Systematized bracket-enamel adhesion test

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Abstract

Objective: Bracket-enamel adhesion studies should address the most important issues – systematic tests and comparable results. Therefore, the purpose of this study is to standardize the bracket-enamel adhesion test by controlling all variables systematically.

Materials and methods: Fifty-six human enamel-bracket adhesions were tested by controlling significant factors including enamel surface, adhesive paste and bracket. The thermocycling was completed (15 sec, 2000 cycles, 5 and 55 degree Celsius). The shear test was exerted by Instron Universal machine (Model 5566, Instron Corp., UK). Moreover, the failure site characteristics were evaluated. The results were analysed using Weibull analysis.

Results: The mean debonding stress and Weibull modulus were 9.47 MPa (S.D.=1.41) and 7.58, respectively. This result was compared to relevant study using t-test. The result suggested that bonds of bracket to human enamel were significantly lower than bond to bovine enamel ($P<0.01$) despite the similar research methodology. Additionally, the failure sites were analysed using Adhesive Remnant Index (ARI). Most brackets of this study showed the ARI of three.

Conclusions: To summarize, the mean stress to break the bond was 9.47 MPa (S.D.=1.41). It could also be concluded that meaningful comparison should arise from systematic studies. However, among the similar research studies, the debonding stress of bracket to bovine enamel was higher than human premolar enamel.

Key words: dental debonding, debonding stress, light-curing of dental adhesives, orthodontic brackets, Weibull analysis, human enamel.

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Introduction

Bracket-enamel adhesion failure has always been a clinical concern. Struggling to find the optimal bond strength, orthodontists discontentedly uncovered that these studies lacked the most important factors – systematic tests and comparable results.[1-3] Tests of similar basic types of metal brackets and bonding materials showed the wide range of debonding stress. Despite these inconsistencies, bracket-enamel bond strength has still been repeatedly reported in terms of new adhesive, modern type and configuration of bracket and its base.[4-8] As was the case, the reported results of these tests were also widely different. Consequently, the difference of debonding stress of these laboratory tests led to the unsound evidence for clinical feasibility, not to mention the contrasting result to the clinical trials.[9]

Further, appropriate statistical analysis recommended for stress test is Weibull analysis. This is due to the fact that the probability of survival is more suitable to explain the bond failure. Instead of mean stress, the probability of survival and Weibull modulus be calculated.[2,5,10]

To our knowledge, both practical and standardized adhesion tests could not be found. Also, there is no standard Weibull modulus. Therefore, it was the aim of this study to systematically determine the enamel-bracket debonding stress by controlling all variables. As a result, this study might provide the meaningful information for reasonable comparison. The null hypothesis was that there would be no difference between the debonding stress of all systematized similar studies. Further, the standard Weibull modulus will be established.

Materials and methods

Fifty-six sound first premolar teeth extracted for orthodontic purposes were collected. The patient age was limited only between 15 and 25 years. Extracted teeth were cleaned, placed in distilled water and sterilized as recommended by Shaffer *et al*.[11] Teeth were then sectioned about 1 mm below the cemento-enamel junction and the crowns were mounted in chemical-cured acrylic resin (Howmedica™, Howmedica International Ltd., London, UK). In order to calibrate the adhesive thickness and ensure that the tooth surface would be parallel to the shearing force, a standard surface was produced by polishing specimen against a silicon carbide abrasive paper with a mean grit size of 400 (Silicon carbide grinding paper, Grit 400, Buehler-Met II™, Buehler, An ITW, Co., Lake Bluff, IL, US). The enamel was then etched with 37% phosphoric acid for 1 minute (Scotchbond ™, 3M Unitek Corp., Monrovia, CA, US), rinsed with air/water spray for 15 seconds and dried with oil free compressed air. The primer was then applied to the etched enamel and bracket base (Adhesive Primer, Transbond™, 3M Unitek Corp., Monrovia, CA, US). Bonding agent was applied on the brackets and then the brackets were placed on the tooth surfaces (Transbond XT™, Mini Diamond™, 3M Unitek Corp., Monrovia, CA, US). The brackets then were again pressed by the loading device to the pressure of 1000 g (Penetrometer, Sabri Dental Enterprise, Ltd., Downers Grove, IL, US). The light was shed on every direction for 40 seconds (Spectrum™, Model No.201R, Dentsply Canada Ltd., Woodbridge, Canada) and stored in distilled water. After specimens were all bonded, they were soaked for 15 seconds in the contrast baths set at 2000 thermo-cycles between five and fifty-five degrees Celsius (Automated thermocycler, Type J5B4D, MECC, Co. Ltd., Tokyo, Japan). The specimens were stored in distilled water for 48 hours in the incubator at 37 degrees Celcius (Memmert™, model BM600, GmbH Co., Schwabach, Germany). Shear testing was performed using Instron Universal machine

(Model 5566, Instron Corp., Buckinghamshire, UK). Each specimen was selected randomly and inserted into a brass prefabricated mount. At the time of insertion, the brackets were aligned until the occlusal surface of the bracket contact was even with the cutting blade. The blade was set parallel to the prepared enamel surface. The speed of cutting is 0.5mm/min. The debonding stress was calculated in MPa unit. The bracket base area was calculated to be 10.2 mm^2 as bracket dimensions were examined under the stereomicroscope at x 25 magnification with a grid for measurement. This detail information was given in table 1.

After shearing, the tooth surface and corresponding debonded brackets were examined under a stereomicroscope at x 25 magnification. The failure interface was evaluated using an Adhesive Remnant Index (ARI) system proposed by Artun and Bergland.[12] A comprehensive literature search was carried out to retrieve previous studies to whose our results could be compared. The inclusion criteria considered studies with the same enamel-bracket adhesion tested, well documented and peer reviewed. Only one study met the criteria.[13] When the inclusion criteria were expanded to bracket with similar bracket base surface, ten studies met the criteria.[13-22] All these studies were reviewed. In addition, test of the metal bracket adhesion that showed Weibull statistical data were also reviewed. So far, only two studies with Weibull analysis used the similar bracket and adhesive.[17,20] One of them, however, bonded the brackets *in vitro* and removed the teeth for debonding test.[17]

Descriptive and Weibull statistical data was analysed for the result of this study. T-test was used to compare our results altogether with study reported by others. To illustrate the failure sites data, the descriptive statistics were utilized.

Results

The shear debonding stresses were illustrated in table 2. Other descriptive variables were also given. This stainless steel bracket showed the mean shear debonding stress of 9.47 MPa (S.D.=1.41). Weibull analysis was also given in table 3, 4 and figure 1.

Further, the Weibull modulus, characteristic strength and debonding stresses at probability of survival at the level of 0.05, 0.90 and 0.95 of this study and relevant studies were reported. Reynolds recommended the appropriate stress level for clinical orthodontics at six to eight MPa.[23] Therefore, the probabilities of survival at these specific stress levels were also showed. Moreover, the ARI index showed the most prevalent ARI score of three, which meant they left the largest amount of resin on the tooth surface. No enamel fracture was observed in any teeth. No bracket had an air bubble underneath.

Table 1 The stainless steel Edgewise bracket and its specification.

Proprietary name	Mini-Diamond™
Type	Stainless steel Edgewise bracket
Area of base (mm^2)	10.2
Bracket base texture	Optimesh 4 lines per 1 mm
Specification	Mandibular incisor narrow mini twin Thick base, 0.018"slot (0.46 mm) -1° Torque 0° Tip
Batch No.	350-0025
Manufacturer	Ormco Corp. Glendora, CA, USA.

We used the t-test to compare our results altogether with study reported by others. The results were showed in Table 2. The result of the group testing bovine teeth demonstrated higher debonding stress than human tooth studies ($P<0.01$). In summary, the null hypothesis was rejected. There as difference between the debonding stresses from similar studies when different tooth types were compared.

The similar studies using similar bracket base surface (Victory™ and Gemini™ series, 3M Unitek) were also listed in the table 3 and 4. Likewise, most recent studies showed the tendency that debonding stress of bracket to bovine enamel was higher than human enamel.

The comparison was also extended to the ARI scores (table 5). As mentioned, most of brackets in this study left the resin on the tooth surface (ARI=3). Unfortunately, two studies gave

Table 2 The comparative table of the studies of bracket-enamel adhesion tests. Both studies tested the bond strength using the same brackets and adhesive paste.

Author(s)/ year	Bracket type (tooth, brand, company)	N(tooth type)	Thermo cycling	Enamel surface flattening	Load pressed on bracket	Speed of blade (mm/min)	SBS (MPa)	Failure site (mode)
This study	Lower incisor Mini-diamond Ormco	56(Human premolars)	2000 cycles (15sec, 5&55C)	Polish (400 grit)	1000g	0.5	9.47±1.41	ARI 3
Staudt et al. 05[13]	Upper right central incisor, Mini-diamond Ormco	15 (Bovine mand primary incisors)	No	No	N/A	0.5	19.2±6.9*	ARI 2

* $p<0.01$

Table 3 Descriptive statistics of the debonding stress.

Author(s)/ yr	Enamel and bracket type	Thermocycles	Mean (MPa)	S.D.	Weibull modulus	Characteristic strength (MPa)
This study	Human premolar Lower incisor Mini-diamond, Ormco	2000	9.47	1.41	7.58	10.04
Elekdag-Turk et al. 08[20]	Bovine incisor Lower incisor, Gemini,3M Unitek	0 2000 5000	18.08 17.14 16.70	1.57 3.17 3.97	13.37 5.65 4.03	18.77 18.54 18.50
Cal-Neto et al. 06[17]	Human premolar Victory series, 3M Unitek	N/A	11.35	2.36	4.90	12.39

the failure site information in different format.[14,16] Therefore, their results were not comparable. Furthermore, the remaining human premolar study had the most prevalent failure site of ARI zero and one. For bovine teeth test, the most prevalent ARI scores were reported to be zero and two (table 5).

Discussion

It has been generally accepted that bracket-enamel adhesion is an important issue in contemporary orthodontics. Most clinicians try to identify the bracket and bonding procedure granting optimal bond strength. Although researchers had put their best effort to these tests, variability in bracket-enamel adhesion strengths was not uncommon. Therefore, the present study was designed to determine the shear debonding stress systematically.

Since there were several factors that may affect the shear debond stress, this study aimed

Table 4 The Weibull analysis of the debonding stress.

Author(s)/ yr	Thermocycles	Probability of survival at stress of			Stress (MPa) at probability of survival at the level of		
		6 MPa	7 MPa	8 MPa	0.95	0.90	0.05
This study	2000	0.98	0.94	0.84	6.77	7.45	11.60
Elekdag-Turk et al. 08[20]	0 2000 5000	N/A	N/A	N/A	N/A	N/A	15.03 10.96 10.59
Cal-Neto et al. 06[17]	N/A	N/A	N/A	N/A	N/A	7.83	14.69

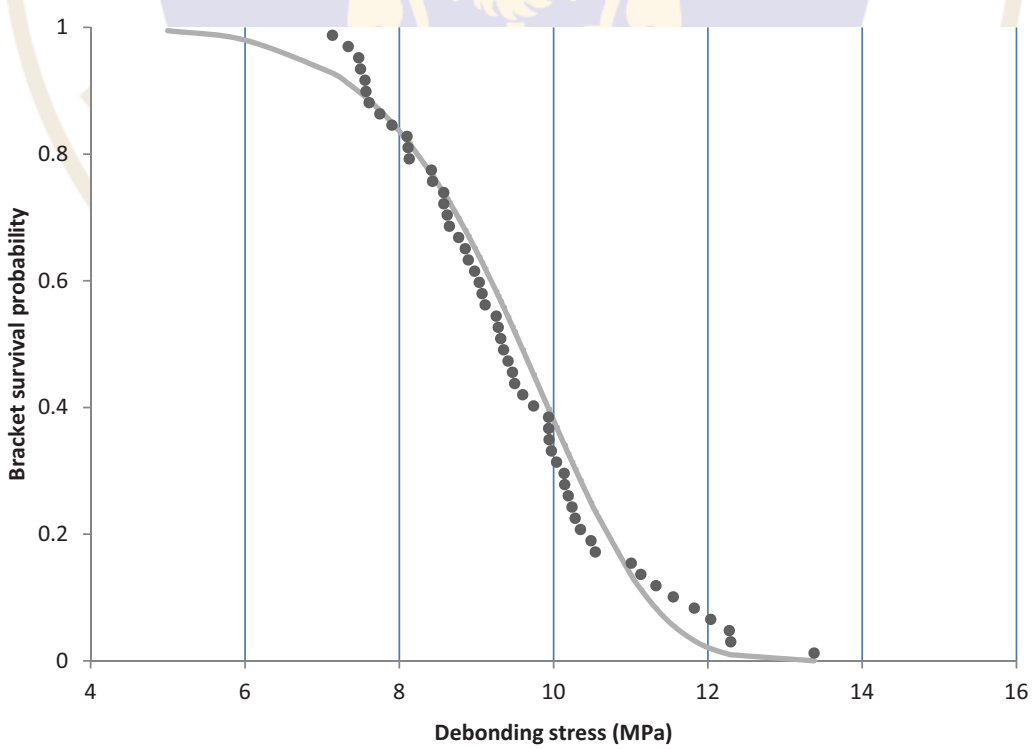

Figure 1 shows Weibull survival probability line. The round dot represents the plot of the deboning stress data. The vertical lines at 6 to 8 MPa refer to the optimal debonding stress level for clinical orthodontics.

to control all these factors. There were some concerns that age of the donor and different tooth types may give different results. Therefore, age and tooth type were strictly controlled in this study. Tooth surfaces were also prepared to achieve a parallel surface to the shearing force. To minimize the cement thickness, the only bracket used was the mandibular central incisor bracket that had a nearly flat bracket base. Moreover, to be comparable, only common etching and bonding system were used in this study. Consequently, the design of this study validated the comparison of the result to a certain extent.

The Weibull analysis showed the characteristic strength and the Weibull modulus of 10.04 and 7.58, respectively. The characteristic strength represents the 63.2 percentile of the strength distribution, which defines the debonding stress at which 63.2 per cent of the units will have failed.[24] In addition, Weibull modulus is the slope of the Weibull plot that is used to describe the variation in the strength or asymmetric strength distribution as a result of flaws and microcracks, which may develop within the microstructure.[25] It indicates the class of failure. Therefore, the higher Weibull modulus may indicate a more clinically reliable system.[26] The systematic research methodology

Table 5 the ARI of all adhesion tests, percentages and the most prevalent values.

Authors/year	Bracket type	N(tooth type)	Percentage of ARI				The most prevalent ARI
			0	1	2	3	
This study	Lower incisor, Mini-diamond, Ormco	56(Human premolars)	9	9	27	55	3
Rajagopal et al.04[16]	Upper premolar Gemini series, 3M Unitek	20(human upper premolars)	N/A	N/A	N/A	N/A	2.40±1.47*
Cal-Neto et al. 06[17]	Victory series, 3M Unitek	23(human premolars)	43.5	17.4	4.3	34.8	0
Ryou et al.08[18]	Premolar, Gemini, 3M Unitek	10(Human premolars)	10	50	20	20	1
Turk et al. 07[19]	Premolar Gemini series, 3M Unitek	10 (Human upper premolars)	20	60	10	10	1
Elekdag-Turk et al. 08[20]	Lower incisor, Gemini,3M Unitek	75 (Bovine incisors) Cycles of thermocycling 0 2000 5000	 20 40 68	 36 16 4	 40 36 20	 4 8 8	2 0 0
Staudt et al. 05[13]	Upper right central incisor, Mini-diamond, Ormco	15 (Bovine mand primary incisors)	0	13.3	73.3	13.3	2
Cozza et al. 06[22]	Upper incisor, Victory series, 3M Unitek	10(Bovine incisors)	50	30	20	0	0

*This ARI is from Bishara classification. The mean score of 2.40 means most of resin composite was left on tooth.

of this study yielded the reliable result substantially. This study provided the least standard deviation. The Weibull graphical diagram also confirmed this fact. All brackets showed the shearing stress plots that were closed to the line. Moreover, Reynolds recommended the appropriate stress level for clinical orthodontics at six to eight MPa.[23] Therefore, the probabilities of survival at these specific stress levels were calculated. They were all pass Reynold's standard. Since there is no establishment of standard characteristic strength of enamel-bracket adhesion, it might be rational to propose the characteristic strength of 10.04 form our study as the standard for further studies.

The study by Staudt et al.[13] is the only study that tested the same bracket to this study. However, neither thermocycling, nor tooth surface flattening were found. They also conducted the test on bovine enamel. Although the methods were very similar, the results were statistically significantly different. We speculated that this could be the result of different tooth type. Moreover, when taking the systematic table into consideration, the comparison could basically be explained by categorizing these papers into two groups, human and bovine teeth. Considering the test result systemically, all bovine studies[13,20-22] showed higher bracket-enamel debonding stress than human studies.[14-19] In Addition, the standard deviations of these studies were also so small especially when they were compared to the arithmetic means. Therefore, even without statistical analysis, the differences of these arithmetic means were so significant. This comparison substantiated the rejection of null hypothesis and confirmed that bovine enamel may yield higher stress than human enamel. In contrast to previous classic papers,[27-28] at present, these papers also showed a strong trend substantiating this higher bond strength of bracket-bovine enamel. Therefore, it is more practicable to use bovine teeth as long as the clinicians realize that the strength may show the tendency towards higher.

Conclusions

Although bracket-enamel adhesion test can be very sensitive due to many factors involved, well controlled systematized test may provide a more meaningful comparison among shear debonding stress. In contrary to the previous studies, many present studies revealed that bovine incisor enamel-bracket bond strength is higher than that of human premolar tooth enamel. This study also supported this claim.

It is important to bear in mind that only well controlled test provide the reliable bond strength. Although the design of this study has already been systematically considered, generalization of these results to the clinical practice should also be done with caution. In addition, some clinicians may prefer the unique mechanics provided by a particular design of a specific bracket even with lower bond strength.

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Ethical approval: It is the aim of this laboratory study to be conducted without violating any human right and ethics. There will be no revealing of the names of tooth donators. However, this study was finished before the establishment of Mahidol University-Centre of Reinforcement of Human Research (MU-CERif).

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