In Vitro Evaluation of a Moisture-Active Adhesive for Indirect Bonding

Arndt Klocke, Dr med dent, MS[a]; Jianmin Shi, MSc[b]; Bärbel Kahl-Nieke, Dr med dent, PhD[c]; Ulrich Bismayer, Dr rer nat, PhD[d]

Abstract: The aim of this in vitro investigation was to evaluate bond strength for a cyanoacrylate adhesive in combination with an indirect bonding technique. Eighty bovine permanent mandibular incisors were randomly divided into four groups of 20 teeth each. The influence of two factors on shear bond strength was investigated: (1) type of adhesive (Smartbond® cyanoacrylate, Sondhi Rapid Set® composite sealant) and (2) time of debonding (30 minutes and 24 hours after bonding). Stainless steel mesh–based brackets were used. Although, bond strength was not significantly different for the two debonding time periods, significantly lower bond strength measurements were found for the cyanoacrylate adhesive ($P <$ .001). The mean bond strength for the cyanoacrylate adhesive group was 5.44 ± 1.65 MPa for debonding 30 minutes and 6.92 ± 1.48 MPa for debonding 24 hours after the bonding procedure vs 16.16 ± 5.25 MPa and 14.98 ± 2.85 MPa for the composite adhesive groups debonded at 30 minutes and 24 hours, respectively. The Weibull analysis indicated that there was an increased risk of bond failure at clinically relevant levels of stress for indirect bonding with the cyanoacrylate adhesive. (_Angle Orthod_ 2003;73: 697–701.)

Key Words: Indirect bonding; Cyanoacrylate; Bond strength; In vitro

INTRODUCTION

Cyanoacrylate-based adhesives can be considered moisture-active and require the presence of moisture for the initiation of the polymerization process.[1] The difference between commercially available cyanoacrylate-based instant glue and the currently available cyanoacrylate orthodontic adhesive Smartbond® (Gestenco, Gothenburg, Sweden) is that the bonding material contains silica gel to make it more viscous.[2] Polymerization of the material consists of two steps:[1,3] (1) isocyanate reacts with water and forms a carbamic acid component that rapidly decomposes to carbon dioxide and the corresponding amine and (2) the amine reacts with residual isocyanate groups, cross-linking the adhesive through substituted urea groups. This polymerization process results in a rather short working time of approximately five seconds and might be considered disadvantageous in direct bonding but is well suited for indirect bonding purposes.

Reports on in vitro bond strength with cyanoacrylate-based adhesives in orthodontics were first published by Howells and Jones[4] who investigated a powder-liquid system that exhibited a significant decrease in bond strength when tested at seven days or longer after bonding to the tooth surface. Hydrolysis of the material might have been the reason for the decrease in bond strength. A more recent cyanoacrylate-based product has been found to be stable after storage for up to 150 days after bonding.[5] Reports on in vitro bond strength for direct bonding with the currently available material Smartbond® (Gestenco, Gothenburg, Sweden) are not in agreement with each other and a wide range of bond strengths has been reported.[1,2,6–8]

Indirect bonding was first reported by Silverman et al[9] in 1972. Most current indirect bonding techniques are based on the technique developed by Thomas.[10] This involves the fabrication of a composite custom base of the bracket in the laboratory. During chairside bonding, only a thin layer of sealant is needed for attachment of the custom-fitted bracket base to the tooth. An indirect bonding technique without fabrication of a custom bracket base has also been described. This technique uses water-soluble glue to posi-

[a] Associate Professor, Department of Orthodontics, College of Dentistry, University of Hamburg, Hamburg, Germany.

[b] PhD student, Department of Earth Sciences, Institute of Mineralogy, University of Hamburg, Hamburg, Germany.

[c] Professor and Chair, Department of Orthodontics, University of Hamburg, Hamburg, Germany.

[d] Professor and Head, Department of Earth Sciences, Institute of Mineralogy, University of Hamburg, Hamburg, Germany.

Corresponding author: Arndt Klocke, Dr med dent, MS, Department of Orthodontics, Kieferorthopaedie, ZMK-Klinik, Pav. O 53, U.K.E., Martinistr. 52, 20246 Hamburg, Germany (e-mail: klocke@uke.uni-hamburg.de).

tion the brackets in the laboratory, and the glue is removed from the bracket base after the fabrication of the transfer tray.[11] The cyanoacrylate-based adhesive Smartbond® is recommended by the manufacturer for indirect bonding purposes and can be used with the latter technique. Because no information regarding performance of the new adhesive in combination with this technique has been presented so far, the aim of the present study was to investigate shear bond strength of the cyanoacrylate adhesive in indirect bonding.

MATERIALS AND METHODS

Bonding procedure

Eighty freshly extracted bovine permanent mandibular incisors were obtained from a local slaughterhouse and stored in 0.5% chloramine solution before the experiment. The teeth were randomly assigned to four groups of 20 specimens. After cleaning the teeth with a brush and pumice-water slurry at a slow speed, they were embedded in chemically cured dental acrylic (Palavit G, Heraeus Kulzer, Wehrheim, Germany) in plastic cylinders to allow for standardized and secure placement during testing. Maxillary central incisor .018″ slot stainless steel mesh base brackets (Mini Mono, order no. O711-0103, Forestadent, Pforzheim, Germany) were used throughout the study. The average surface area of the bracket base was 13.5 mm².

The indirect bonding technique was performed as follows. An alginate impression of each specimen was obtained and poured in orthodontic stone. On the dry model, the teeth were painted with diluted separating medium and allowed to dry for 24 hours. The bracket base was cleaned with alcohol. In groups A and B water-soluble glue was applied to the bracket base. The bracket was placed on the model, and the glue was allowed to dry for 30 minutes. In groups C and D, Transbond XT® adhesive (3M-Unitek, Monrovia, Calif) was applied to the bracket before placement on the cast and was cured with a halogen curing light (Polylux II, Kavo, Biberach, Germany) for two minutes. This extended curing interval was chosen to achieve complete polymerization of the adhesive on the model.

Transfer trays were made from vinyl polysiloxane impression material (Silagum AV-Putty soft, DMG, Hamburg, Germany). After the transfer tray material had set, the specimens were soaked in warm water for 30 minutes. The transfer trays were removed from the plaster models. In groups A and B, a brush and warm water were used to thoroughly remove the water-soluble glue from the bracket base. In groups C and D, the composite adhesive on the custom bracket base was cleaned by sandblasting with 50 μm aluminium oxide for three seconds.

Seven days after fabrication of the transfer trays the second part of the bonding procedure was performed.[12]

Groups A and B. The teeth were etched with Smartbond® etching gel for 20 seconds, then rinsed thoroughly with wa-

ter. The enamel surface remained wet after rinsing. Smartbond® adhesive was applied to the bracket base. The transfer tray was placed on the tooth, and the tray was held firmly in place for five minutes.

Groups C and D. The teeth were etched with 37% phosphoric acid gel (Ormco, Orange, Calif) for 30 seconds, rinsed thoroughly with water and air-water spray, and dried with compressed air for 20 seconds. Sondhi Rapid Set® sealant (3M-Unitek) was applied according to the manufacturer's recommendations. This material is a chemically cured composite containing the dimethacrylates, Bis-GMA, (2,2-bis[4(2-hydroxy-3-methacryloyloxy-propylogy)-phenyl]propane and TEGDMA and triethylene glycol dimethacrylate. It consists of two components: component A is painted on the tooth, and component B is painted on the custom base of the bracket.

After bonding was completed, the transfer trays were removed and the specimens were stored in distilled water.

Debonding procedure

In groups A and C, debonding was performed 30 minutes after the bonding procedure and groups B and D were debonded 24 hours after bonding. The brackets were debonded with a Zwicki Z2.5 universal testing machine (Zwick, Ulm, Germany) at a cross-head speed of one mm/minute.[13,14] The plastic cylinders with the embedded teeth and the brackets were mounted on a joint and were aligned in the testing apparatus to ensure consistency for the point of force application and direction of the debonding force for all specimens. A stainless steel wire loop (0.020 inches in diameter) was engaged under the occlusal bracket wings to produce a shear-peel force parallel to the bracket base in an occlusogingival direction. The load at failure was recorded.

For each specimen, the substrate surface was examined with an optical stereomicroscope (magnification 10×) and an Adhesive Remnant Index (ARI) was determined[15]:

- 0, no adhesive left on the tooth;
- 1, less than half of the adhesive left on the tooth;
- 2, more than half of the adhesive left on the tooth;
- 3, all adhesive left on the tooth, with distinct impression of the bracket mesh.

ARI scores were assessed by the same operator.

Statistical analysis

To calculate shear bond strength, the debonding forces (N) were converted into stress values (MPa) by taking into account the surface area of the bracket base. Bond strengths of the different groups were compared by two-way analysis of variance (ANOVA) ($P < .05$) with the factors time of bond strength measurement (30 minutes and 24 hours after bonding) and type of adhesive (Smartbond®, Transbond XT® in combination with Sondhi Rapid Set® sealant). A

TABLE 1. Shear Bond Strength (Mean, Standard Deviation) and Weibull Parameters[a]

Group	Mean (MPa)	SD (MPa)	Weibull Modulus	Correlation Coefficient	Characteristic Bond Strength (MPa)	Shear Stress at 10% Probability of Failure (MPa)	Shear Stress at 5% Probability of Failure (MPa)
A Smartbond 30 min	5.44	1.65	3.20	0.971	6.12	3.03	2.42
B Smartbond 24 h	6.92	1.48	4.80	0.986	7.55	4.73	4.07
C Sondhi Rapid Set 30 min	16.16	5.25	2.55	0.959	18.59	7.71	5.81
D Sondhi Rapid Set XT 24 h	14.98	2.85	6.06	0.948	16.13	11.13	9.88

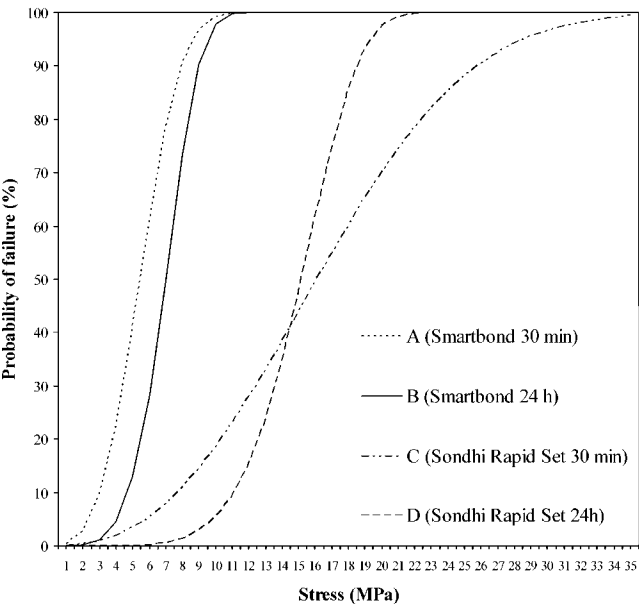[a] MPa indicates mega pascals, SD, standard deviation.

FIGURE 1. Weibull distribution plots. Groups: A, Smartbond® adhesive, debonded 30 minutes after bonding procedure; B, Smartbond® adhesive, debonded 24 hours after bonding procedure; C, Sondhi Rapid Set® sealant, debonded 30 minutes after bonding procedure; and D, Sondhi Rapid Set® sealant, debonded 24 hours after bonding procedure.

Weibull analysis was performed and the Weibull modulus, characteristic bond strength, correlation coefficient, and the stress level at 10% probability of failure were calculated. Kruskal-Wallis and Mann-Whitney nonparametric tests were used to determine if there were any significant differences in the ordinal ARI values ($P < .05$).[16,17]

RESULTS

The mean shear bond strengths, standard deviations, and the parameters of the Weibull analysis (modulus, correlation coefficient, characteristic bond strength, and stress at 10% and 5% probability of failure) are given in Table 1. Figure 1 shows the Weibull distribution plots of the probability of failure at a certain shear stress level for the different groups.

ANOVA indicated that there were no significant differences in bond strength for debonding at 30 minutes and at 24 hours ($F = 0.044$, $P = .834$), whereas bond strengths for the two adhesives were significantly different from each other ($F = 174.115$, $P < .001$). There were no significant interaction effects of the two factors, type of adhesive, and time of bond strength measurement ($F = 3.449$, $P = .067$).

The lowest Weibull modulus (2.55) and highest standard deviation (5.25 MPa) of the four groups was calculated in group C (see Table 1), indicating a wide scatter of the data (see also Figure 1). No enamel fractures were found in any of the specimens. Means, standard deviations, and ranges of the ARI results are given in Table 2. The Kruskal-Wallis test indicated that there were significant differences among the groups ($\chi^2 = 19.875$, $P < .001$). The Mann-Whitney test showed that the ARI score for group D was significantly higher than that for groups A, B, and C. The ARI scores of groups A, B, and C were not significantly different from each other.

DISCUSSION

In the present study, bond strengths for composite and cyanoacrylate adhesives as used in indirect bonding tech-

TABLE 2. Frequency Distribution of Adhesive Remnant Index (ARI)[a] Scores

Group	ARI Scores							
	0	1	2	3	Median	Mean	SD[b]	Range
A Smartbond 30 min	—	16	2	2	1.00	1.30	0.66	1–3
B Smartbond 24 h	—	10	10	—	1.50	1.50	0.51	1–2
C Sondhi Rapid Set 30 min	—	12	8	—	1.00	1.40	0.51	1–2
D Sondhi Rapid Set XT 24 h	—	1	19	—	2.00	1.95	0.22	1–2

[a] Adhesive remnant index: 0, no adhesive left on the tooth; 1, less than half of the adhesive left on the tooth; 2, more than half of the adhesive left on the tooth; 3, all adhesive left on the tooth, with distinct impression of the bracket mesh.

[b] SD indicates standard deviation.

niques were largely different from each other. Thirty minutes after debonding, the mean bond strength with the cyanoacrylate adhesive was only 33.7% (5.44 MPa) of that for the composite adhesive (16.16 MPa), and 24 hours after bonding it was 46.2% (6.92 MPa) when compared with the composite adhesive control group (14.98 MPa). ANOVA showed significantly lower bond strength with the cyanoacrylate material.

So far, in vitro evaluations of direct bonding with cyanoacrylate adhesive have presented conflicting data: Bishara et al[6,7] reported a mean bond strength of 5.8 MPa, 30 minutes after bonding and 7.1 MPa, 24 hours after bonding. The control groups bonded with the composite adhesive Transbond XT® were measured at 5.2 MPa (30 minutes after bonding) and 10.4 MPa (24 hours after bonding). The bond strength values for the cyanoacrylate adhesive are very similar to those obtained in the present study for indirect bonding with the material. However, Bishara et al[6] concluded that both the cyanoacrylate and the composite adhesive that were tested had adequate bond strength at 30 minutes and at 24 hours from initial bonding. Örtendahl and Örtengren[2] compared bond strength with the new material for different types of brackets 24 hours after direct bonding and found mean bond strengths of more than 20 MPa for six out of eight bracket types and of more than 15 MPa for the remaining two bracket types that were investigated. These measurements were significantly higher than those for control groups bonded with a composite adhesive. On the other hand, Al-Munajed et al[8] found significantly lower bond strength measurements of Smartbond® when compared with a composite adhesive and concluded that cyanoacrylate adhesives are unsuitable for use as a bonding agent in routine orthodontic practice. In general, interstudy comparison of bond strength values is difficult because of variation in materials and methods, which have been used in bond strength studies.[18]

In indirect bonding techniques, the adhesive layer thickness has been found to influence bond strength.[19–21] Whereas direct bonding of brackets allows pressure to be exerted directly on the bracket and generally results in close proximity of the bracket base to the enamel surface, the adhesive layer thickness might be increased in indirect bonding where pressure is applied to the transfer tray. Indirect techniques using a custom bracket base allow for a very thin layer of the sealant. However, the use of the cyanoacrylate adhesive does not permit the fabrication of a custom base. Örtendahl and Örtengren[2] emphasized that with the new material it is imperative for the surfaces to be bonded to be as close together as possible because cyanoacrylate cannot fill gaps or spaces. Therefore, increased adhesive layer thickness in indirect bonding might result in reduced bond strength with the cyanoacrylate adhesive.

Mean bond strength measurements might be of limited value for interpreting in vitro bond strength.[22,23] With the use of a Weibull analysis it is possible to predict the prob-

ability of failure of a sample at any level of stress.[24] The Weibull function takes into account the weaker values in the distribution, which are clinically important.[25] When interpreting in vitro data, the force required to cause 5% bond failures may be the type of information that has the most clinical relevance.[26] Littlewood et al[27] suggested that the bond strength of a material with a 5% chance of failure should be at least 5.4 MPa. In the present study, bond strength at a 5% chance of failure was only 2.4 MPa for group A and 4.1 MPa for group B. However, groups C and D, which were bonded with the composite adhesive were characterized by bond strengths higher than 5.4 MPa (5.8 MPa for group C and 9.9 MPa for group D). Therefore, there might be an increased risk of bond failure for indirect bonding with the cyanoacrylate material.

Another method of interpreting Weibull data was given by Hobson et al[13] who based their interpretation of the Weibull analysis on clinically sufficient bond strength levels according to Reynolds[28] and calculated the probability of failure at a stress level of eight MPa. When taking into account this level of shear stress in the composite adhesive control groups, 10.9% (group C) and 1.4% (group D) of bonds are likely to fail. However, for Smartbond® groups A and B, the probability of failure at eight MPa of shear stress is much higher and was calculated at 90.6% (group A) and 73.2% (group B). This means that the majority of bonds are likely to fail at this level of stress. This is in agreement with a recent in vivo investigation of direct bonding, which showed a significantly higher bond failure rate of 22.1% for the cyanoacrylate material compared with 5.1% for the composite resin.[3]

Another concern with the cyanoacrylate adhesive in indirect bonding is the stress on the bond induced during removal of the transfer tray. Because there is a considerable probability of bond failure at moderate levels of stress, it might be advisable to remove the tray very carefully to avoid bond failure. In the present study, no bracket failures were noted on tray removal. However, the experimental set-up may be different from the clinical procedure because single tooth trays were used, which enable very cautious removal of the tray. A multitooth tray commonly used in clinical practice is likely to result in higher forces on the bond during tray removal and hence a higher risk of bond failure.

ARI scores for all groups investigated ranged between one and two except for two specimens in group A, where an ARI score of three was recorded. The median ARI score was significantly higher in group D. The ARI scores indicate that failures with the cyanoacrylate adhesive primarily occurred within the adhesive. This is in agreement with the findings of Eliades et al[1] and Karamouzos et al[3] who attributed a high frequency of cohesive failures of Smartbond® to the presence of reduced network connectivity and bulk discontinuities due to void inclusion.

CONCLUSIONS

- Bond strength for an indirect bonding technique using the cyanoacrylate adhesive was found to be significantly lower than for indirect bonding with a modified Thomas technique using a composite adhesive.
- The Weibull analysis indicated a higher risk of bond failure at clinically relevant levels of stress when the cyanoacrylate adhesive was used for indirect bonding compared with the indirect bonding technique using a composite custom bracket base and a composite sealant.

REFERENCES

1. Eliades T, Katsavrias E, Eliades G. Moisture-insensitive adhesives: reactivity with water and bond strength to wet and saliva-contaminated enamel. *Eur J Orthod.* 2002;24:35–42.
2. Örtendahl TW, Örtengren U. A new orthodontic bonding adhesive. *J Clin Orthod.* 2000;34:50–54.
3. Karamouzos A, Mavropoulos A, Athanasiou AE, Kolokithas G. In vivo evaluation of a moisture-activated orthodontic adhesive: a comparative clinical trial. *Orthod Craniofacial Res.* 2002;5: 170–178.
4. Howells DJ, Jones P. In vitro evaluation of a cyanoacrylate bonding agent. *Br J Orthod.* 1989;16:75–78.
5. Kahl B, König A, Hilgers RD, Schwarze CW. Äthylcyanoacrylat (Cyano-Veneer®) als kieferorthopädischer Bracketkleber. *Fortschr Kieferorthop.* 1993;54:263–267.
6. Bishara SE, Laffoon JF, VonWald L, Warren JJ. Effect of time on the shear bond strength of cyanoacrylate and composite orthodontic adhesives. *Am J Orthod Dentofacial Orthop.* 2002;121: 297–300.
7. Bishara SE, VonWald L, Laffoon JF, Warren JJ. Effect of using a new cyanoacrylate adhesive on the shear bond strength of orthodontic brackets. *Angle Orthod.* 2001;71:466–469.
8. Al-Munajed MK, Gordon PH, McCabe JF. The use of a cyanoacrylate adhesive for bonding orthodontic brackets: an ex-vivo study. *J Orthod.* 2000;27:250–260.
9. Silverman E, Cohen M, Gianelly AA, Dietz VS. A universal direct bonding system for both metal and plastic brackets. *Am J Orthod.* 1972;62:236–244.
10. Thomas RG. Indirect bonding, simplicity in action. *J Clin Orthod.* 1979;13:93–105.
11. White LW. A new and improved indirect bonding technique. *J Clin Orthod.* 1999;33:17–23.
12. Shiau JY, Rasmussen ST, Phelps AE, Enlow DH, Wolf GR. Bond strength of aged composites found in brackets placed by an indirect technique. *Angle Orthod.* 1993;63:213–220.
13. Hobson RS, Ledvinka J, Meechan JG. The effect of moisture and blood contamination on bond strength of a new orthodontic bonding adhesive. *Am J Orthod Dentofacial Orthop.* 2001;120:54–57.
14. Oesterle LJ, Shellhart WC, Belanger GK. The use of bovine enamel in bonding studies. *Am J Orthod Dentofacial Orthop.* 1998;114:514–519.
15. Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. *Am J Orthod.* 1984;85;333–340.
16. Bulman JS, Osborn JF. Significance tests. Part 3. *Br Dent J.* 1989; 166:261–264.
17. Sheats RD, Pankratz VS. Common statistical tests. *Semin Orthod.* 2002;8:77–86.
18. Fox NA, McCabe JF, Buckley JG. A critique of bond strength testing in orthodontics. *Br J Orthod.* 1994;21:33–43.
19. Jost-Brinkmann PG, Schiffer A, Miethke RR. The effect of adhesive-layer thickness on bond strength. *J Clin Orthod.* 1992;26: 718–720.
20. Schiffer A, Jost-Brinkmann PG, Miethke RR. Die Zugfestigkeit von Bracketklebungen in Abhängigkeit von der Kleberschichtstärke-eine in vitro Untersuchung. *Fortschr Kieferorthop.* 1992; 53:297–303.
21. Aguirre MJ, King GJ, Waldron JM. Assessment of bracket placement and bond strength when comparing direct bonding to indirect bonding techniques. *Am J Orthod.* 1982;82:269–276.
22. Reynolds IR, von Fraunhofer JA. Direct bonding of orthodontic brackets, a comparative study of adhesives. *Br J Orthod.* 1976;3: 143–146.
23. Pickett KL, Sadowsky PL, Jacobson A, Lacefield W. Orthodontic in vivo bond strength: comparison with in vitro results. *Angle Orthod.* 2001;71:141–148.
24. McCabe JF, Carrick TE. A statistical approach to the mechanical testing of dental materials. *Dent Mater.* 1986;2:139–142.
25. Johnston CD, Sherry PF. The effects of sandblasting on the bond strength of molar attachments—an in vitro study. *Eur J Orthod.* 1999;21:311–317.
26. Sargison AE, McCabe JF, Gordon PH. An ex vivo study of self-, light-, and dual-cured composites for orthodontic bonding. *Br J Orthod.* 1995;22:319–323.
27. Littlewood SJ, Mitchell L, Greenwood DC. A randomized controlled trial to investigate brackets bonded with a hydrophilic primer. *J Orthod.* 2001;28:301–305.
28. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod.* 1975;2:171–178.