博 士 論 文

A new approach to micro-tensile bond strength: double-ended method

（両端に試験片を設定した新しい微小引張り試験の検討）

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ABSTRACT:

The purpose of this study was to evaluate the micro-tensile bond strength of two universal adhesive systems to enamel by using a new approach: double-ended micro-tensile bond strength test (D-MTBS).

One hundred and ten human third molars were used for this study. Twenty third molars were randomly employed to test 4 groups (n=5) in the traditional micro-tensile bond strength method (MTBS): CLEARFIL Universal Bond self-etch (KUE), CLEARFIL Universal Bond etch-and-rinse (KUS), Scotchbond Universal Adhesive self-etch (3MS) and Scotchbond Universal Adhesive etch-and-rinse (3ME). The rest were used to test in the D-MTBS method where each tooth was cut mesially and distally to make enamel/dentin discs (two discs/tooth). The discs were then made flat at their enamel ends by grinding with #600-grit SiC for 20 s under running water and bonded with each other at their dentin ends with CLEARFIL SE Bond 2 according to the manufacturer's instruction. The enamel ends were then randomly treated with CLEARFIL Universal Bond and/or Scotchbond Universal Adhesive in either etch-and-rinse or in self-etch modes to obtain 6 groups (n=15): KUS VS KUE , 3MS VS 3ME , KUS VS 3MS , KUE VS 3ME , KUS VS 3ME and KUE VS 3MS . After storage at 37°C for 24 h, bond strength of specimens were obtained by subjecting the beams (1 mm^2) to a universal tester at a crosshead speed of 1mm/min. The MTBS data achieved from the traditional method were

analysed with one-way ANOVA followed by Tukey's test (α= 0.05). The D-MTBS data were analysed to evaluate the winners between adhesive systems and their application modes.

The results of MTBS method revealed no significant difference between the adhesives and the application modes (F = 2.068; p > 0.05). However, according D-MTBS results, Etch-and-rinse mode performed better than self-etch mode in case of enamel bonding and CLEARFIL Universal Bond showed better results than Scotchbond Universal Adhesive.

D-MTBS method enabled us to compare two different universal adhesive systems and their different application modes at the same time. Further studies could prove the new double-ended micro-tensile bond strength method as the stepping-stone to the evolution of the next generation micro-tensile bond strength test method.

INTRODUCTION

Recently, a new type of single-step self-etch adhesive categorized as "universal" or "multi-mode" has been introduced and became a new big hot spot. These adhesives have shown high level of comprehensiveness and are accepted by dentists across the globe[1]. Universal adhesives can be used with multiple substrates, such as dentin, enamel, silica-based glass ceramics, zirconia ceramics, and metal alloys[2, 3]. Although they are generally used as single-step systems, an additional etching step prior to the application of the adhesive is often done particularly in case of enamel

bonding[4, 5].

In recent years, many researchers have studied “universal” materials; among them, CLEARFIL Universal Bond and Scotchbond Universal Adhesive are two of the most commonly studied. These research articles fully demonstrate their stability and clinical practicality[6, 7]. With fewer steps the clinicians can save more chair time and for the patients the treatment experience become more comfortable[8].

Until now, many researchers focused on measuring the micro-tensile bond strength of different universal systems in different modes[9-12]. However, the traditional micro-tensile bond strength test method (MTBS)[13] often fails to differentiate between the performances of the adhesives along with their different application modes, especially when the bond strength values are close. Moreover, with MTBS, only one adhesive can be tested with one specimen. But these drawbacks could be avoided if two adhesive systems (or application modes) could be tested with a single specimen as if two gladiators are facing off in an “Arena”.

Therefore, the purpose of this study was to evaluate the bond strength of two universal adhesive systems to enamel by comparing the traditional micro-tensile bond strength method (MTBS) with a new double-ended micro-tensile bond strength method (D-MTBS). We hypothesized that the D-MTBS will be able to compare two adhesive sytems and their different application modes at the same time with single test specimens.

MATERIALS AND METHOD

The adhesive systems used in this study are shown in Table 1.

Selection and Preparation of teeth for bond strength test:

This study was approved by the Hokkaido University Ethical Committee (#2013-7).One hundred and ten sound human third molars were used for this study.

Twenty human third molars were randomly employed to 4 test groups (n=5) for MTBS method (Fig. 1): CLEARFIL Universal Bond self-etch (KUE), CLEARFIL Universal Bond etch-and-rinse (KUS), Scotchbond Universal Adhesive self-etch (3MS) and Scotchbond Universal Adhesive etch-and-rinse (3ME). Each tooth was ground mesially or distally with 600-grit SiC under running water for 60 s to expose flat enamel surfaces. Each adhesive was used as per the respective manufacturer's instructions, to be followed by resin composite (Clearfil AP-X, A3 Kuraray Noritake Dental Inc., Tokyo, Japan) build-up. For the etch-and-rinse groups, enamel etching was done for 15 seconds, rinsed with water and then dried prior to the application of the adhesive. After storage in water at 37°C for 24 h, resin-bonded teeth were sectioned perpendicular to the adhesive interface to produce beams, using the IsoMet diamond saw under cool water lubrication. A total of 30 beams (cross-sectional area: 1 mm×1 mm) per group were tested.

The rest ninety human third molars were used to test in the D-MTBS method (Fig. 2) where each tooth was cut mesially and distally to make enamel/dentin discs (two discs/tooth). The discs were then made flat at their enamel ends by grinding with #600-grit SiC for 20 s under running water and bonded with each other at their dentin ends with CLEARFIL SE Bond 2 according to the manufacturer's instruction. The enamel ends were then randomly treated with CLEARFIL Universal Bond and/or Scotchbond Universal Adhesive in either etch-and-rinse or in self-etch modes to obtain 6 test groups (n=15 teeth): KUS VS KUE, 3MS VS 3ME , KUS VS 3MS , KUE VS 3ME , KUS VS 3ME and KUE VS 3MS . After storage at 37° C for 24 h, bond strength of specimens were obtained by subjecting the beams (1 mm^2) to a universal tester at a crosshead speed of 1 mm/min. A total of 50 beams per group were tested.

Micro-tensile bond strength test:

Each specimen was attached to the jig with a cyanoacrylate adhesive (Model Repair II Pink, Dentsply- Sankin, Tokyo, Japan). Micro-tensile bond strength test was carried out at a crosshead speed of 1 mm/min (EZ Test, Shimadzu Co., Kyoto, Japan) until failure occurred. Bond strength was expressed in MPa. The data achieved from the traditional method were analyzed with one-way ANOVA followed by Tukey's test ($\alpha = 0.05$). The D-MTBS data were analyzed to evaluate the winners between adhesive systems and their application modes.

RESULTS

MTBS:

As shown in Table 2, the results of MTBS method revealed no significant difference between the adhesives and the application modes ($F = 2.068$; $p > 0.05$).

D-MTBS:

D-MTBS results are shown in Fig. 3. Our results indicated that, etch-and-rinse mode performed better than self-etch mode in case of enamel bonding and CLEARFIL Universal Bond showed better results than Scotchbond Universal Adhesive.

DISCUSSION

The MTBS method failed to show significant differences between the tested universal adhesives along with their different application modes used in this study (Table 2; $p > 0.05$). With MTBS only one material and application mode can be tested with one specimen. The method often fails to differentiate between materials which show similar bond strength values. Wellington and his research team also reported same obsevations[14]. In terms of chemical composition both CLEARFIL Universal Bond and

Scotchbond Universal Adhesive are very similar (Table 1). We presume this similarity resulted in their indifferent bond strength values in the current study.

With the D-MTBS method we could compare both adhesives at the same time with their different application mode (Fig. 3). Consequently, we had a winner for each beam leading to a clear winner for each group. Our D-MTBS results suggested that etch-and-rinse mode performed better than self-etch mode in case of enamel bonding. Similar results were reported by Grégoire and his coworkers[15]. The etching step prior to the application of adhesive might have helped to deminerlized the enamel. The ethanol contained in both adhesive systems helped to keep other constituents in solution and rewetted the surface. Moreover the ready penetration of ethanol through the deminerlized collagen by capillary forces was accompanied by monomers.

D-MTBS test results also suggested that KU bonded better than 3M with enamel (Fig. 3). Clearfil Universal Bond (pH 2.3) is more acidic than Scotchbond Universal Adhesive (pH 2.7). We presume this increased acidity is the reason behind Clearfil Universal Bond's winning over Scotchbond Universal Adhesive, especially in case of bonding with enamel. Others studies also support this observation[16-20].

Based on the results of this study we proved that D-MTBS could not only differentiate between two similar materials at the same time, also compared their different application modes. Universal adhesives as single-step self-etch adhesives are much more convenient to use. However, according to our findings and reports from other studies[21, 22], etch-and-rinse mode should be given priority for clinical use, especially in

case of enamel bonding.

In the future, efforts should be given to find out suitable analyses to show significant differences between adhesives tested in D-MTBS methods.

CONCLUSION

The D-MTBS method enabled us to compare two different universal adhesive systems and their different application modes at the same time. Further studies could prove the new double-ended micro-tensile bond strength method as the stepping-stone to the evolution of the next generation micro-tensile bond strength test method.

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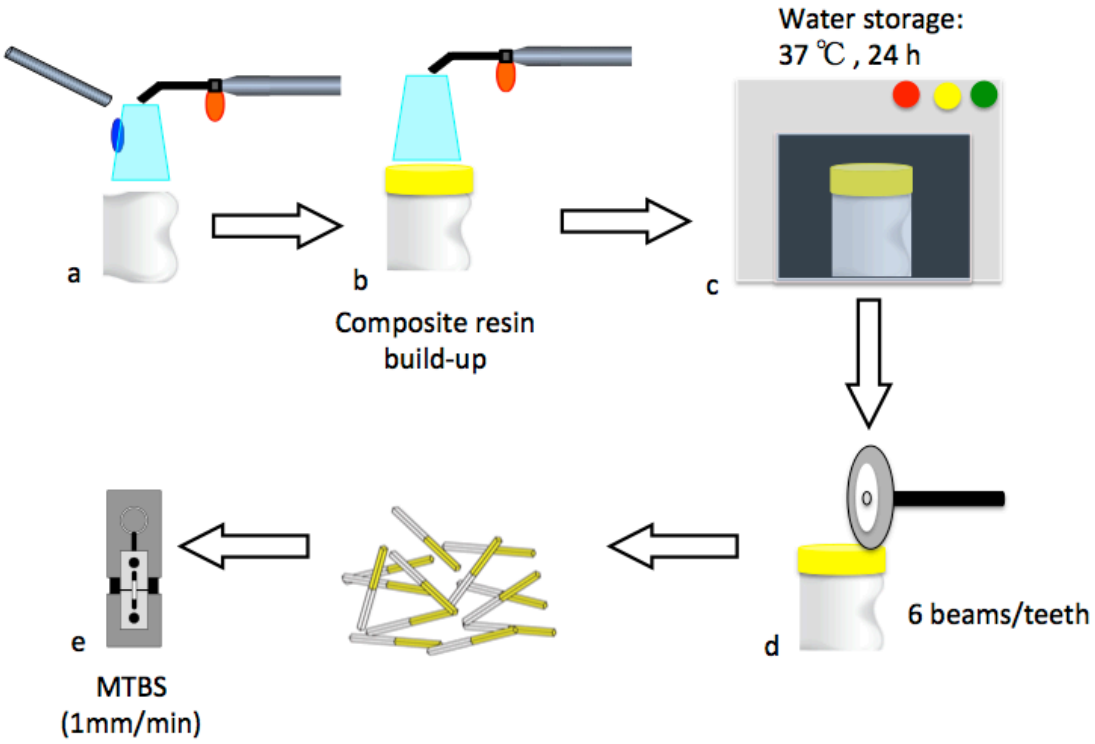

Fig. 1- Schematic illustration of the MTBS method. a. Each adhesive was used as per the respective manufacturer's instructions, b. resin composite build-up (Clearfil AP-X, A3 Kuraray Noritake Dental Inc., Tokyo, Japan), c. storage in water at 37°C for 24 h, d. Resin-bonded teeth were sectioned perpendicular to the adhesive interface to produce beams (6/teeth), using the IsoMet diamond saw under cool water lubrication and e. bond strength of specimens were obtained by subjecting the beams (1 mm^2) to a universal tester at a crosshead speed of 1mm/min.

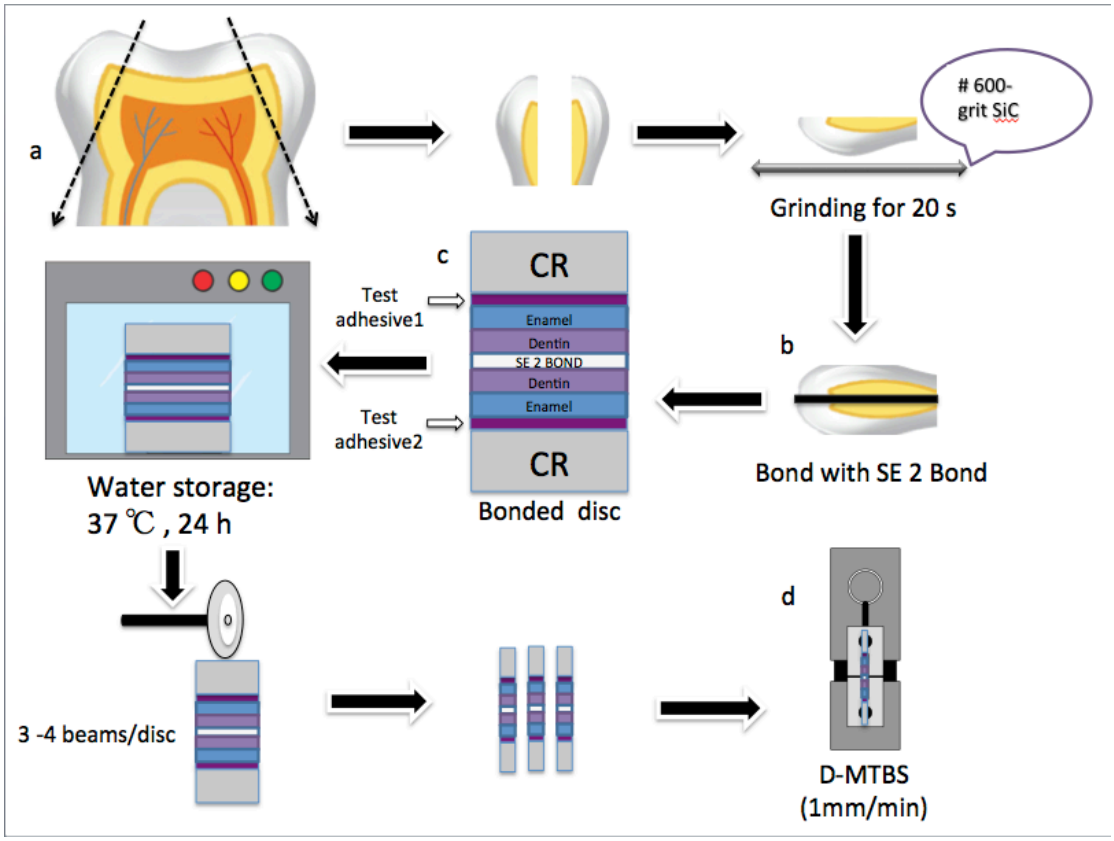

Fig. 2- Schematic illustration of the D-MTBS method. a. Each tooth was cut mesially and distally to make enamel/dentin discs (two discs/tooth), b. the discs were bonded with each other at their dentin ends with CLEARFIL SE Bond 2, c. the enamel ends were treated with CLEARFIL Universal Bond and/or Scotchbond Universal Adhesive in either etch-and-rinse or in self-etch modes and d. bond strength of specimens were obtained by subjecting the beams (1 mm^2) to a universal tester at a crosshead speed of 1mm/min.Until one of the adhesives tandard deviationsfailed.

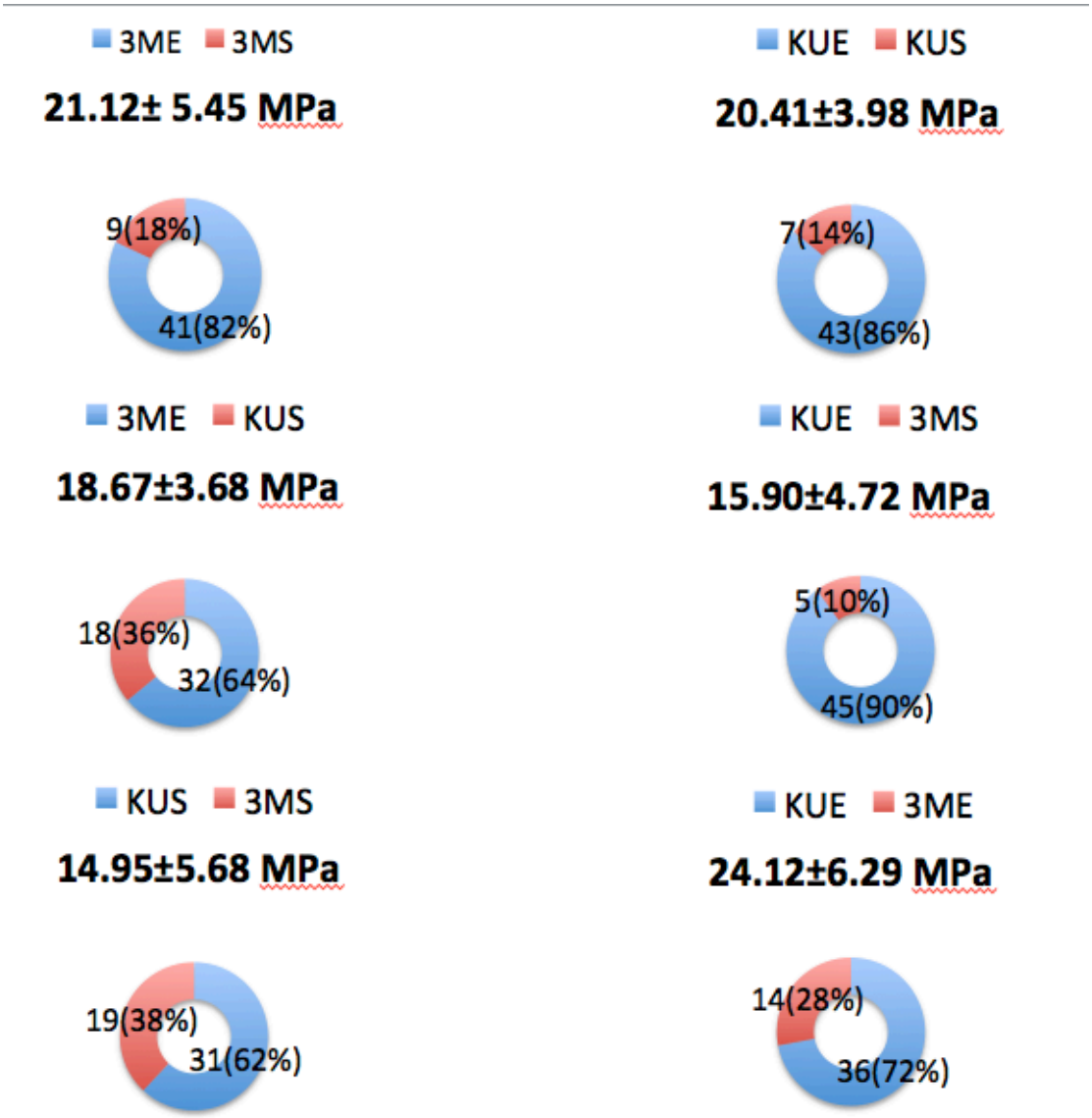

Fig. 3- Mean bond strength in MPa ± standard deviations (SD) of the tested adhesives in D-MTBS method. The proportional plotting graphs show the number and percentage of failures.

Adhesive (Code/Manufacturer/ Lot number)	Type	Chemical formulation	Application technique
CLEARFIL Universal Bond (KU/Kuraray Noritake Dental Inc., Tokyo, Japan/1L0003)	Universal	10-MDP, Bis-GMA, HEMA, ethanol, water, Silane coupling agent, fillers, Initiators.	1.Apply the adhesive and rub for 10 s. 2.Dry gently by blowing mild air for > 5 s until the adhesive no longer moves. 3.Light cure for 10 s.
Scotchbond Universal Adhesive (3M/3M ESPE, St Paul, MN, US/572054)	Universal	10-MDP,Bis-GMA,HEMA, ethanol, water, dimrthacrylate resin, methacaylate-modifiend polyalknoic acid copolymer, polyacrylic acid copolymer, silane, fillers, Initiators.	1.Apply the adhesive and rub for 20 s. 2.Dry gently for about 5 s until it no longer moves and the solvent evaporates. 3.Light cure for 10 s.
CLEARFIL SE BOND 2 (SE2/Kuraray Noritake Dental Okayama, Japan/000003)	Two-step Self-etch	**Primer**:10-MDP, HEMA, Hydrophilic aliphatic dimethacry, CQ DEPT, water **Bond**:10-MDP, Bis-GMA, HEMA, Hydrophobic aliphatic dimethacrylate, CQ DEPT	1.Apply the primer and leave for 20 s. 2.Gentle air-blowing for > 5 s. 3.Apply the bond. 4.Gentle air-blowing to make the film uniform. 5.Light-cure for 10 s.

Bis-GMA: bisphenol-A-diglycidyl methacrylate; CQ: camphorquinone; HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacryloxydecyl dihydrogen phosphate.

Table 1- Composition and application instructions of the adhesives used.

Adhesives and application modes	MPa ± SD
KUE	21.21 ± 5.63
3ME	19.03 ± 3.56
KUS	20.09 ± 6.19
3MS	18.19 ± 4.07

Tukey's test; $p > 0.05$
SD: Standard deviation, MPa: Megapascal

Table 2- Mean bond strength in MPa ± standard deviations (SD) of the tested adhesives in MTBS method.