Shear Bond Strength of Different Functional Monomer in Universal Adhesives at the Resin Composite/Base Metal Alloys Interface

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Abstract

This study examined the shear bond strength (SBS) of four universal adhesives: Single Bond Universal, Clearfil Universal Bond, OptiBond Universal and G-Premio Bond, at the resin composite/base metal alloys (RC/BMA) interface with positive (Alloy primer + Adper Simgle Bond 2) and negative (Adper Single Bond 2 alone) controls. Using a randomized control group design, we enrolled a sample of 60 metal alloys. The predictor variable was adhesive, which bonded RC to BMA (10 specimens per group). All specimens were incubated at 37 °C in distilled water for 24 hours. The SBS test was performed. The failure mode was analyzed using stereomicroscope. The data were statistically analyzed using one-way ANOVA and Tukey's test ($P<0.05$). The highest to least SBS ranged, as follows: (1) Alloy Primer + Adper Single Bond 2, (2) Clearfil Universal Bond and G-Premio Bond, (3) Single Bond Universal and OptiBond Universal and (4) Adper Single Bond 2 alone ($P<0.05$). Adhesive failure was found in all negative control specimens, but < 40% in the other groups failed adhesively. The SBS of the universal adhesives are between those of the positive and negative controls. The universal adhesives may be alternative to the positive/standard control for BMA treatment because of its ease and less working time.

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Introduction

Porcelain fused to metal crowns/bridges may be broken because of trauma, parafunctional habits, inadequate tooth preparation or occlusal adjustment.[1,2] Repair of this fracture is often difficult. Apart from mechanical retention, e.g. abrasion with aluminum oxide,[3] chemical adhesion using metal primers to fix base metal alloys (BMA) with resin composite during repair of porcelain fused to metal crowns/bridges.[4-7] The often-used primers include carboxylic monomer, 4-methacryloyloxyethyl trimellitate anhydride (4-META) and phosphate monomers, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) or glycerol phosphate dimethacrylate (GPDM). These monomers contained in commercially available metal primers were effective for improving the bond strength of resin materials to BMA. 4-META-based primers provide higher bond strength than 10-MDP-containing primers.[5,8]

The latest-generation universal adhesives have been marketed over the past decade. With the "all-in-one" concept, their multi-approach capability enables the clinician to repair porcelain fused to metal crowns/bridges with RC easily and quickly.[9] However, shear bond strength (SBS) of the universal adhesives at the RC/BMA interface remain underreported in the literature. Most of the published studies examined bond strength of universal adhesives to tooth structures such as dentine.

The principal aim of this fracture mechanic-based investigation was to compare SBS of four universal adhesives (Single Bond

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Universal, Clearfil Universal Bond, OptiBond Universal and G-Premio Bond) at the RC/BMA interface with positive control (Alloy Primer + Adper Single Bond 2) and negative control (Adper Single Bond 2 only). We hypothesized that (1) the tested universal adhesives provided comparable SBS with the positive control, (2) their SBS was higher than the negative control.

Materials and methods

Specimen preparation

This was a randomized control group study using 60 BMA disk specimens (10.0 mm in diameter, 4.0 mm in thickness; Alloy Name: N.P.(V) with 72% Ni, 15% Cr, 9% Mo, 2% Al, 1.8% Be and <3% of Fe, Si, C; Dental Art Lab, Bangkok, Thailand). We used this alloy to simulate a metallic part of porcelain fused to metal crowns/bridges.

At first, each specimen was embedded in polyvinyl chloride pipe with dental gypsum. The specimen's surfaces were polished with 600 grit Silicon carbide abrasive paper (3M Wetordry Abrasive Sheet, 3M, MN, USA) followed by ultrasonic cleaning for 10 min. in distilled water and subsequently dried with oil-free air from a triple syringe for 10 sec. Metals differs from dentin because it elicits no moisture during the surface pretreatment. Hence, the dryness of BMA was supposed to be easily identified and standardized with human eyes, as also described by other investigators.[4]

We selected the four universal adhesives (Single Bond Universal, Clearfil Universal Bond, OptiBond Universal, G-Premio Bond) to be test groups because they bond to BMA very well and become popularized in dental practice worldwide. The specimens treated by Alloy Primer + Adper Single Bond 2, a standard adhesive for porcelain fused to metal crowns/bridges repair, served as positive controls, while negative controls were the specimens conditioned by Adper Single Bond 2. Table 1 showed types, brand names, manufacturers' details, lot numbers and chemical composition of the adhesives used in this study.

Ten specimens were randomized into each group. All of them, except positive controls, were treated with one adhesive according to their group using a microbrush, and then dried with oil-free air from a triple syringe for 10 sec., and then light cured for 20 sec. (Elipar FreeLight 2 LED Curing Light, intensity 1000 mw/cm^2, 3M ESPE, MN, USA). The positive control samples were first conditioned with Alloy Primer using a microbrush, and then dried with oil-free air from a triple syringe for 10 sec., and subsequently treated with Adper Single Bond 2; thereafter, dried again in the same manner for 10 sec. before being light-cured for 20 sec.

Material	Composition
Singlebond universal (3M, Deutschland GmbH, Neuss, Germany) Lot: 483316	MDP, Bis-GMA, HEMA, DMA, methacrylate functional copolymer, silane, filler, initiators, ethanol, water
Clearfil universal bond (Kuraray Noritake Dental Inc., Okayama, Japan) Lot: 4K0025	MDP, Bis-GMA, HEMA, colloidal silica, ethanol, silane, sodium fluoride, camphoquinone, ethanol, water
Optibond universal (Kerr Corporation, California, USA) Lot: LK02785	GPDM, GDM, HEMA, dimethacrylate, acetone, ethanol
G-premio bond (GC Corporation, Tokyo, Japan) Lot: 1611221	4-MET, MDP, MDTP,HEMA, dimethacrylate, ethanol, acetone
Single bond 2 (3M ESPE, St. Paul, Minnesota, USA) Lot: N378816	Bis-GMA, HEMA, DMA, methacrylate functional copolymer, filler, photoinitiators, ethanol, water
Alloy primer (Kuraray Noritake Dental Inc., Okayama, Japan) Lot: 580093	MDP, VBATDT, acetone

Table 1. Materials used in the study.
10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; *Bis-GMA,* bisphenol A-glycidyl methacrylate; *HEMA,* 2-hydroxyethyl methacrylate; *DMA,* dimethacrylate; *GPDM,* glycerol phosphate dimethacrylate; *GDM,* 1,3-glycerol dimethacrylate; *4-MET*, 4-methacryloxyethyl trimellitic acid; *VBATDT,* 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithion.

Pieces of polyethylene adhesive tape (ScotchBlue Painter's Tape, 3M ESPE, MN, USA) approximately 80 microns in thickness with 2.0 mm diameter circular holes were placed on the pretreated surfaces of the specimens to help demonstrating the bonding area. A silicone mold (3.0 mm in diameter, and 2.0 mm in thickness) was placed on the adhesive tape top, and then filled with RC (Filtek Z350 XT (A3E), 3M ESPE, MN, USA), and light-cured for 40 sec afterwards. The bonded specimens were kept at room temperature for 30 min. and afterwards in distilled water in an incubator (Incubator; Contherm 160M, Contherm Scientific Ltd., Lower Hutt, New Zealand) at 37°C for 24 hours.

Shear bond strength test and surface analysis

The SBS was measured using a universal testing machine (EZ-S 500N, Shimadzu corporation, Kyoto, Japan) with the load applied in the direction parallel to the RC/BMA interface at a crosshead speed of 0.5 mm/min. (Figure. 1). The SBS was calculated by dividing the highest bond strength by the surface area of the RC/BMA interface. The debonded interfaces were examined using a stereomicroscope

(ML9300 Trinocular Polarizing Microscope, Meiji Techno Co. Ltd., Saitama, Japan) at x40 magnification to study the failure mode.[10] The failure mode was classified into 3 types: adhesive failure at the RC/BMA interface; cohesive failure within the RC substance; and mixed failure were a combination of both.

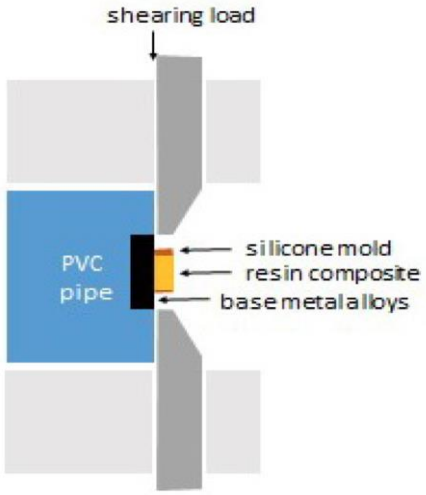

Figure 1. Shear test configuration.

Statistical analysis

The data were collected during the study period using a standardized collection form and entered into a statistical database (Statistical Package for Social Sciences, version 13.0; SPSS, Chicago, IL, USA) for analyses. The data were statistically analyzed using one-way ANOVA and Tukey's test. For all analyses, $P<0.05$ was considered statistically significant.

Results

The highest and least of SBS was in a range as follows: (1) Alloy Primer + Adper Single Bond 2, (2) Clearfil Universal Bond and G-Premio Bond, (3) Single Bond Universal and OptiBond Universal and (4) Adper Single Bond 2 alone (*P<0.05*). In this cohort, no specimen showed cohesive failure. G-Premio Bond had significantly higher SBS and lower percentages of adhesive failure than Single Bond Universal and OptiBond Universal (*P<0.05*).

However, there was no statistical difference between Clearfil Universal Bond and G-Premio Bond, as well as between Single Bond Universal and OptiBond Universal. This suggested that a combination of carboxylic monomer and phosphate monomer in case of G-Premio Bond neither improved the SBS nor reduced the weakness of this adhesive. Details were also summarized in Table 2.

Group	Mean bond strength (SD)	Failure mode		
		Adhesive	Mixed	Cohesive
1. Single Bond Universal	14.68 (1.56)[a]	40	60	0
2. Clearfil Universal Bond	19.45 (1.09)[b]	20	80	0
3. OptiBond Universal	13.77 (0.88)[a]	40	60	0
4. G-Premio Bond	19.52 (1.35)[b]	20	80	0
5. Positive control	23.95 (1.35)[c]	0	100	0
6. Negative control	9.45 (1.21)[d]	100	0	0

Table 2. Mean bond strength, standard deviation (Megapascal) and failure mode (%).
The value with identical letters indicates no statistically significant difference.

Discussion

Shear forces always affect a restoration during all of its service life, and coupled with tensile and compressive forces, can cause the restoration failure. Many factors also deteriorate a restorative material, for example, temperature variations, restoration type and size and tooth factors. Hence, evaluation of the survival rate of a dental material based on the effect of "pure" shear forces only seems unreasonable. However, it has generally been accepted that SBS is one important indicator of mechanical performance before *in vivo* studies and clinical use of an adhesive.[11] The main purpose of this experiment was to compare SBS and failure mode of 4 universal adhesives at the RC/BMA interface with two standard controls.

The results of this study reflected the incomparable properties of adhesives. Using the SBS test, the positive control was the most durable adhesive, promoting the use as a standard agent. However, its bonding efficacy decreases dramatically, when Alloy Primer is used alone.[12] Although 10-MDP initiates strong adhesion,[13] the adhesive layer created by Adper Single Bond 2 is necessary for the adhesion to be stabilized.[12]

Our core interest was directed to the four universal adhesives: Single Bond Universal, Clearfil Universal Bond, OptiBond Universal and G-Premio Bond. We found that SBS and favorable failure mode were not linked to functional monomer types of the adhesive. The mechanical properties of G-Premio Bond and Clearfil Universal Bond were not significantly different, suggesting no synergistic role of carboxylic monomer in combination with

phosphate monomer. Nevertheless, caution must be applied because compositions other than the functional monomers in both adhesives are not identical. The GPDM-based adhesive, OptiBond Universal, had the poorest SBS and failure mode. It may be due to the fact that adhesion of GPDM to BMA is less effective than the bonding of 10-MDP.[14]

In the 10-MDP-containing adhesive group Alloy Primer + Adper Single Bond 2, Single Bond Universal, Clearfil Universal Bond and G-Premio Bond exhibited brand-dependent mechanical properties. The latter two had better adhesive performance than Single Bond Universal, but was not superior to the positive control. Llerena-Icochea et al,[15] found a nonlinear correlation between SBS and percentage of 10-MDP in the adhesives. They measured the SBS of the adhesives to zirconia ceramic, whose oxide layer can be directly bonded with 10-MDP. Our findings were different because we studied the SBS at the RC/BMA interface. According to Yang et al,[16] 10-MDP-based primers resisted to the negative effect of thermocycling and water storage on the SBS, this preparation technique was also used in our study. Had our finding supported the Yang et al's results,[17] no significant difference would have been found within this subgroup. Hence, the adhesive durability against shear forces is unlikely to depend mainly on the presence of 10-MDP in the adhesives. A possible explanation is that each adhesive contains different 10-MDP impurities.[17]

The use of Adper Single Bond 2 only (negative control) was significantly linked to the least SBS, and all specimens in this group failed adhesively. The possible explanation is that this conventional adhesive lacks the functional monomers, which is necessary for bonding to the oxide layer of BMA and therefore yields very low durability.[13] It is, therefore, unsuitable for the RC/BMA interface. In contrast, our findings encourage the use of Alloy Primer + Adper Single Bond 2 to indirectly repair porcelain fused to metal crowns/bridges because it owned the best SBS. The other universal adhesives seem appropriate in some situations only, for example, direct repair of porcelain fused to metal crowns/bridges in a handicapped or pediatric patient who cannot tolerate long dental treatment.

Apart from mechanical properties, the biocompatibility of adhesives attracts much attention from investigators. Bench researches showed cytotoxicity of the adhesive monomers, for example, bisphenol A-glycidyl methacrylate (Bis-GMA) and 2-hydroxyethyl methacrylate (HEMA) in Adper Single Bond 2, Single Bond Universal and Clearfil Universal Bond, as well as 10-MDP in Single Bond Universal, Clearfil Universal Bond and G-Premio Bond. These compounds induce the formation of reactive oxygen species and deplete antioxidants, e.g. glutathione, resulting in changes in cellular microenvironments. The increased reactive oxygen species levels hamper the cell-death control with antioxidant genes and proteins. Bis-GMA and HEMA exhibit cell-growth suppression and cell-cycle alteration, and increase oxidative stress and apoptosis. However, functional monomers in universal adhesives were found to be less *in vitro* toxic than monomers of the conventional agents. Despite uncertain clinical relevance, this observation may highlight another advantage of universal adhesives over the conventional systems.[18] In other word, it is reasonable to apply rubber dam isolation when the universal adhesives are used in direct repair of porcelain fused to metal crowns/bridges. In addition to preventing moisture contamination, which eliminates adhesive polymerization, rubber dam application depletes the possible contact of human tissues with adhesives and hydrofluoric acid (that is used during surface pretreatment of porcelain). The principle of non-maleficence: *"First, do no harm",* should be employed until proved otherwise.

To the best of our knowledge, this investigation was the study comparing the SBS and failure mode of the adhesives with different functional monomers at the RC/BMA surface. However, there are some study limitations. First, the *in vitro* experimental study design threatens the generalizability or external validity of the study's results in clinical practice. Second, adhesives and BMA may differ from one lot to another. Manufacturing standardization is beyond the scope of this study. Lastly, as mentioned above, SBS is not the only factor influencing the clinical survival rate of an adhesive. Taken all together, our study's results should be interpreted with cautions.

Conclusions

The SBS of universal adhesives at the RC/BMA interface are ranged between the

positive/standard control 'Alloy Primer + Adper Single Bond 2' and the negative control as Adper Single Bond 2. These properties are brand-dependent, regardless of the presence and types of functional monomer. The tested universal adhesives are an alternative to the positive/standard control for BMA surface treatment because of its ease and reduced working time.

Declaration of Interest

The authors report no conflict of interest.

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