survival curve and log rank test were used to test similarity. The significance level for all of the analysis procedures was set at $P \leq 0.05$.

3. Results

The three adhesives showed different bond strengths on enamel and prosthetic surfaces (Table 2). In descriptive comparisons, Table 2 shows that the Transbond XT adhesive system had the highest (mean) values for shear bond strength (15.51 MPa) on enamel. The highest mean values on all surfaces were obtained with Scotchbond Universal on the Filtek Supreme XTE surface (16.61 MPa) and were average on all other surfaces. Most of the SBSs were higher than the SBS on enamel required by Reynolds [9]. The lowest means were achieved on e.max Press with iBond adhesive (3.44 MPa) and in general on metal surfaces (especially on Herador) with all of the different adhesives. These SBSs were sometimes lower than required for clinical use [9].

The distributions of shear bond strengths in the various adhesive systems are summed up graphically as a box plot diagram in Figure 3 (circles indicate outliers). The Kruskal–Wallis test showed that there were highly significant to non-significant differences between the adhesives in the surface groups (Table 3). Testing for similarity using the Kaplan–Meier survival curve and log rank test also showed that there were significant differences in the survival distributions. The Kaplan–Meier curves showed that, on some surfaces, most of the adhesives showed a lower cumulative survival than the minimum required by Reynolds [9].

Table 2: Descriptive statistics on SBS values for the different bonds used on enamel and prosthetic surfaces.

Surface material	Adhesive		Shear bond strength
Enamel	Transbond XT primer	Mean	15.51
		n	10
		SD	3.49
Enamel	Scotchbond	Mean	12.09
		n	10
		SD	3.85
Enamel	iBond	Mean	6.96
		n	10
		SD	2.00
Enamel	*Total*	Mean	11.52
		n	30
		SD	4.73
SR Adoro	Transbond XT primer	Mean	8.82
		n	10
		SD	3.39
SR Adoro	Scotchbond	Mean	9.03
		n	10
		SD	3.02
SR Adoro	iBond	Mean	8.99
		n	10
		SD	1.94
SR Adoro	*Total*	Mean	8.94
		n	30
		SD	2.75
Filtek Supreme XTE	Transbond XT primer	Mean	9.32
		n	10
		SD	2.80
Filtek Supreme XTE	Scotchbond	Mean	16.61
		n	10
		SD	2.38
Filtek Supreme XTE	iBond	Mean	11.43
		n	10
		SD	2.58
Filtek Supreme XTE	*Total*	Mean	12.45
		n	30
		SD	4.00
Wisil	Transbond XT primer	Mean	7.62
		n	10
		SD	2.37
Wisil	Scotchbond	Mean	4.69
		n	10
		SD	1.84
Wisil	iBond	Mean	5.39
		n	10
		SD	2.89
Wisil	*Total*	Mean	5.90
		n	30
		SD	2.65
Dispersalloy	Transbond XT primer	Mean	6.62
		n	10
		SD	1.61
Dispersalloy	Scotchbond	Mean	6.71
		n	10
		SD	1.70
Dispersalloy	iBond	Mean	9.28
		n	10
		SD	1.92
Dispersalloy	*Total*	Mean	7.54
		n	30
		SD	2.10
Herador	Transbond XT primer	Mean	3.78
		n	10
		SD	1.50
Herador	Scotchbond	Mean	5.14
		n	10
		SD	1.89
Herador	iBond	Mean	4.69
		n	10
		SD	1.63
Herador	*Total*	Mean	4.54
		n	30
		SD	1.72
e.max Press	Transbond XT primer	Mean	7.07
		n	10
		SD	2.62
e.max Press	Scotchbond	Mean	17.20
		n	10
		SD	3.21
e.max Press	iBond	Mean	3.44
		n	10
		SD	2.05
e.max Press	*Total*	Mean	9.24
		n	30
		SD	6.46
ZirCAD	Transbond XT primer	Mean	4.29
		n	10
		SD	2.18
ZirCAD	Scotchbond	Mean	12.33
		n	10
		SD	4.15

Table 2: Continued.

Surface material	Adhesive		Shear bond strength
ZirCAD	iBond	Mean	10.01
		n	10
		SD	3.08
ZirCAD	*Total*	Mean	8.88
		n	30
		SD	4.65
Mark II	Transbond XT primer	Mean	6.37
		n	10
		SD	2.72
Mark II	Scotchbond	Mean	11.16
		n	10
		SD	3.76
Mark II	iBond	Mean	10.85
		n	10
		SD	3.13
Mark II	*Total*	Mean	9.46
		n	30
		SD	3.83

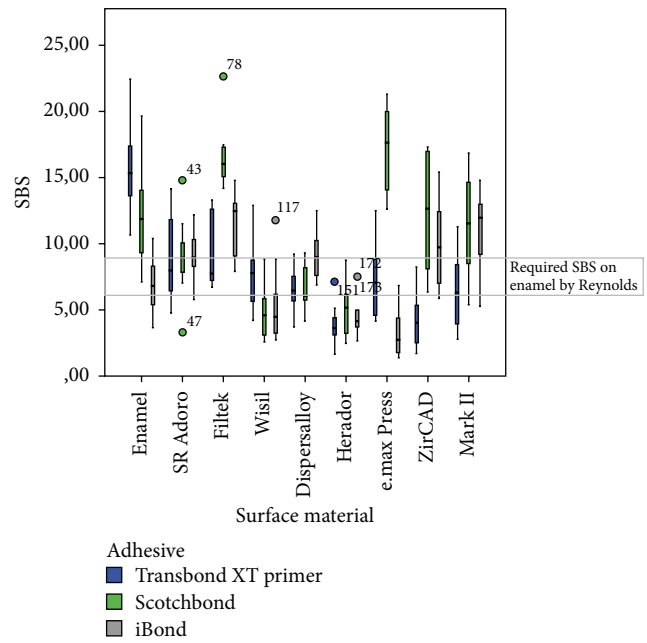


Figure 3: Distribution of shear bond strength (SBS) in MPa in the different adhesives and surfaces used (n = 270). All three adhesives showed different bond strengths, mostly higher and sometimes lower than the values required by Reynolds on enamel [9]. The circles indicate outliers.

The quality of the bonding failure mode was examined and evaluated under a microscope at tenfold magnification. Statistical analysis of the distribution of the ARI scores again showed that they were not normally distributed. The Kruskal–Wallis test showed that there were highly significant to nonsignificant differences (Table 3). Figure 4 shows the distribution of the ARI scores for the different primers, and Figures 5(a) and 5(b) provide typical examples illustrating ARI scores.

4. Discussion

The three adhesive systems investigated in this study showed adhesive strength values on enamel that satisfied or were greater than the minimum required by Reynolds (5.9–7.8 MPa) for the clinical use of brackets [9]. Comparisons in the enamel group showed highly significant differences between the three adhesives with regard to shear bond strength. iBond in particular showed a lower SBS. The view held by several authors that only a weaker bond can be expected is thus confirmed [14].

The Transbond XT primer can be regarded as one of the standard adhesive systems in orthodontics. It has been the subject of many studies examining its adhesive strength [9, 12, 15–17]. In the present study, a mean value was measured for the Transbond XT primer that was comparable to that reported in other studies for the bracket-adhesive bond [18, 19]. From the authors' point of view, Transbond XT primer with a conventional acid etching technique can still be regarded as the gold standard for bonding brackets on enamel, except in special clinical situations, as mentioned below.

The second bonding system, iBond, also showed SBS values similar to those reported in the literature [20]. In comparison with the last adhesive, it needs to be pointed out that Scotchbond™ and Scotchbond Multipurpose Plus™ are not the same as Scotchbond Universal, although they sound similar. Scotchbond Universal is a further development of Adper Easy Bond™, which has been available since December 31, 2012.

A literature search did not identify any comparable studies using a similar study design for the Scotchbond Universal adhesive system, the third adhesive system used in the present study. A few publications about Scotchbond Universal are only concerned with the prosthetic area. Takamizawa et al. [21] reported much higher SBS values, which are not essential for bracket bonding (28.4–48.6 MPa). Comparison with the orthodontic area would be difficult, as these SBSs might lead to unwanted enamel fractures during debonding (ARI = 4).

One advantage of self-etching adhesives is that the substance can be used in a moist environment, due to the aqueous components in the self-etching primer. Hydrophilic adhesive systems are able to repel moisture from the enamel surface, so that the adhesive can penetrate the unconditioned enamel without obstruction [8]. In contrast to conventional adhesive systems, therefore, no absolute drying is required. This can have positive effects, above all when bonding brackets in the inferior and posterior teeth [22, 23], since it makes adhesion easier especially on exposed teeth, as the enamel surface being glued is quite often contaminated with saliva or even blood. As described in the literature in these clinical situations, self-conditioning adhesive systems have better bond strength

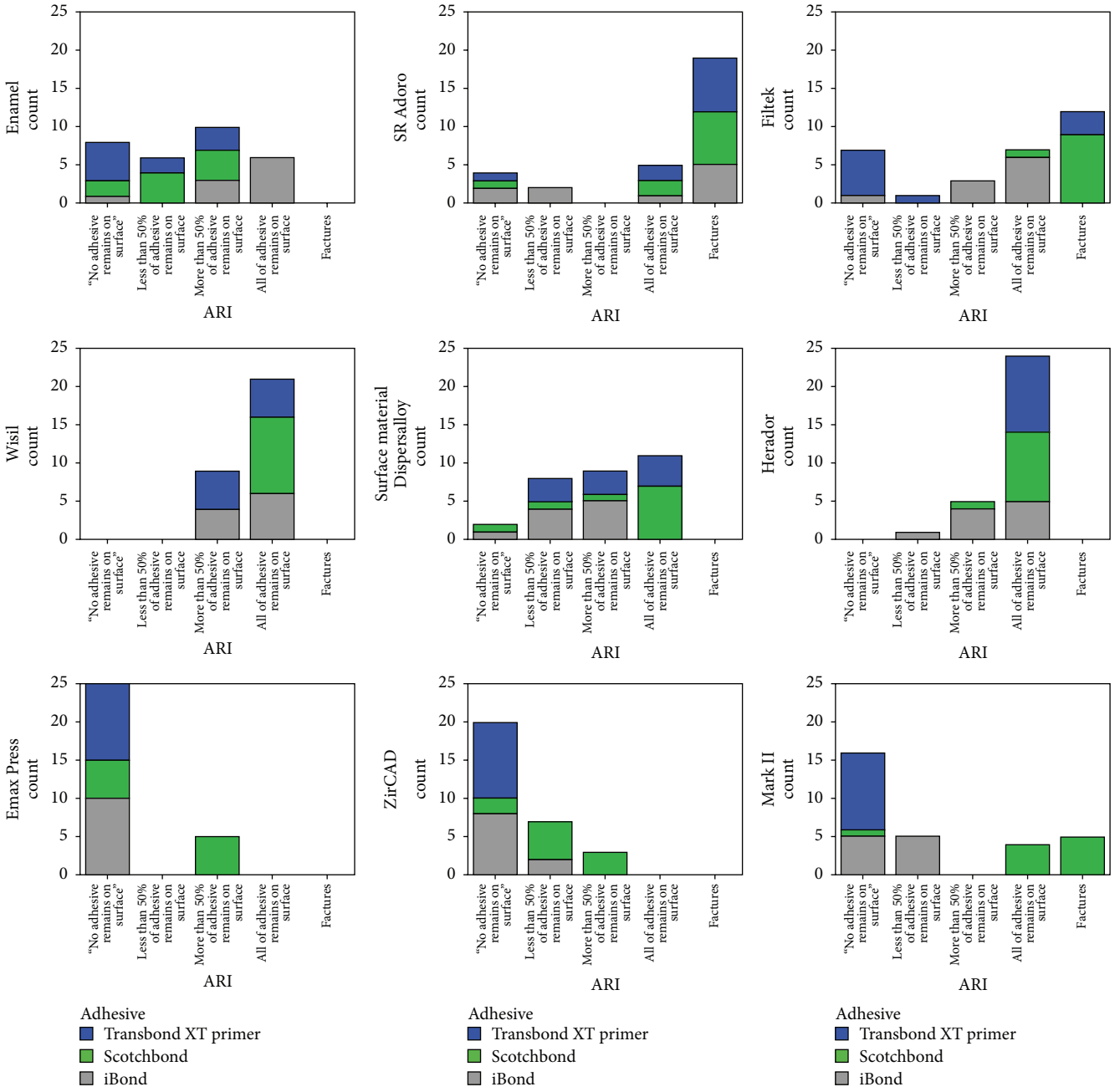
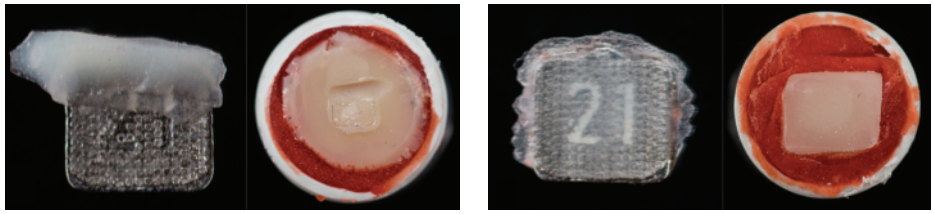


Figure 4: Distribution of the adhesive remnant index (ARI) scores for the three different adhesives used in this study (Transbond XT = blue, iBond = grey, and Scotchbond = green; $n = 270$). An ARI of 0 corresponds to 0% adhesive on the tooth and 100% adhesive on the bracket. An ARI of 1 corresponds to less than 50% of the adhesive on the tooth and more than 50% of it on the bracket. An ARI of 2 corresponds to more than 50% of the adhesive on the tooth and less than 50% of it on the bracket, and an ARI of 3 corresponds to 100% of the adhesive on the tooth and 0% on the bracket. An ARI of 4 corresponds to surface fractures.

values than conventional adhesive systems [24, 25]. The shallower etching pattern in self-conditioning adhesive systems leads to less dissolution of the dental enamel, resulting in reduced loss of hard tooth tissue [26]. The study by Hosein et al. [26] found that, during the process of etching the enamel with self-conditioning adhesive systems, the enamel loss was lower, at 0.03–0.74 μm, than with conventional adhesive systems, at 1.11–4.57 μm.

No specialized products are currently available for bonding orthodontic brackets to restorative materials. As required by Reynolds 30 years ago, the SBS should not be below the cohesive strength of enamel, in order to avoid enamel fractures during debonding [9]. If prosthetic tooth surfaces are bonded with the bracket, the debonding will not damage the enamel, but there is nevertheless a risk of inducing defects or cracks on crowns, veneers, fillings, or other types



(a) (b)

Figure 5: Typical examples of adhesive remnant index (ARI) scores of the bracket base and surface. (a) ARI score 4, with fractures in the group bracket base and surface; (b) ARI score 0, with 0% of the adhesive on the surface and 100% adhesive on the bracket.

Table 3: The Kruskal–Wallis test showed highly significant to nonsignificant differences in shear bond strength (SBS) and adhesive remnant index (ARI) in the three groups.

Surface material		SBS	ARI
Enamel	Chi-square test	18.512	11.686
	Df	2	2
	Asymptotic significance	0.000	0.003
SR Adoro	Chi-square test	0.281	1.636
	Df	2	2
	Asymptotic significance	0.869	0.441
Filtek Supreme XTE	Chi-square test	20.847	14.017
	Df	2	2
	Asymptotic significance	0.000	0.001
Wisil	Chi-square test	6.970	6.444
	Df	2	2
	Asymptotic significance	0.031	0.040
Dispersalloy	Chi-square test	9.223	6.691
	Df	2	2
	Asymptotic significance	0.010	0.035
Herador	Chi-square test	3.583	8.561
	Df	2	2
	Asymptotic significance	0.167	0.014
e.max Press	Chi-square test	23.086	11.600
	Df	2	2
	Asymptotic significance	0.000	0.003
ZirCAD	Chi-square test	16.423	15.612
	Df	2	2
	Asymptotic significance	0.000	0.000
Mark II	Chi-square test	9.762	19.815
	Df	2	2
	Asymptotic significance	0.008	0.000

Df: degrees of freedom (statistics).

of restored surface [27]. Scotchbond Universal and iBond are described by their manufacturers as generating reliable bonds for permanent indirect restorations in prosthetics and conservative dentistry. According to the manufacturers' information, these adhesives contain the monomer MDP, which also creates an adhesive bond with composite, metal, and ceramic surfaces. In this study, the highest means for all prosthetic surfaces were obtained with Scotchbond Universal without an additional primer. The only pretreatment used was sandblasting with alumina particles. Scotchbond Universal may therefore be helpful for reducing equipment costs in orthodontics, as fewer substances need to be used to achieve similarly adequate adhesive bond strengths with different materials. In case of iBond on ceramic surfaces, only one additional ceramic primer is recommended. This might be a disadvantage, as two steps are needed for adequate bonding on glass-ceramic and monochromatic feldspar ceramic surfaces. In the case of Transbond XT, three additional primers (plastic conditioner, metal primer, and porcelain conditioner) are needed. In conclusion, one-step adhesives may be particularly helpful for reducing material costs. Another advantage might be that eliminating the need for selective etching on enamel and bonding brackets, without an additional primer on prosthetic surfaces, may reduce the risk of errors during application and may reduce the amount of chair time [28]. In some surface groups, however, SBS values lower than the minimum required by Reynolds (5.9–7.8 MPa) were found, especially in the metal groups [9]. This might lead to incomplete curing of the adhesive, as not enough light can enter the gap between the light-opaque bracket base and the restorative surface [27]. In this case, the use of a dual-curing resin has to be recommended. With regard to composite [29, 30] and ceramic [31] materials in the present study, higher mean values as well as comparable values were found.

In general, the bonding strength of the adhesive system used should only be large enough to resist the forces that arise in the orofacial region. Contrasting with this, there is the requirement that the system must be easy to remove without causing iatrogenic damage such as chipping and cracking of the enamel or prosthetic surface [4]. In contrast to the requirements for composite fillings in conservative dentistry, where the fillings are intended to remain in place for as long as possible, an adhesive that is used in orthodontics has to be removable at the end of the course of treatment without causing any harm to teeth or restorative material. Once the goal of the treatment has been achieved, a multibracket device must be completely removable. The results of the adhesive remnant index show an inhomogeneous distribution for the three bonding systems. At least some of the adhesives showed an ARI value of 4. In this situation, cracks or fractures on prosthetic restorations were detected. Some of these adhesives might therefore not be safe for clinical use (Figure 5(a)).

Shear bond tests are a recognized in vitro testing procedure for measuring adhesive force. To allow better comparison of the results obtained, they are converted by many authors from N/mm^2 into MPa [32]. There are numerous testing parameters that can influence in vitro adhesiveness values—such as the type of adhesive used, the material properties of the bracket base, the way in which the test pieces are stored, the diameter of the adhesive gap, the shearing velocity of the test machine, the type and duration of light-curing, and the dental or prosthetic material used. With the exception of the adhesive type, all of the other parameters were standardized in the present study to the DIN 13390-1 and DIN 13990-2 standards. Variabilities in the interindividually differing structure of the human enamel are negligible, with a test figure of 10 as required in the test standard [10].

In general, the results of in vitro experiments are never precisely comparable with those of in vivo situations, since application-sensitive substrates and the complexity of the interactions involved are subject to error, and standardization can never succeed 100% [4]. However, the results of in vitro experiments can provide important information for in vivo situations and are of decisive value for clinical practice and everyday clinical use.

5. Conclusions

Within the limitations of an in vitro study, Transbond XT showed the highest SBS on human enamel and can still be regarded as the gold standard on enamel. Scotchbond Universal provides the best average bonding on all other types of surface (metal, composite, and porcelain), with no need for additional primers. It might therefore be helpful for simplifying bonding in orthodontic procedures on restorative materials. If metal brackets have to be bonded to a metal surface, the use of a dual-curing resin is recommended. Further in vivo studies will be needed in order to obtain clinical confirmation of these promising results.

Ethical Approval

This study was conducted in accordance with the Declaration of Helsinki. This article does not include any experiments involving human participants or animals performed by any of the authors. In relation to ethical guidelines, the human teeth used represent residual biological material.

Competing Interests

The authors hereby declare that they have no conflict of interests.

Acknowledgments

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Research Article

Effect of Removal of Enamel on Rebonding Strength of Resin Composite to Enamel

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Objective. To examine the effect of removing the surface layer of enamel on the rebonding strength of resin composite. *Methods*. Teeth in four groups ($n = 10$) were etched, a small amount of resin composite was bonded and debonded, then specimens in three groups were ground for different lengths of time (10 s, 20 s, 30 s) to remove an increasing amount of enamel, one group was left untouched. The teeth were bonded again and the bond strengths of 1st and 2nd bonding were compared and analysed against the amount of enamel loss in different groups (7 μm (±2); 12 μm (±1); 16 μm (±3)). Specimens were examined with SEM and by noncontacting optical profilometer. *Results*. Although results indicated higher rebonding strength with increasing enamel removal ANOVA showed low statistical differences between the groups ($p > 0.05$). However, values between first bonding and rebonding strengths differed significantly ($p < 0.05$) in the group that was not ground. SEM revealed that enamel-surfaces that were ground after debonding etched well, compared to the surfaces that still contained adhesive remnants. *Conclusions*. Removal of small amount of enamel refreshed the surface for rebonding. Rebonding strengths without grinding the surface before bonding were lower than bond strength to intact enamel.

1. Introduction

The etching of enamel is the basis of bonding resins and resin composites to enamel, as it increases the surface area and the surface energy of enamel [1]. Etching produces microporosities into which the adhesive can flow and form a structure of tags, and this micromechanical retention leads to a stronger bond [2, 3]. The depth of the resin tag formation is reported to vary between 5 and 50 μm [3–6]. It has been demonstrated that increasing the length of the tags contributes little to the bond strength [5]. Etching produces a preferential etching pattern depending on the direction of the enamel rods. The two most common patterns are type one where the center of the prisms is dissolved and type two where the prism periphery is dissolved. A third pattern also exists where no prism structure is evident [7–9].

Phosphoric acid etching with an acid concentration of 10–30% produces the highest bond strengths [10]. These acid concentrations generate maximum enamel dissolution and deposits removable by thorough water-rinsing. In clinical work, concentrations of 30–40% are commonly used. Bond strengths measured by so-called shear bond method to etched enamel with different etching times are as an average 20 MPa, which is considered sufficient and represents the highest bonding values which are available with dental tissues and resin composites [11, 12]. A large variety of dental restorative and active orthodontic treatments are based on enamel bonding. However, sometimes debondings occur and there is need for rebonding of the restoration or orthodontic device. Typically, some amount of enamel is removed for the rebonding procedure.

There can be a difference in the generated etching pattern depending on whether the enamel is intact or instrumented, for example, by grinding. In mature enamel, there is a mineralization gradient, increasing from inner to outer enamel, and there can also be differences in the composition of enamel due to patient's age, properties of saliva, fluoride concentration of drinking water, and so forth. In a young intact tooth, the outermost layer of enamel is aprismatic, that is, more condensed. Also, salivary calcium ions can mineralize the enamel and fluorine ions can transform the hydroxyapatite to fluoroapatite. Therefore the superficial enamel layer is usually harder than the inner enamel [9, 13, 14]. The etching result of intact enamel depends on the characteristics of the specific area of enamel and may not be uniform over the whole enamel area [15, 16]. In some studies, it has been reported that when etching with 32–35% phosphoric acid, the intact and ground or otherwise instrumented enamel surfaces both develop a porous surface and the bond strength is similar for both surfaces [17].

When rebonding orthodontic brackets, or when recementing loose adhesive restorations, the properties of the underlying layer of previously treated enamel can affect the rebonding strength. The surface of the enamel can contain adhesive remains even after removing all visible adhesive with a scaler [18]. The remaining adhesive can decrease the roughness of the enamel surface and therefore diminish also the rebonding strength [19, 20]. On the other hand, it has also been suggested that the residual adhesive provides a surface for the new adhesive to bond to, chemically or mechanically [21]. It has been reported that reetching does not remove this residual adhesive [19, 20], and therefore a method that does remove the surface enamel should be employed. The bond strength values in rebonding can be inconsistent. Some studies have found the rebonding strength to be lower than the initial bond strength [19, 20, 22, 23], whereas other studies have found the rebonding strength to be higher than the initial bond strength and attribute this to, for example, an increase in enamel roughness caused by the residual adhesive removal [24].

The objective of this study was to examine systematically the effect of removing the surface layer of enamel in rebonding procedures and to analyse microstructural changes at the teeth surfaces.

2. Materials and Methods

The teeth used in the study were extracted molars acquired from the teaching clinic of Institute of Dentistry, University of Turku, Turku, Finland. The roots of the teeth were cut off with a histological saw (Secotom-50, Struers A/S, Ballerup, Denmark) and 40 teeth were horizontally embedded in acrylic resin inside plastic cylinders. A circular area (minimum diameter 3.6 mm) of enamel was exposed and polished with a polishing machine (LaboPol-21, Struers A/S, DK-2750 Ballerup, Denmark), using first a 180-grit (FEPA) and then a 2400-grit SiC paper. The coarser paper was used to remove a bulk of enamel to get a wide enough surface for the bonding, and the finer paper to finish and smooth the rough surface. A control group of intact enamel ($n = 10$) was also prepared, where the teeth were half-embedded in acrylic resin.

The enamel substrates were etched using a 32% phosphoric acid etching gel (Table 2) and a small cylindrical amount (height 2-3 mm, Ø 3.6 mm) of Transbond XT orthodontic adhesive (Table 2) was bonded on the enamel. The etching and bonding proceeded as follows: enamel was etched for 15 seconds, rinsed with water, and air-dried according to the manufacturer's instructions. A cylindrical (height 5 mm, Ø 3.6 mm) mold cut from a plastic tube was placed on the enamel, the adhesive was dispensed into the mold and light cured for 60 s (20 s from above and 20 s from two sides) with a handheld light-curing unit (light-emitting diode, Elipar S10, 3M ESPE, St. Paul, MN, USA), with the intensity of 1834.8 mW/cm^2 and the wavelength peak of 455 nm ± 10 nm according to the manufacturer. The plastic mold was removed and the specimens were stored in distilled water in 37°C for four hours. After that the specimens were tested for initial bond strength with a testing machine (LLOYD Instruments, AMETEK Lloyd Instruments Ltd., West Sussex UK) with so-called shear bond strength test with cross-head speed of 1 mm/min. Load-displacement curves were recorded. Testing was made in air at room temperature. Then the specimens were stored overnight in distilled water in 37°C. The teeth in the control group of intact enamel were cleaned with pumice, rinsed with water, air-dried, and then etched, bonded, and tested with the same procedures as the rest of the specimens.

The next day the specimens were divided into four groups ($n = 10$): one group was left untouched and in three groups a small amount of enamel was ground off with an automatic polishing machine (RotoPol-11, Struers A/S, Pederstrupvej 84, DK-2750 Ballerup, Denmark) using a 4000-grit SiC paper (Struers A/SDK-2750 Ballerup, Denmark). The groups underwent grinding with the same settings (4000-grit SiC paper, 150 RPM, 5 N) but with different grinding times: 10 s, 20 s, and 30 s. The groups and their treatments are presented in Table 1. The roughness of the SiC papers was chosen so that with the other grinding factors they removed a desired amount of enamel. Then all the enamel substrates were etched and bonded again with the previously described procedures, stored for four hours, and tested for bond strength with LLOYD testing machine. The amount of enamel that was ground off was determined with additional measuring-samples: five substrates were each ground for 10, 20, and 30 seconds and measured. The samples were measured with a micrometer (Coolant Proof Micrometer, Mitutoyo Corporation, Japan). Every sample was measured five times to avoid error, and an average thickness was calculated for every sample. The average amount of enamel that was removed was 7 μm (±2) for 10 s, 12 μm (±1) for 20 s, and 16 μm (±3) for 30 s. All procedures were performed by the same operator.

The enamel surfaces were also imaged for visual analysis using a scanning electron microscope (SEM, JSM-5500, Jeol USA, Inc., Peabody, MA). The substrates were gold-sputtered and imaged. A few substrates of interest of different treatments were selected for examining from the prepared

Table 1: Different groups and their treatments.

Group	Treatment
E	Intact enamel, etched, and bonded
G0	Prepared flat surface, etched, bonded and debonded, then reetched, and rebonded
G1	Prepared flat surface, etched, bonded and debonded, ground for 10 s, then reetched, and rebonded
G2	Prepared flat surface, etched, bonded and debonded, ground for 20 s, then reetched, and rebonded
G3	Prepared flat surface, etched, bonded and debonded, ground for 30 s, then reetched, and rebonded

Table 2: Materials used in the study.

Materials	Manufacturer	Lot no.	Contents	Wt%
Transbond XT	3M Unitek (Monrovia, CA, USA)	N568393	Silane treated quartz	70–80
			Bis-GMA	10–20
			EBPADMA	5–10
			Silane treated silica	<2
Etching gel	3M Unitek (Monrovia, CA, USA)	576331	Water	55–65*
			Phosphoric acid	30–40*
			Amorphous silica	5–10*

Bis-GMA indicates bisphenol-A-diglycidyl ether dimethacrylate and EBPADMA bisphenol-A-bis(2-hydroxyethyl ether) dimethacrylate.
*The specific chemical identity and/or exact percentage (concentration) of this composition has been withheld as a trade secret.

groups. One sample was cut and imaged in transverse section, to observe the depth of the resin tags.

Surface roughness of one specimen from each group was determined by optical noncontacting profiler (Contour-GT-K1, Bruker, Billerica, MA, USA) and analysed with a Bruker Vision 64 software (version 5.41, update 4, Bruker, Billerica, MA, USA), to see how it would correspond to the view of the SEM micrographs. Microroughness of the surface was reported as average surface roughness (R_a).

Statistical analysis was performed with SPSS Statistics version 22.0. The data was tested for normality and a one-way ANOVA was performed with a Tukey's post hoc test. Regression analysis was used to demonstrate correlation between grinding time, that is, removal of enamel layer before rebonding and the rebonding strength.

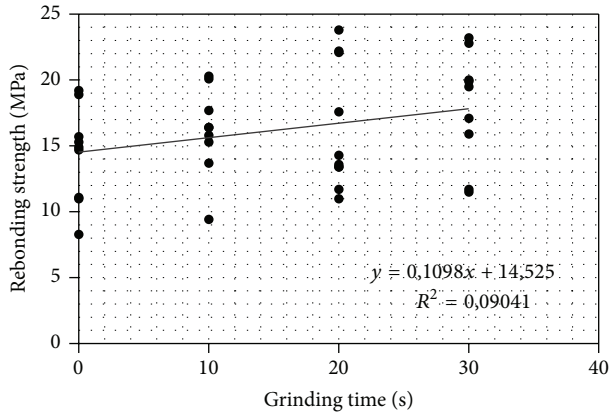

Figure 1: Regression line between the grinding time of enamel before rebonding and rebonding strength.

3. Results

3.1. Bond Strength Measurement. The increase in the grinding time, that is, removal of the enamel layer, showed trend for higher rebonding strengths as demonstrated by the regression analysis (Figure 1). The mean bond strength value for intact enamel was 18.3 MPa (±3.4), and the bond strengths of the 1st bonding were 19.4 MPa (±5.2) for G0 group, 15.7 MPa (±5.2) for G1, 17.5 MPa (±4.9) for G2, and 20.3 MPa (±5.9) for G3. The rebonding strength for the G0 group was 14.3 MPa (±3.6), and the rebonding strengths for the ground substrate groups were 16.1 MPa (±3.3) for G1, 16.3 MPa (±4.8) for G2, and 18.0 MPa (±4.3) for G3 (Table 3). Although there was trend between the increase of rebonding strength and longer grinding time before rebonding (Figure 1), ANOVA did not show statistical differences between groups ($p > 0.05$). Within the groups, the values between first bonding and rebonding strengths differed significantly in G0 group ($p < 0.05$). Figure 2 shows typical load-displacement curves

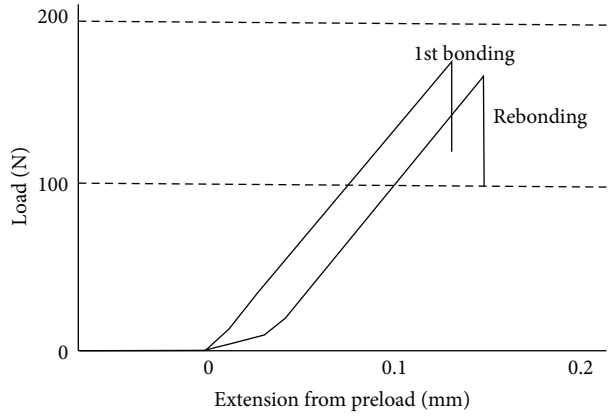

Figure 2: Typical load-extension curves of debonding the composite after the first bonding and after rebonding (curves are from groups G0 1st bonding and G0 rebonding).

Table 3: Bond strengths (MPa) of the composite to enamel after the first bonding and rebonding. Enamel substrate has been ground for 0, 10, 20, and 30 seconds (s) and the corresponding removal of enamel is given in micrometers (μm). Surface roughness after acid etching of the ground enamel substrate is given as value of average surface roughness (R_a).

	G0	G1	G2	G3
Grinding time	0	10	20	30
Thickness of removed enamel	—	7 (±2)	12 (±1)	16 (±3)
Surface roughness (one specimen)	0.301	1.945	0.857	0.343
Bond strength (1st bonding)	19.4* (±5.2)	15.7 (±5.2)	17.5 (±4.9)	20.3 (±5.9)
Bond strength (rebonding)	14.3* (±3.6)	16.1 (±3.3)	16.3 (±4.8)	18.0 (±4.3)

An asterisk $*$ indicates statistical difference ($p < 0.05$) between values.

Table 4: Surface roughness parameters of R_a and R_t of the substrates of test groups in μm. For defining parameters R_a and R_t, see Figure 3.

	Intact enamel	Etched intact enamel (E)	Etched enamel before 1st bonding	Reetched after debonding (G0)	Ground 10 s, etched (G1)	Ground 20 s, etched (G2)	Ground 30 s, etched (G3)
R_a	0.954	2.307	1.928	0.301	1.945	0.857	0.343
R_t	6.245	113.359	27.084	7.463	21.344	13.688	5.175

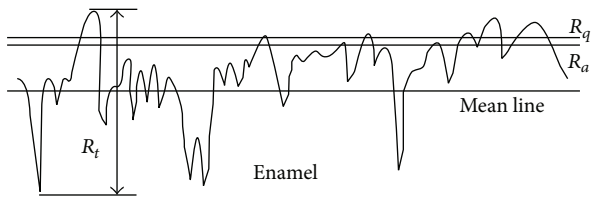

Figure 3: Description of surface roughness parameters: average roughness value (R_a): the arithmetic mean of the height of peaks and depth of the valleys from a mean line (R_t).

demonstrating brittle type of debonding failure for the first bonding and minor ductility for the early stage of loading of the rebonded specimens. Grinding times, amounts of enamel loss, surface roughness parameters, and bond strengths are presented in Table 3. Average surface roughness (R_a) for the intact enamel was 0.954 μm, for the etched enamel (group E) 2.307 μm, for group G0 0.301 μm, for group G1 1.945 μm, for group G2 0.857, and for group G3 0.343 (Table 4).

3.2. SEM Examination. Figure 4 shows representative SEM images and noncontacting profilometer images of the enamel surface of the study groups. The intact enamel surface exhibited signs of wear, that is, pits and grooves. The ground enamel surface was rather smooth, and grinding the enamel surface after the first bonding resulted in a similar surface as in the initial substrate surface, indicating that the adhesive was removed by the procedure. The reetched substrate surfaces showed clear etching patterns, though with different pattern types. The G0 substrate that was not ground before reetching and rebonding contained remnants of adhesive resin after reetching, whereas the ground substrates showed clearly etched enamel surfaces. It can be seen that the reetching of the not-ground substrate did not remove the remaining adhesive resin layer, but turned it into "adhesive resin-mash." The most common fracture pattern, presented in almost all the specimens, was adhesive failure. The enamel fractured in three specimens. SEM of the cross section of the adhesive interface showed depth of the resin tags to be 5–10 μm into the enamel (Figure 5).

4. Discussion

This study aimed to examine differences between removal of different amounts of enamel before rebonding. There was a trend indicating that higher rebonding strength was obtained with increasing enamel removal, although statistical analysis did not show strong relationship between the variables. It is likely that bigger number of specimens would have increased the statistical significance between the variables. It is also possible that the testing method to measure the bond strength with predominantly shear type of stress could have also contained microlocations of tensile stress which could have increased the standard deviations and lowered the statistical differences between the groups. The fracture type was brittle fracture as demonstrated by the load-extension curve (Figure 2). Brittleness of the fracture type is due to the cross-linked polymer matrix of the resin composite. Interestingly, the load-extension curve of the rebonded group showed at the early stage of the loading ductility of some degree which may relate to the presence of partly loose remnants of the resin composite and adhesive resin on the enamel surface on to which the rebonding was made. In the loading event, partly loose particles debond with lower level of stress which can be seen in early stage on the load-extension curve.

It was observed that the G0 group showed significantly higher initial bonding strength (19.4 MPa) than corresponding rebonding strength (14.3 MPa) which suggests that remnants of adhesive resin harm etching the surface for rebonding. Therefore, it is beneficial to reveal a fresh enamel surface before rebonding by grinding the surface. The intact enamel

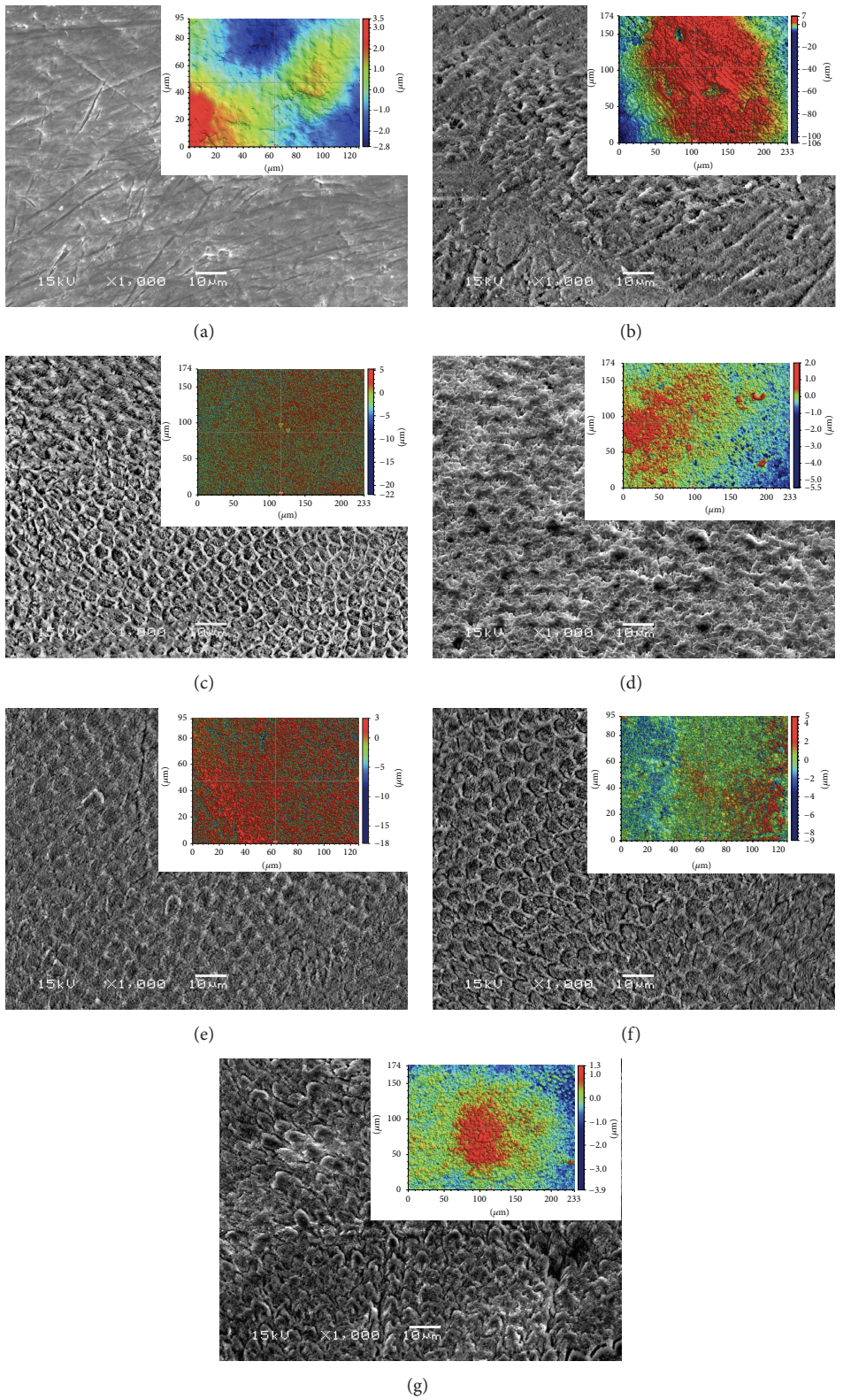

Figure 4: SEM images and corresponding optical profilometer image of the enamel surface of (a) intact, unetched enamel specimen (b) group E before bonding, (c) prepared enamel specimen before 1st bonding, (d) G0 before rebonding, (e) group G1 before rebonding, (f) group G2 before rebonding, and (g) group G3 before rebonding. Original magnification ×1000, bar = 10 μm.

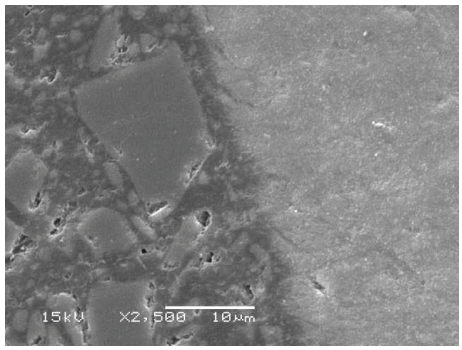

Figure 5: SEM image of the cross section of the interface between etched enamel and resin composite (original magnification ×2500, bar = 10 μm).

surface etched relatively well, despite presence of aprismatic layer or hypermineralization of the enamel surface.

The results of this study revealed that the differences between rebonding directly on the debonded surface or on a slightly ground surface were minor. If the rebonding strength is desired to be as high as the initial bonding strength, then removal of enamel can be recommended. A thickness of a maximum 17 μm of enamel was removed during the grinding process resulting in a similar bond strength as with initial bonding of intact enamel. According to the literature, residual adhesive resin clean-up removes approximately 5–30 μm of enamel [25–27], depending on the grinding system that is used. Even a loss of 40–60 μm of enamel has been reported to occur in the entire debonding and clean-up procedure [6, 28]. In the present study, a 7 μm grinding created a reetchable enamel surface which was also confirmed by the surface roughness measurement for R_a and R_t. Interestingly, surface roughness parameters lowered when the enamel was ground further.

Grinding of enamel can be made clinically by rotating silicon carbide finishing bur and damage to the enamel is only minor [16, 29, 30]. On the other hand, there are reports stating that even with a clean-up with a silicon carbide finishing bur small remnants of adhesive can remain on enamel [19, 20]. Although it was found in the present and other studies [29] that the reetching produced a regular etching pattern to ground enamel surface after debonding, it has also been suggested that the reetching step could be omitted, to avoid risk of enamel fracture due to rebonding strengths that are too high [31].

Clinically, the desired bond strength is different in different areas of dentistry: in restorative dentistry the highest possible bond strength is desirable, whereas in orthodontics the bond must be strong enough to keep the appliances in place for the duration of the treatment but at the same time allow easy detachment of the appliance once the treatment is over. If the bond strength is too high, it can result in enamel damage or discomfort for the patient at the removal. Further research is needed to investigate the amount of enamel loss in debonding and rebonding process with brackets of different types and materials.

5. Conclusions

(i) Rebonding strengths without grinding the surface before bonding were lower than bond strength to intact enamel.

(ii) Removal of small amount of enamel refreshed the surface for rebonding.

Competing Interests

The authors declare that they have no competing interests.

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Research Article

Transmission of Curing Light through Moist, Air-Dried, and EDTA Treated Dentine and Enamel

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Objective. This study measured light transmission through enamel and dentin and the effect of exposed dentinal tubules to light propagation. *Methods*. Light attenuation through enamel and dentin layers of various thicknesses (1 mm, 2 mm, 3 mm, and 4 mm) was measured using specimens that were (1) moist and (2) air-dried ($n = 5$). Measurements were repeated after the specimens were treated with EDTA. Specimens were transilluminated with a light curing unit (maximum power output 1869 mW/cm^2), and the mean irradiance power of transmitting light was measured. The transmission of light through teeth was studied using 10 extracted intact human incisors and premolars. *Results*. Transmitted light irradiance through 1 mm thick moist discs was 500 mW/cm^2 for enamel and 398 mW/cm^2 for dentin ($p < 0.05$). The increase of the specimen thickness decreased light transmission in all groups ($p < 0.005$), and moist specimens attenuated light less than air-dried specimens in all thicknesses ($p < 0.05$). EDTA treatment increased light transmission from 398 mW/cm^2 to 439 mW/cm^2 (1 mm dentin specimen thickness) ($p < 0.05$). Light transmission through intact premolar was 6.2 mW/cm^2 (average thickness 8.2 mm) and through incisor was 37.6 mW/cm^2 (average thickness 5.6 mm). *Conclusion*. Light transmission through enamel is greater than that through dentin, probably reflecting differences in refractive indices and extinction coefficients. Light transmission through enamel, dentin, and extracted teeth seemed to follow Beer-Lambert's law.

1. Introduction

Light transmission through human tooth became a matter of interest when resin based light cured orthodontic adhesives spread in common use and due to the increased use of indirectly luted restorations which are bonded with dual-curing resin composite luting cements [1]. Indirect luting is used especially in prosthodontics but could also be used in orthodontics. In dual-curing resin, composite light is typically used to coinitiate polymerization and in order to achieve proper curing, light irradiation through the curing process is often needed. Polymerization occurs when free radicals are generated, and this can be due to chemical process or curing light initiation [2]. Ceramic inlays, onlays, and veneers are usually bonded with dual-curing cements, which requires light transmission through the ceramic material. In dual-curing resins the cement includes chemical activator, which increases the amount of free radicals when the curing light is insufficient and photoinitiators are unable to produce free radicals required for polymerization [3]. Because the light initiated adhesives are also used in orthodontics to bond brackets, it is important to study the light transmission through the tooth to evaluate the possibility of light curing with transillumination.

Light curing adhesives are used under orthodontic appliances, such as stainless steel brackets that do not let any light pass through [4]. A common way to cure the adhesive under the bracket is to cure 20 s from both mesial and distal side of the bracket. This method may provide clinically satisfactory bond strengths, but there is a risk that the adhesive is polymerized only from the edges of the bracket leaving the center incompletely polymerized [5]. Furthermore the curing

procedure of the adhesive under the bracket, especially in the posterior area, might be difficult to execute.

To solve this problem it was suggested by Tavas and Watts [6] that light curing resin based adhesive could be cured with transillumination through the tooth [6, 7]. To achieve a satisfactory degree of monomer conversion and bond strength, it has been shown that the curing time must be prolonged [8, 9] because of the dentin and enamel barrier. Oesterle and Shellhart [9] presented in their study that a 10 s increase in transillumination time, total curing time being 50 s, enhances the bracket bond strength comparable to labial curing. However extended curing times have a risk of temperature rise in the pulp chamber [10], and the risk is also proportional to the power output of the light curing unit [10, 11].

Light transmission through dentin and enamel is not well known. It has not been reported in the literature whether the hard tissues of the tooth follow the Beer-Lambert law. The Beer-Lambert law is the relation between absorbance and material concentration, but the linearity is limited if the media is highly scattering, as it is in the case of enamel and dentin. It has been suggested that light scatters mainly from the dentin tubules when light irradiation is applied parallel to the dentin tubules [12]. On the other hand, it has been shown that the obliterated dentin tubules do not significantly affect light transmission through dentin [13, 14]. Most studies are only concerned with the optical properties of dentin, but in the orthodontic field teeth are usually intact so the enamel's optical properties must also be taken into consideration. Light transmission through enamel is less well known, and the existing studies consider the light scattering effect only from the esthetic standpoint.

The aim of this study was to measure the light irradiance attenuation of curing light in respect to test specimen thickness for both enamel and dentin.

2. Material and Methods

Teeth used in this study were extracted human third molars, premolars, and incisors, restored in a Chloramine-T/distilled water solution. Teeth were sound without visually detected cracks, caries, or dental fillings. Forty third molars were vertically cut in the buccolingual direction into slices with a thickness of 1 mm to obtain 20 enamel specimens and 20 dentin specimens. The test specimens were cut so that the enamel specimens contained only enamel and the dentin specimens only dentin without visible remnants of any other tissue, and the average diameter of the round shaped test specimens was 5,5 mm (SD 0,58). The specimens were cut with histological saw (Secotom-50, Struers A/S, Ballerup, Denmark) and finished on a polishing machine (LaboPol-1, Struers A/S, Ballerup, Denmark) using 500 grit-SiC paper. After preparing the test specimens they were stored in oil-free distilled water.

The experiment was carried out by measuring light attenuation through 1 mm, 2 mm, 3 mm, and 4 mm of enamel and through 1 mm, 2 mm, 3 mm, and 4 mm of dentin ($n = 5$ in each). Transmitted irradiance was measured first when the specimens were moist, and the measurements were repeated when the specimens were gently air-dried. The increase in thickness was executed by piling the specimens on the sensor. Piled test specimens were picked up randomly among 20 existing specimens. The tip of the light curing unit (LCU) was held as close as possible to the specimen on the sensor. The maximum power output of the light curing unit (led emitting diode, Elipar™ S10, 3 M ESPE, St. Paul, MN, USA) used in this study was 1869 mW/cm^2 when set fully against the sensor, with a center wavelength of 455 nm ± 10 according to the manufacturer. The mean irradiance of transmitted light was measured by a MARC® spectrometer and analyzed using BlueLight® (MARC Resin Calibrator, BlueLight analytics inc., Halifax, Nova Scotia, Canada).

After measuring light transmittance, the enamel and dentin specimens were treated with 19.5% EDTA ethylenediaminetetraacetic acid (File Eze® 19%, Ultradent Products, Inc., 505 West 10200 South Jordan, UT 84095) for 1 minute on both sides to remove the smear layer in order to expose the dentin tubules and enamel rods. After EDTA treatment, light transmittance was measured as described previously, through 1 mm thick enamel and dentin specimens, first as moist and then as air-dried.

Ten extracted human incisors and ten premolars (extracted mainly for orthodontic reasons) were used to study light attenuation through teeth. Each tooth was placed with its labial surface facing the sensor (Ø 4 mm) and the LCU tip on the lingual surface. The thickness of each tooth was measured at the thickest point of the crown, perpendicular to the longitudinal axis of the tooth with a digital slide gauge (Vernier, Millikan Way, Beaverton), with an accuracy of 0.02 mm. The standardized distance of the LCU tip from the sensor was 10 mm for the premolars and 7 mm for the incisors.

Data was analyzed with SPSS (IBM SPSS Statistics for Windows, Version 21.0. Armonk, NY), using a preset level of statistical significance of $p < 0.05$. The normally distributed data was analyzed using Pearson correlation coefficient, a two-way analysis of variance (ANOVA), and Tukey's *post hoc* test. In the ANOVA, the transmitted irradiance (mW/cm^2) was the dependent variable and the factors were thickness and moist/air-dried specimen and their interactions.

3. Results

The two-way ANOVA revealed a significant difference in light attenuation between enamel and dentin ($p < 0.005$) and also in moist/air-dried specimens ($p < 0.05$) as shown in Figures 1 and 2. The increase in test specimen thickness significantly affected the mean irradiance (mW/cm^2) of transmitting light ($p < 0.005$), and for the 4 mm dentin group, the transmitted irradiance was below the level of detection. The values of mean irradiances are presented in Table 1 among Tukey's *post hoc* test results.

After EDTA treatment, the transmission of curing light through 1 mm thick test specimens was significantly higher ($p < 0.05$), except in the moist enamel group where the difference was not statistically significant (Figure 3). A

Table 1: The average mean irradiance and standard deviations (mW/cm^2) of each group, where $n = 5$. Superscripts describe statistical difference between groups.

Specimen thickness	Enamel		Dentin	
	Moist	Air-dried	Moist	Air-dried
1 mm	500,6 (58,0)^{A,a}	389 (49,9)^{B,a}	398,2 (38,4)^{C,a}	251 (66,7)^{D,a}
2 mm	209,2 (11,6)^{A,b}	117 (24,4)^{B,b}	164,8 (21,6)^{C,b}	49,4 (16,8)^{D,b}
3 mm	92 (21,8)^{A,c}	36 (5,5)^{B,c}	51,8 (18,5)^{C,c}	15,4 (6,6)^{D,c}
4 mm	40,8 (9,7)^{A,d}	12,8 (1,9)^{B,d}	19,4 (3,1)^{C,d}	0 (0)^{D,d}

Table 2: Average thicknesses and mean irradiances of incisors and premolars.

	Incisors	Premolars
Specimen thickness	5,6 mm (SD 0,91)	8,2 mm (SD 0,37)
Mean irradiance (mW/cm^2)	37,6 (SD 26,6)	6,2 (SD 6,9)

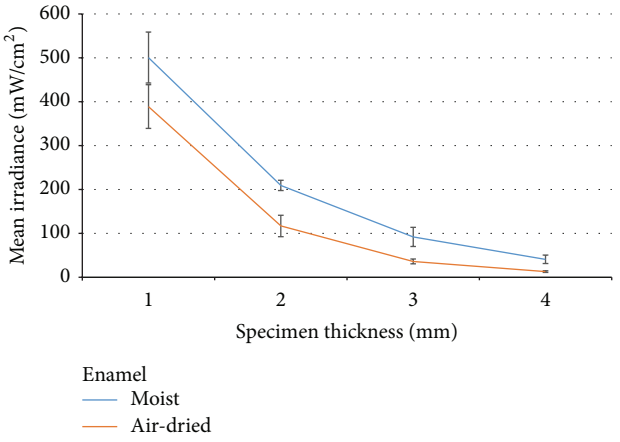

Figure 1: Mean irradiance (mW/cm^2) through enamel specimens at different thicknesses.

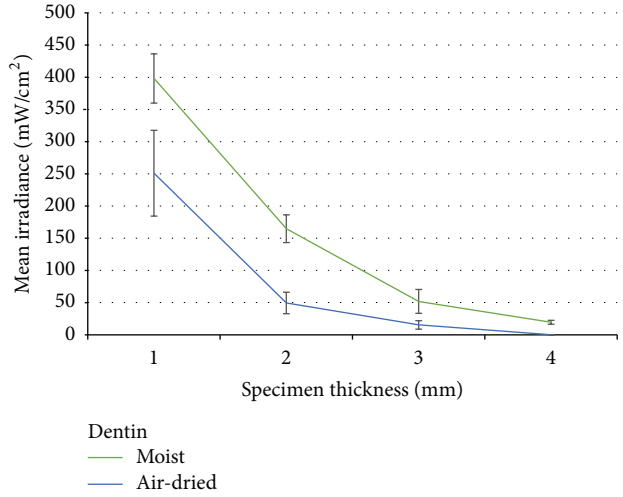

Figure 2: Mean irradiance (mW/cm^2) through dentin specimens at different thicknesses.

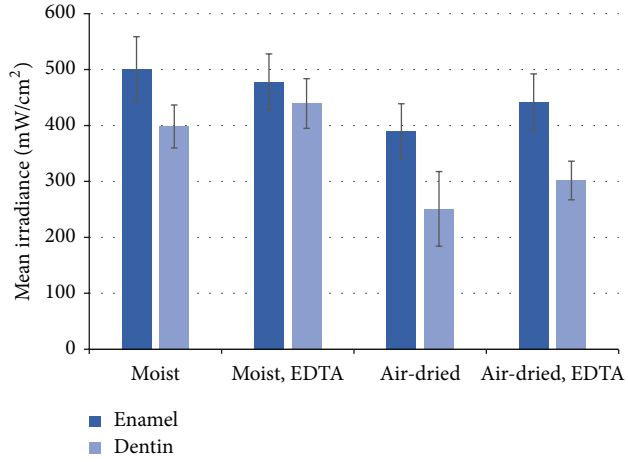

Figure 3: Mean irradiance (mW/cm^2) through 1 mm specimens before and after EDTA treatment. Vertical bars demonstrate standard deviation.

two-way ANOVA revealed statistically significant differences between enamel and dentin ($p < 0.05$), moist or air-dried ($p < 0.05$), and whether the test specimens were treated with EDTA or not ($p < 0.05$). However due to a wide standard deviation, there was no statistical difference in the increase of transmitted light intensity between enamel and dentin when treated with EDTA.

Thicknesses and mean irradiances (mW/cm^2) of light through incisors and premolars are presented in Table 2. Light transmitted somehow through incisors, but transmission through premolars was not significant, with the highest irradiance of 18 mW/cm^2. Incisors and premolars were measured moist. The regression analysis was done to calculate the correlation between incisors and premolars. The coefficient of determination was $R^2 = 0.65$ and the correlation coefficient was $r = 0.81$ ($p < 0.001$) (Figure 4).

4. Discussion

In this study it was demonstrated that light transmission is considerably influenced when there is dentin or enamel between the light source and a light irradiance power detector. This issue has relevance when light curing adhesives and resin composites are cured through dentin and enamel,

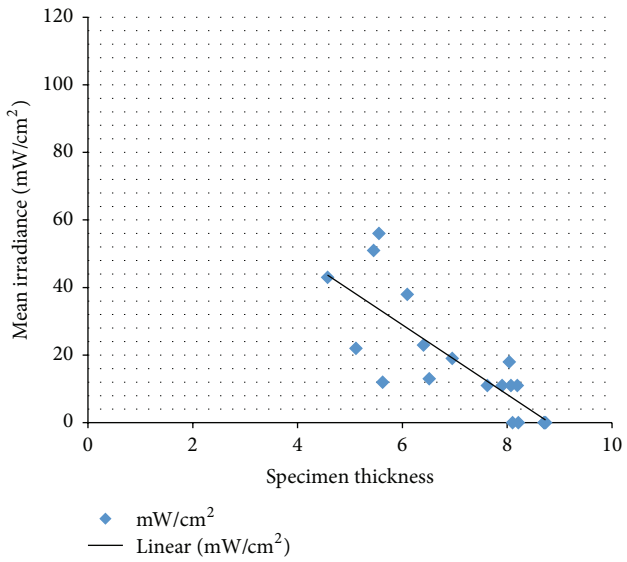

FIGURE 4: Mean irradiances (mW/cm^2) through incisors and premolars.

which can be the case in orthodontic applications. It has been shown that some dual-curing resin composite luting cements require light irradiance to reach an adequate degree of monomer conversion [1]. Thus, the findings of this study have relevance also for restorative dentistry and adhesive prosthodontics. The direction of light transmission in relation to microstructure of dentin and enamel varies in different applications.

Vaarkamp et al. [12] suggested that the tubules are the main cause of light scattering in dentin, because the light transmission was more intense when the dentin was irradiated parallel to dentin tubules. However, earlier studies concerning light transmission through dentin were performed with laser light that has been largely replaced by curing using LED light. Propagation of curing light through a highly scattering media may differ when different light sources are being used. In this study all the specimens were cut in the same direction, so there is no variation between specimen size and shape that could cause alteration to the results. All the dentin discs were cut near the dento-enamel junction; thus the variation in tubule size, shape, and amount may vary because of the physical alteration.

Enamel reflects the wavelength of blue light in certain directions [15], which is due to anisotropy of the enamel [16]. The wavelength of blue light is relatively low (450–495 nm), and it has been observed that translucency increases as the wavelength of the light increases [17]. Light propagates through enamel also by scattering along enamel rods and hydroxyapatite crystals, and the refractive index for enamel is 1.63 and 1.54 for dentin [18]. When enamel specimens were air-dried, the light transmittance decreased significantly, because the water around the enamel rods was replaced with air. The refractive index for water is 1.33 and for air is 1.00, which explains the difference in light attenuation. The different refractive indexes of water and air may have influenced the results, because of the scattering from the water between the piled test specimens. The findings from this study support the results of Brodbelt et al. [19], because the light transmission reverted when the specimens were rewetted.

When light propagates through turbid media (scattering media), the light is composed of absorbed, transmitted, and reflected light [20]. However, the absorbance values are hard to approximate from the Beer-Lambert law, because scattering cannot be separated and considered as an independent phenomenon [21]. Based on the results of this study, it can be suggested that the light attenuation through enamel and dentin follows Beer-Lambert law within the used wavelength of blue light that was used in this study. In case of enamel, reflected light is the wavelength of blue light and is called Rayleigh scattering. Rayleigh scattering appears when light scatters from electrically polarized particles that are minor to the wavelength of light resulting in the scattering which is visible blue light [22]. The scattering of curing light is also affected by the specimen surface texture. The test specimens were polished with the 500 grit-SiC paper, and some of the test specimens were treated with 19.5% EDTA. The EDTA treated surfaces are presumably rougher than the ones polished with 500 grit-SiC paper, so the scattering from the test specimen surface may have been influenced. Edge-loss effect may have influenced the results, since there was no mold to inhibit scattering to the edges [23]. This should be taken into consideration when doing further research about optical properties of a tooth.

In this study the smear layer was removed with EDTA from 1 mm thick test specimens after present studies had been executed, and the light transmittance was then measured again. The reason why the EDTA treatment was not performed on thicker specimens was due to the inability to create a continuous tubule structure with a piling technique. The EDTA treatment did not significantly affect light propagation through dry enamel, which was predictable; hence the hydroxyapatite crystals contribute light propagation through enamel the most, rather than the enamel rods [12]. Hence the light attenuation was only 2% greater when the dentin tubules were obliterated, the results of this study support the findings of Turrioni et al. [13] and Kienle et al. [14], who suggest that the light propagation through dentin is a result of scattering of intertubular dentin. The specimens in this study are cut from the different axis of the tooth than ones in previously mentioned studies, so that may explain the difference between results in light attenuation. To achieve greater clinical relevance, the influence of the resin adhesive system to the light attenuation through tooth needs further studies. This is relevant especially when the light curing is executed from the palatal/lingual side of the tooth, so that the curing light has to penetrate also through adhesive resin system.

The difficulty in metal bracket bonding is obtaining a satisfactory degree of conversion when using light initiated resin based adhesives. However, it has been observed that the light curing adhesives provide better bond strengths than adding a chemical initiator to the adhesive [23]. It has been also revealed that when using dual-curing adhesives, the degree of conversion is significantly higher when the

adhesive is light-cured than auto-cured [24]. These studies are performed with ceramic composites, so that the light curing is performed through 3 mm thick composite blocks. However, the optical properties of translucent composites differ from the optical properties of human tooth [25]. The results of this study offer systematic information about light attenuation through human tooth and can therefore be used when evaluating alternative curing modes for orthodontic adhesives.

Since the output power of light curing units has increased, the curing times have diminished. The energy required to cure the resin is approximately 16 J/cm^2 [26], so with an output power of 1900 mW/cm^2 the required curing time is 8.4 s. According to the measurements of extracted human incisors and premolars, bonding brackets by transillumination would require a 426 s curing time to reach the energy of 16 J/cm^2. It can be assumed that light propagation through vital teeth is greater, due to the scattering effect of blood and nervous tissue. Even though light attenuates greatly when transmitting through the entire tooth, there are studies that show clinically satisfactory bond strengths when orthodontic brackets are bonded using the transillumination technique [7–9]. This may be due to light scattering from the edges of the tooth, where the enamel/dentin barrier is minor compared to that through the thickest point of the tooth. In this study the sensor was placed so that the light transmission was measured only from the thickest point of the tooth. Further investigation is needed to study the bond strengths and the degree of conversion when light curing of the resin based adhesive is performed with transillumination through tooth and when cured indirectly from the mesial and distal side of the bracket.

5. Conclusions

(i) Light transmission through enamel and dentin seems to follow Beer-Lambert's law in the wavelength of blue light.

(ii) Light transmission through dentin was less than that through enamel.

(iii) Moist dentine and enamel transmitted light better than air-dried counterparts.

(iv) The exposed dentin tubules enhanced the light transmission through dentin.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

Analysis of Shear Bond Strength and Morphology of Er:YAG Laser-Recycled Ceramic Orthodontic Brackets

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Objective. The aim of this study was to compare the recycling of deboned ceramic brackets via an Er:YAG laser or via the traditional chairside processing methods of flaming and sandblasting; shear bond strength and morphological changes were evaluated in recycled brackets versus new brackets. *Materials and Methods*. 3M Clarity Self-Ligating Ceramic Brackets with a microcrystalline base were divided into groups subjected to flaming, sandblasting, or exposure to an Er:YAG laser. New ceramic brackets served as a control group. Shear bond strengths were determined with an Electroforce test machine and tested for statistical significance through analysis of variance. Morphological examinations of the recycled ceramic bracket bases were conducted with scanning electron microscopy and confocal laser scanning microscopy. Residue on the bracket base was analyzed with Raman spectroscopy. *Results*. Faded, dark adhesive was left on recycled bracket bases processed via flaming. Adhesive was thoroughly removed by both sandblasting and exposure to an Er:YAG laser. Compared with new brackets, shear bond strength was lower after sandblasting ($p < 0.05$), but not after exposure to an Er:YAG laser. The Er:YAG laser caused no damage to the bracket. *Conclusion*. Er:YAG lasers effectively remove adhesive from the bases of ceramic brackets without damaging them; thus, this method may be preferred over other recycling methods.

1. Introduction

Lasers, which are increasingly employed in oral medicine, have good monochromaticity, excellent coherence, strong directionality, and high brightness. In orthodontics, lasers are used for debonding brackets [1, 2], accelerating tooth movement [3, 4], managing dislodged brackets [5, 6], and preventing enamel demineralization around brackets [7, 8].

Since their commercialization in 1986 [9], ceramic brackets have been favored by adult patients and orthodontic doctors due to their aesthetics, which are better than those of metal brackets. During fixed orthodontic treatments, the rebonding of brackets for various reasons is difficult to avoid [10]. Replacing a new ceramic bracket each time can be not only a waste of resources but also an economic burden on patients. Since ceramic brackets are expensive and maintain their shape and complete structure [11] after falling off, it is of great significance to develop methods of recycling and rebonding dislodged ceramic brackets in the same patients. Previous findings have varied with regard to the effectiveness of flaming [12, 13], silica coating [14, 15], and sandblasting [16, 17]. In 2013, Ahrari et al. [6] reported that an Er,Cr:YSGG laser removed most of the adhesive from dislodged ceramic bracket bases with some degrees of damage to the ball-base and that the shear bond strength of recycled brackets was comparable to that of new brackets. This investigation prompted us to evaluate whether a laser could be used to facilitate the refurbishing of ceramic brackets.

Er:YAG and Er,Cr:YSGG lasers are emitted in the wavelengths of 2,940 μm and 2,780 μm, respectively, and these wavelengths match two of the absorption peaks of water. Specifically, the wavelength of the Er:YAG laser (2,940 μm) is indicated for the treatment of hard and soft tissues [17], so this kind of laser is applied widely in clinical medicine.

In this study, we measured the shear bond strengths of new brackets and dislodged brackets processing by flaming and ultrasonic cleaning, sandblasting, or exposure to an Er:YAG laser. The adhesive remnant index (ARI) was

recorded after removal of the ceramic brackets. Scanning electron microscopy (SEM) was employed to examine the morphology of new bracket bases and processed bases. Confocal laser scanning microscopy (CLSM) was used to observe the three-dimensional structure of bracket bases, and T64000 Raman spectroscopy was used to analyze the ingredients in bracket bases.

2. Materials and Methods

A total of 105 premolar teeth extracted for orthodontic reasons were collected from Beijing Stomatological Hospital. This study was approved by the local Ethics Committee (number 2013-06). Periodontal tissue remnants were removed cleanly, and teeth were stored in 0.9% NaCl at 4°C for up to 6 months until use. All teeth were examined under 10x magnification; any carious, damaged, obviously cracked, hypoplastic, or tetracycline-stained teeth or teeth with dental fluorosis were rejected. Forty-five teeth were used to prepare recycled ceramic brackets for three experimental groups, and the other 60 teeth were separated into four groups for analysis of shear bond strength. Clarity Self-Ligating Ceramic Brackets (3M Unitek, USA) with a microcrystalline base were used in this investigation. Sixty maxillary premolar brackets were allocated to the four groups ($n = 15$ brackets per group).

2.1. Sample Preparation and Group Design. Forty-five new ceramic brackets were bonded to unetched and slightly wet tooth surfaces with Transbond XT adhesive (3M Unitek, USA), allowing easy separation of the bonded brackets from the teeth with tweezers. Before bonding, the buccal enamel of each tooth was cleaned with nonfluoridated pumice powder and rubber prophylactic cups for 20 s, rinsed, dried with air spray, and etched with a 35% gel of phosphoric acid (Heraeus Kulzer, Germany) for 30 s. Sixty ceramic brackets were bonded to teeth with Transbond XT adhesive in accordance with the manufacturer's instructions. Excess resin around the bracket base was removed with a dental probe. Adhesives were light-cured for 10 s on each side of the bracket with a curing light (Beyond, USA).

A total of 45 recycled ceramic brackets were generated and randomly divided into three experimental groups; the control group consisted of 15 bonded, new ceramic brackets. In the flame group, previously bonded (recycled) ceramic brackets were processed via flaming, which was achieved by heating the bracket base on an alcohol burner and burning off the adhesive, rinsing the base under high-pressure water vapor, ultrasonic cleaning for 5 min, and blow-drying. In the sandblasting group, previously bonded (recycled) ceramic brackets were processed via sandblasting using a Macro-Cab Danville Engineering sandblasting machine (MacroCab, USA) with 50 μm aluminum oxide abrasive powder (Hager & Werken, Germany), maintaining a 5 mm distance between the ceramic bracket base and the handpiece head, and sandblasting until the adhesive was not visible to the naked eye and then rinsed with water-air spray for 15 seconds to remove residual powder. In the laser group, previously bonded (recycled) ceramic brackets were processed with an Er:YAG laser (Doctor Smile, Italy) at a wavelength of 2940 nm, a beam diameter of 400 μm, an energy density of 60 J/cm^2, an irradiant power of 6 W, and a repetition rate of 20 Hz, with the ceramic bracket base held perpendicular to the laser until adhesive removal was complete. The operator wore special goggles to protect the eyes during laser exposure. All bonding procedures were performed by the same researcher.

2.2. Shear Bond Strength. Specimens were submerged in distilled water at 37°C for 24 h. Shear bond strength was measured with a universal testing machine (AG-X, Shimadzu, Japan). The cutting blade was placed between the bracket's wing and the base, parallel to the base and perpendicular to the slot of the bracket [18]. Debonding was accomplished with a bar speed of 1 mm/min until the bracket dislodged; a computer recorded the maximum force.

2.3. ARI. After testing of bond strength, the amount of adhesive residue on each tooth surface was observed under a stereomicroscope at 10x magnification. Enamel surfaces were scored according to ARI: 0, no adhesive left on the tooth; 1, ≤50% of the adhesive left on the tooth; 2, >50% of the adhesive left on the tooth; and 3, all adhesive left on the tooth, with a distinct impression of the bracket base [19].

2.4. Morphology of Ceramic Bracket Bases and Residual Component Analysis. Three brackets were randomly selected from each group. The morphology of the ceramic bracket bases before and after processing was observed by spraying carbon under SEM at 300x magnification (SS550, Shimadzu, Japan). CLSM (OLS3100, Olympus, Japan) was used to detect three-dimensional changes in the ceramic bracket base. Component analysis of residues on the base was conducted with Raman spectroscopy (T64000, Horiba Jobin Yvon, France). Spectroscopy data were processed with OriginPro 8 software to produce a Raman spectrogram. Components were evaluated based on the occurrence of the Raman characteristic displacement peak compared with the Raman spectra database.

2.5. Statistical Analysis. All statistical analyses were conducted with SPSS 19.0 for Windows (IBM, USA). The Kolmogorov-Smirnov test indicated that the data for shear bond strength were normally distributed; these data were subsequently subjected to analysis of variance. Further comparisons between groups were conducted with the least significant difference test. ARI scores were analyzed using the Kruskal-Wallis test. The threshold for significance was set at $p < 0.05$.

3. Results

The shear bond strength of recycled ceramic brackets processed by sandblasting was lower than that of new brackets ($p = 0.00$; Table 1). However, the shear bond strength of neither the flame group and nor the laser group significantly differed from the control group (flame $p = 0.79$, laser $p = 0.90$; Table 1), indicating that flame and laser processing were better than sandblasting for ceramic brackets.

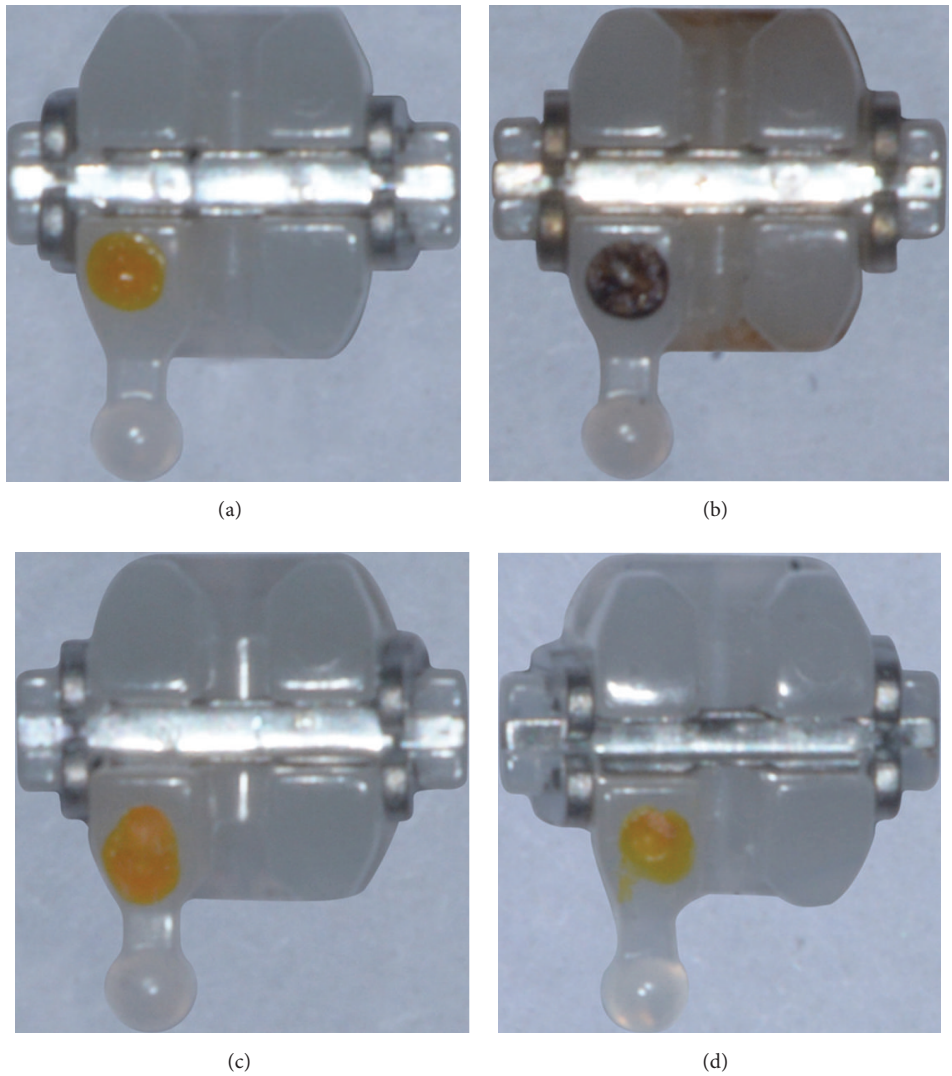


Figure 1: Photographs of ceramic brackets. (a) New bracket. (b) Flame-processed bracket. (c) Sandblasted bracket. (d) Bracket exposed to an Er:YAG laser.

Table 1: Comparison of shear bond strength (N).

Group	n	Mean ± standard deviation	p
New	15	286.053 ± 82.857	—
Flame	15	278.894 ± 56.201	0.79
Sandblasting	15	117.006 ± 68.049	0.00*
Er:YAG laser	15	282.590 ± 77.953	0.90

* $p < 0.05$ versus the control group.

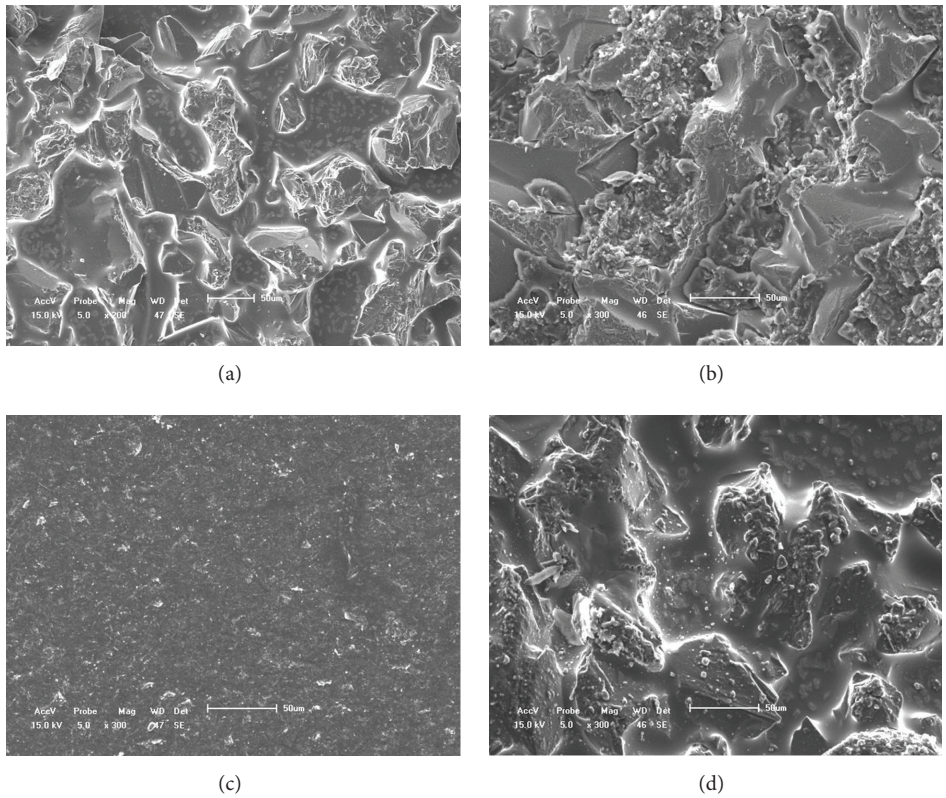
Table 2: ARI scores.

Group	n	0	1	2	3	Total
New	15	4	2	1	8	28
Flame	15	2	2	6	5	29
Sandblasting	15	0	1	3	11	40
Er:YAG laser	15	2	3	5	5	28

ARI scores did not differ between the experimental and control groups ($p > 0.05$; Table 2). However, the ARI scores for sandblasted brackets were higher than those for other brackets (Table 2), suggesting that more adhesive was left on the surface of these teeth and that lower relative bond strength was achieved between the adhesive and sandblasted brackets.

New ceramic brackets had a clear porcelain appearance, while brackets processed by flaming were faded and dark (Figure 1). The colors of brackets exposed to sandblasting or to the laser were similar to that of control brackets (Figure 1).

SEM and CLSM demonstrated that new ceramic bracket bases were made up of irregular microcrystalline structures. Some residual adhesive was evident in the hollows of the microcrystalline structures of ceramic brackets processed by flaming (Figure 2). Sandblasting removed all adhesive, but destroyed the bracket bases (Figure 2). However, the Er:YAG



(a) (b) (c) (d)

Figure 2: SEM (300x magnification) of the bases of ceramic brackets. (a) New bracket. (b) Flame-processed bracket. (c) Sandblasted bracket. (d) Bracket exposed to an Er:YAG laser.

laser removed all adhesive completely and maintained the integrity of the microcrystalline structure (Figures 2 and 3).

An overlapping Raman peak related to aluminum oxide was visible on Raman spectroscopy (Figure 4). For ceramic brackets processed by flaming, the other Raman peak on the spectrogram suggested that carbide remained on the base (the peak reflected the ash of the adhesive; Figure 4). No other Raman peak appeared for new brackets or for brackets processed via sandblasting or the laser (Figure 4), indicating that there was no adhesive on the bases of these brackets. These results of the Raman analysis were consistent with SEM and CLSM.

4. Discussion

Flaming is a very old method for removing adhesive from the bases of ceramic brackets. Lew et al. [12] heated used ceramic brackets to burn off the residual composite resin from the bracket base. After cooling the brackets to room temperature, Lew et al. removed residual composite resin by lightly scraping the bracket base. The brackets were then rinsed with 100% alcohol and resilanized with a thin layer of porcelain primer. Lew et al. [12] reported that the shear bond strength of dislodged ceramic brackets processed by the flaming method was significantly lower than that of new brackets. Martina et al. [13] also detected a decrease in shear bond strength when ceramic brackets were processed via flaming and ultrasound. In the current study, flaming reduced the shear bond strength of ceramic brackets relative to new brackets, but the difference was not statistically significant. However, SEM and CLSM revealed residual adhesive in the hollows of the microcrystalline structure of the bracket base. Raman spectroscopy suggested that this residue was carbide or ash from the adhesive. In addition, ceramic brackets processed by flaming were faded and dark. Due to these poor aesthetics, we do not recommend the flaming method for recycling ceramic brackets.

Sandblasting is a surface-roughening method that was originally used to improve the bond strength of new brackets. Sandblasting is commonly used to remove adhesive from the bracket base, which is effective for rebonding recycled metal brackets [20]. Most previous reports indicated that shear bond strength significantly decreased when ceramic brackets processed by sandblasting were rebonded. The microcrystalline structures on the base of ceramic bracket are fine and fragile, so we chose a smaller granule, 50 μm aluminum oxide abrasive powder, to manage the brackets in order to get less destruction of the bases. Here, we found that sandblasting removed the adhesive from the bracket base, but sandblasted brackets displayed significantly less bond strength than new brackets. This effect may be due to destruction of the bracket base during sandblasting. Although sandblasting roughens the surface of the bracket base, the reduction in bonding strength detected here was far less than what could have resulted from damage to the microcrystalline structure. Brackets are retained on the tooth surface via mechanical

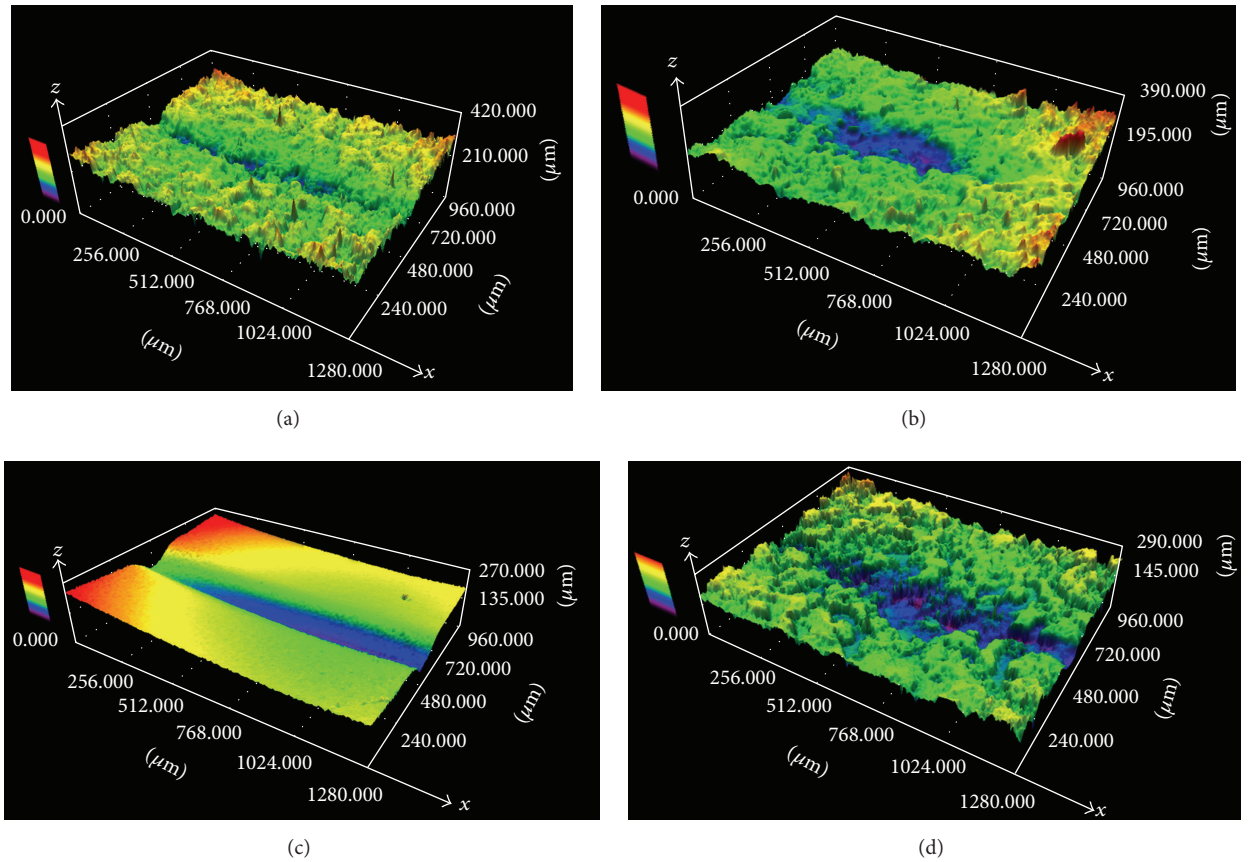


Figure 3: Three-dimensional reconstruction of the bases of ceramic brackets. (a) New bracket. (b) Flame-processed bracket. (c) Sandblasted bracket. (d) Bracket exposed to an Er:YAG laser.

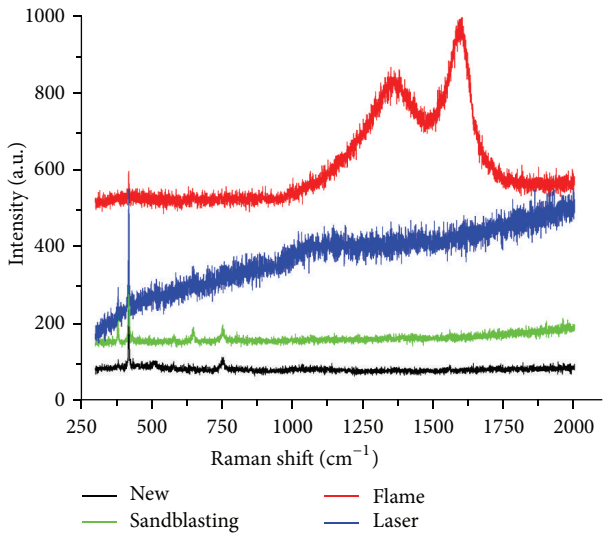


Figure 4: Raman spectroscopy.

interaction between the adhesive and the microcrystalline structure of the bracket; thus, damage to the microcrystalline structure directly influences bond strength.

In recent years, as use of lasers in dentistry is increasing, the hospital does not need to buy the sophisticated equipment for recycling the brackets specially. Processing of metal brackets with a laser was previously successful [21, 22]. In 2013, Ahrari et al. [6] concluded that an Er,Cr:YSGG laser removed most of the adhesive from the bases of dislodged ceramic brackets with some degrees of damage to the ball-base, yielding a shear bond strength that was comparable to that of new brackets. In the present study, exposure of ceramic brackets to an Er:YAG laser at a wavelength of 2940 nm led to complete removal of the adhesive. Absorption of light from an Er:YAG laser is considerably greater in water than in air; the laser transfers enough energy to the water to destroy the adhesive on the bracket. In addition, the use of air and water spray during adhesive removal prevents excessive increases in the temperature of the ceramic bracket. We detected no significant differences between new brackets and brackets exposed to an Er:YAG laser, and the microcrystalline structures of laser-exposed brackets were not damaged. A skilled operator could complete the entire operation in about 2 minutes.

5. Conclusion

Sandblasting significantly reduced the shear bond strength of refurbished brackets and damaged the microcrystalline structure of the brackets, indicating that this technique is unsuitable for processing ceramic brackets. Although flaming-processed brackets had a bond strength that was

similar to that of new ceramic brackets, flaming affected the appearance of the brackets. Exposure to an Er:YAG laser resulted in the complete removal of adhesive from the base of ceramic brackets without damaging them and the shear bond strength of recycled brackets was similar to that of new brackets. In hospitals and private clinics where an Er:YAG laser is available and applied in oral medicine or dental surgery, orthodontist can also use it to refurbish the dislodged ceramic brackets and rebond it to the same patient, which is more effective than traditional methods.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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