Original Articles

Microtensile Bond Strength of Repaired Ceramic Using Resin Composite with Universal Adhesive System Compared to Conventional Bonding System *In Vitro*

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Abstract

The effectiveness of 2 types of ceramic repaired using resin composite and a universal adhesive were compared to a conventional adhesive. Leucite-reinforced glass ceramic ingots (IPS Empress® Esthetic; "EE"; Ivoclar Vivadent, Germany) and lithium-disilicate glass ceramic ingots (IPS e.max® Press; "EM"; Ivoclar Vivadent, Germany) were fabricated into 8x8x4 mm ceramic blocks with a total number of 288. The ceramic surfaces were wet-polished with silicon carbide paper and then treated with 9.5 % hydrofluoric acid (Ultradent® Porcelain Etch; Ultradent, USA). Resin composite (Filtek™ Z350 XT, shade A4; 3M ESPE, USA) was built-up with 2 adhesive systems, one half ("U") using universal dental adhesive (Single Bond™ Universal; 3M ESPE, USA) and the other ("C") using total etch dental adhesive (Adper™ Scotchbond™ Multipurpose Plus; 3M ESPE, USA) combined with ceramic primer (Relyx™ Ceramic Primer; 3M ESPE, USA). The specimens were stored in water at 37°C for 24 hours and then subjected to thermocycling for 10,000 cycles prior to a microtensile bond strength (μTBS) test. The specimens were then divided into a group of 36, for 8 groups, according to type of ceramic, adhesive system, and storage condition. Modes of failure were analyzed using a stereomicroscope (ML 9300; MEIJI, Japan). Three-way ANOVA and a Bonferroni post-hoc test were used to analyze the data (n = 36, α = 0.05). There was no significant difference between the aged and non-aged groups (p = 0.207). However, a Bonferroni post-hoc test revealed significant differences among all tested groups. The highest μTBS was recorded by the "EMC" group (36.310±13.12), while the lowest was found in the "EEU" group (22.020±7.94). The μTBS between the resin composite and ceramic repaired using a conventional adhesive system was higher compared with a universal adhesive system, especially in the lithium-disilicate type.

Keywords: Microtensile Bond Strength, Repaired Ceramic, Thermocycling, Universal Bonding

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Introduction

Esthetics is a major concern in many aspects of daily life, and possibly even more so in dentistry. Dental ceramics are restorative materials that fulfill esthetic requirements in both form and functional aspects. Due to their strength and esthetic properties, ceramics became popular for dental restorations including anterior crowns, veneers, and veneers on substructures.[1] Many classes of ceramic systems are available including feldspathic glass, leucite-reinforced glass, lithium-disilicate glass, and zirconia.

In the past, feldspathic glass ceramic was most commonly used for veneer restorations as its excellent optical properties, translucency, and color; resulted in a natural appearance. Later, leucite-reinforced glass ceramic was developed, composed of about 45 % leucite by volume. IPS Empress® Esthetic (Ivoclar Vivadent, Germany) is one example of commercialized leucite-reinforced ceramics that are most widely used nowadays. This material has flexural strength around 100-120 MPa, with such low strength, the clinical indication of leucite-reinforced ceramic is limited to anterior teeth restoration.[2] Another type of ceramics was developed, containing 65 % of lithium disilicate by volume, lithium-disilicate glass ceramic has flexural strength around 350-450 MPa which is sufficient for 3-unit fix partial denture prosthesis in anterior region.[3]

Despite both providing favorable esthetic result, ceramics have greater strength and durability compared to resin composites. Even so, fractures of ceramic restorations sometimes occur.[4-5] Replacing the fractured ceramic restoration with a new one is the treatment of choice; however, in most situations, patients decline this treatment, due to time constraints and/or financial limitations. In these cases, repairing the ceramic with resin composite can be an attractive alternative treatment.[6]

The ceramic repair procedure begins with surface conditioning which can be performed using many techniques including etching the ceramic surface with 2.5 %, 4.9 %, 5 %, 9.5 %, 9.6 %, 10 % hydrofluoric acid[7-10], sandblasting the ceramic surface with aluminum oxide particles[11], etching the ceramic surface with 1.23 % acidulated phosphate fluoride[12], or silica coating[11]. Although 5 % hydrofluoric acid is particularly suggested as surface conditioning agent by the manufacturer, the gel-like form of 9.5 % hydrofluoric acid can be easily controlled when applying intra-orally for repairing ceramic with resin composite. Moreover, there are many previous studies reported that higher concentration of hydrofluoric acid provided higher bond strength comparing to lower concentration when applying for the same amount of time.[7,13] According to these reasons, 9.5 % hydrofluoric acid was chosen as surface conditioning agent in this study. After surface conditioning, a silane coupling agent is applied in order to promote chemical bonds between organic and inorganic components[14-15], accompanied by an adhesive agent and resin composite. This procedure is known as conventional adhesive system. Disadvantages of this procedure include requiring various products, taking many steps and consuming chair-time, which make conventional adhesive system technique-sensitive. Hence, universal adhesive were developed to resolve these problems.

Many universal adhesives, commercially available now, have been claimed to be able to bond to many substrates such as enamel[16], dentin[16], glass ceramic[16-17] or even zirconia.[18] In this study, Single Bond™ Universal was selected as a representative of universal adhesive system. Due to its composition that differs from the other adhesives, which is the incorporation of silane coupling agent in the bottle, Single Bond™ Universal is able to bond with glass ceramics.[17]

Even though many procedures have been developed for repairing ceramics with resin composites,[19-21] with the performance of each technique intensively investigated, hydrofluoric acid in combination with a silane coupling agent was the most popular method of

choice.[6,19-20,22] However, few studies have compared the microtensile bond strength (μTBS) of the repair using method for two types of ceramic systems. This study compared the performance of a universal adhesive with a conventional adhesive for ceramic repair. Two types of ceramic systems; lithium-disilicate and leucite-reinforced glass ceramics, were chosen.

The null hypotheses tested were: (1) there was no difference in μTBS between repaired ceramic using resin composite with a universal adhesive and conventional adhesive system and (2) there was no difference in μTBS between aged and non-aged groups of repaired ceramic using different adhesive systems.

Materials and Methods

16 ingots of leucite-reinforced glass ceramic (IPS Empress® Esthetic, ETC2; EE; Ivoclar Vivadent, Germany) and 16 ingots of lithium-disilicate (IPS e.max® Press, HTA2; EM; Ivoclar Vivadent, Germany) were fabricated into ceramic blocks with dimensions of 8x8x4 mm (Fig. 1A) using a heat-pressed technique according to manufacturer's instructions. The ceramic blocks were placed in stainless steel holder and passively polished with 240-, 400-, and 600-grit silicon-carbide paper (TOA, Germany) through running water for 2 minutes each, respectively, by grinder-polisher machine (Automet® 250; Buehler, USA). All the ceramic surfaces were treated with 9.5 % hydrofluoric acid (Ultradent® Porcelain Etch; Ultradent, USA), 60 seconds for EE and 20 seconds for EM. Resin composite (Filtek™ Z350 XT, shade A4; 3M ESPE, USA) was used as a build-up material using a silicone mold with dimensions 8x8x4 mm (Fig. 1B), then bonded to the treated ceramic surface according to the manufacturer's instructions. The dimensions of the final specimens of ceramic bonded with resin composite were 8x8x8 mm. Each 2-mm increment was polymerized using a LED light-curing system (Demi™ Plus; Kerr, USA) with 1,100 mW/cm2 intensity for 40 seconds (Fig. 1D). The light guide was held perpendicularly 1 mm above the silicone mold. Light output from the light-polymerizing unit was checked by a radiometer (Model 100 Optilux; Kerr, USA) throughout the experiment.

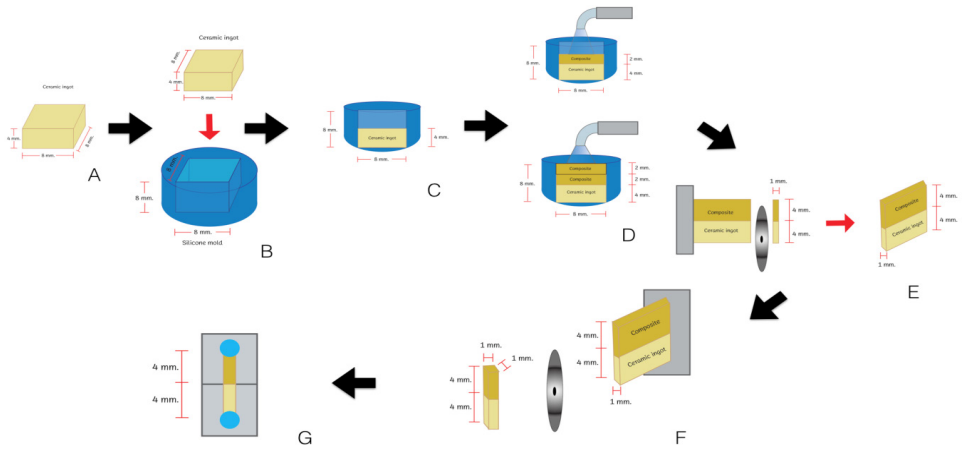

Figure 1 *A) Surfaces of the ceramic ingot with dimensions 8x8x4 mm prepared by silicon carbide paper of various grit, 9.5% hydro fluoric acid, followed by the adhesive procedure according to the manufacturer's instructions.*
B, C) The ceramic ingot was seated inside the silicone mold with 4-mm space for further resin composite buildup.
D) Each 2-mm layer of resin composite was cured by a light-curing unit for 40 seconds.
E) The specimen was then stuck to a plastic block and cut into a slab with dimensions 1x8x8 mm by a diamond wafering blade.
F) The slab was cut into non-trimming bar shapes with dimensions 1x1x8 mm using a diamond wafering blade.
G) The bar-shaped specimen was stuck to an experimental jig for microtensile testing using cyanoacrylate glue.

Following the bonding procedures in Table 1, the specimens were stored in water at 37°C for 24 hours. Thereafter, the specimens were sectioned into slabs with dimensions 1x8x8 mm (Fig. 1E). The specimen was prepared into 1x1x8 mm non-trimming bar-shaped beams. (Fig. 1F), using a low-speed cutting machine at a speed of 350 rpm and loading of 150 g (Isomet® 1000, Buehler, USA) with constant water spray. The bonded specimens were then divided into 8 groups, with 36 specimens in each group according to ceramic type, bonding technique and storage condition. Details of all the groups are shown in table 2.

Table 1: *Materials showing manufacturer, composition, and instructions for use*

Material / Manufacturer	Composition	Procedure following the manufacturer's instructions
RelyX™ Ceramic Primer (lot no. N636821, 3M ESPE, USA)	Methacryloxypropyl trimethoxysilane, ethanol, water	1) Apply 0.04 microliter of primer, measured by micropipette, on the ceramic surface in one direction 2) Allow it to react for 3 mins 3) Blow gently for 10 s, with 2-bar pressure, from 10-mm distance
Adper™ Scotchbond™ Multi-Purpose Adhesive (lot no. 596612, 3M ESPE, USA)	Adhesive: Bis-GMA, HEMA, EMAB, dimethacrylate, initiators	1) Apply 0.04 microliter of adhesive, measured by micropipette, on the ceramic surface in one direction 2) Light-cure for 10 s
Single Bond™ Universal Adhesive (lot no. N553960, 3M ESPE, USA)	Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1) Apply 0.04 microliter of adhesive, measured by micropipette, in one direction and rub it for 20 s on the ceramic surface 2) Blow gently until no movement of liquid with 2-bar pressure, from 10-mm distance 3) Light-cure for 10 s

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate

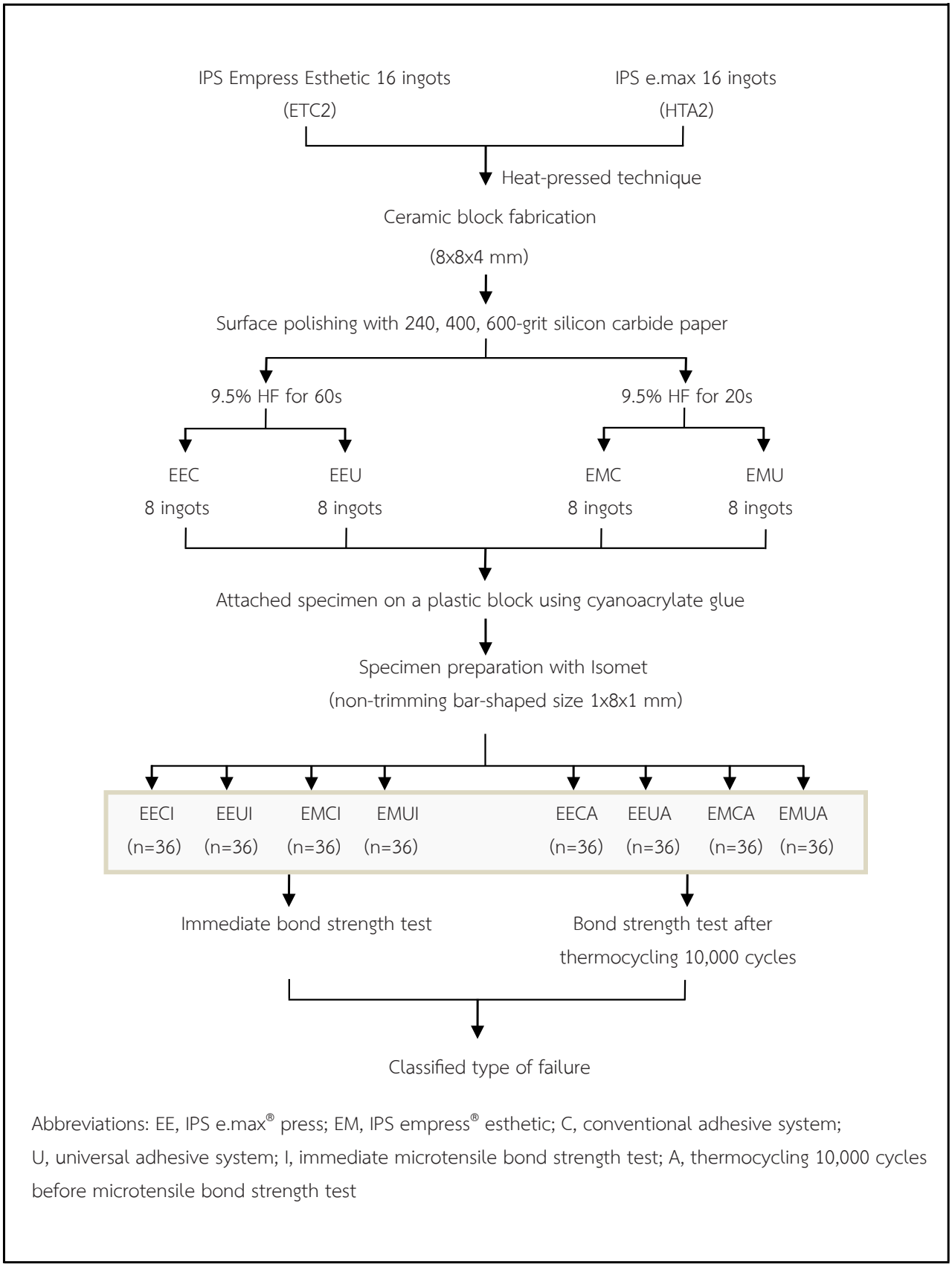

Figure 2 *Diagram of study design*

Specimens were then stuck on an experimental jig for microtensile testing using cyanoacrylate glue (Model Repair II Blue; Dentsply, USA) (Fig. 1G). The μTBS test was performed using a universal testing machine (EZ-S Shimadzu; Shimadzu, Japan) with cross-head speed 1 mm/min and data were recorded in MPa.

The mode of failure was determined using a stereomicroscope (ML 9300; MEIJI, Japan) at a magnification of 40x, and classified into one of four categories as follows:

Type I: Adhesive failure – fracture occurred at the resin-ceramic interface (>50% of failure between resin-ceramic interface)

Type II: Cohesive failure in resin composite – fracture occurred within the resin composite layer (>50% of failure within the resin composite)

Type III: Cohesive failure in ceramic – fracture occurred within the ceramic layer (>50% of failure within the ceramic)

Type IV: Mixed failure – fracture occurred involving both the resin-ceramic interfaces and the neighboring substrates

If any of the specimens were broken prior to test, the bond strength value was recorded as a minimum μTBS of each group.

Data were analyzed using statistical software (IBM SPSS Statistics 20, SPSS). Mean μTBS values were collected and analyzed by three-way ANOVA followed by a Bonferroni post hoc test. Results with *p*-value < 0.05 were considered statistically significant.

Results

A Shapiro-Wilk's test ($p>0.05$) and a visual inspection of their histograms, normal Q-Q plots, and box plots showed that the mean μTBS in all tested groups was approximately normally distributed.

There were no pretest failures in any group. Mean values of μTBS of each group are shown in Table 2. The highest mean μTBS was recorded in the EMCI group and the lowest in the EEUA group.

Table 2: *Mean microtensile bond strengths and number of specimens*

Group	Mean Microtensile Bond Strength (MPa)	Standard Deviation	Number of Specimens (N)
EECI	$28.2^{B,C}$	10.5	36
EECA	26.7^{C}	8.9	36
EEUI	$23.6^{C,D}$	8.6	36
EEUA	20.4^{D}	7.0	36
EMCI	38.3^{A}	13.9	36
EMCA	$34.3^{A,B}$	12.2	36
EMUI	25.3^{C}	6.6	36
EMUA	24.9^{C}	8.1	36

Abbreviations: EE, IPS e.max® press; EM, IPS empress® esthetic; C, conventional adhesive system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling 10,000 cycles before microtensile bond strength test

The overall values of μTBS are shown in Table 2. EMCI and EMCA groups showed significantly highest mean μTBS among the tested group ($P<0.05$), following with EECI, EECA, EMUI, EMUA and EEUI groups respectively ($P<0.05$). EECI group was not significant different from EMCA group ($P>0.05$), and the lowest μTBS was found in EEUA group ($P<0.05$).

Three-way ANOVA results indicated a significant interaction between "bonding" and "ceramic type" ($p=0.013$) (Fig. 3). Thus, the main effect of the two

factors could not be tested. For bonding and ceramic type, EM and EE groups with conventional bonding gave significantly higher μTBS than groups using universal bonding ($p<0.05$). The effectiveness of the conventional adhesive system was higher when using EM ceramic type than EE ceramic type; however, there was no such correlation between the universal bond groups (Fig. 3).

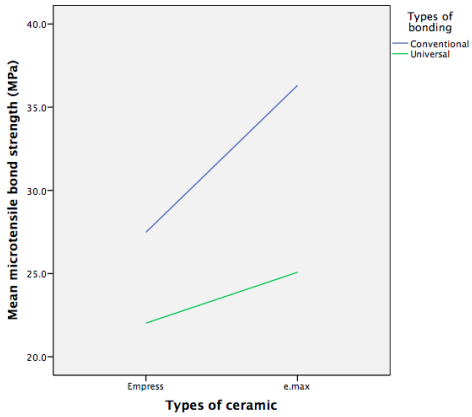

Figure 3 *There was an interaction between the factors "bonding" and "ceramic type" (P-value = 0.013).*

"Aging" was the only factor that did not show any interaction with the others (Fig. 4). Comparing the μTBS between the "immediate" and "aging" groups (Table 3), No significant difference of μTBS was observed between the groups.

Table 3 *Mean microtensile bond strength between "immediate" and "aging" groups*

Group	Number of Specimens (N)	Mean (MPa)	Standard Deviation
Immediate	144	28.9^{A}	11.6
Aging (Thermocycling 10,000 cycles)	144	26.6^{A}	10.4

Upper case letters indicate statistical difference in the row ($p \leq 0.05$).

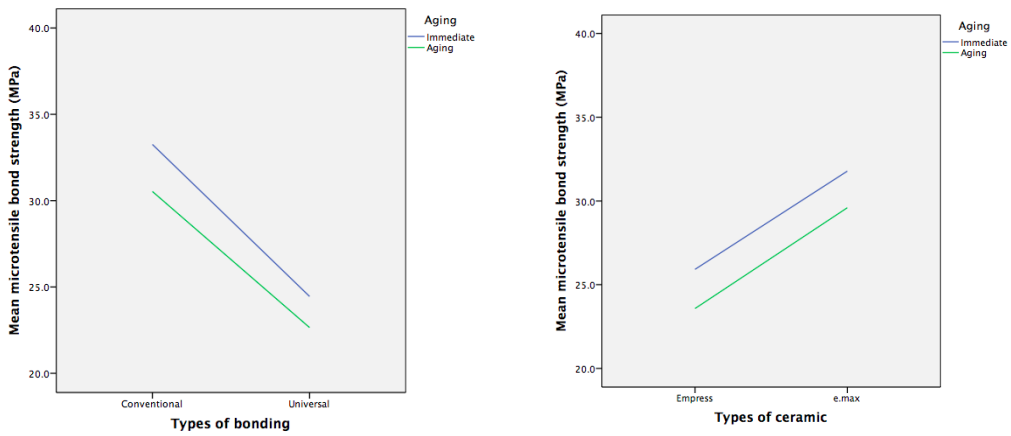

Figure 4 *A showed no interaction between "aging" factor and "bonding" (P-value = 0.689).*
B showed no interaction between "aging" factor and "ceramic type" (P-value = 0.946).

The percentages of failure modes were investigated using a stereomicroscope at 40X magnification. The majority of the failures were adhesive failure between the resin composite and the ceramic (83.34 %), followed by cohesive failure in the resin composite (9.02 %), and mixed failure between the resin composite and the adhesive layer (7.64 %) (Table 4).

Table 4 *Mode of failure of the debonded specimens*

Group	Mode of failure			
	Adhesive	Cohesive (composite)	Cohesive (ceramic)	Mixed failure
EECI	30	3	0	3
EECA	26	5	0	5
EEUI	22	6	0	8
EEUA	35	1	0	0
EMCI	28	5	0	3
EMCA	27	6	0	3
EMUI	36	0	0	0
EMUA	36	0	0	0
Total	240 (83.34%)	26 (9.02%)	0 (0%)	22 (7.64%)

Abbreviations: EE, IPS e.max® press; EM, IPS empress® esthetic; C, conventional adhesive system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling 10,000 cycles before microtensile bond strength test

Discussion

Results indicated that the mean µTBS from the universal adhesive group (Single Bond™ Universal) was low compared with the conventional adhesive system; thus, the first hypothesis, presented that there was no difference in µTBS between repaired ceramic using resin composite with a universal adhesive and conventional adhesive system, was rejected. Silane is known to promote wettability and form flexible siloxane bonds; with one side, the non-hydrolysable group reacting with the carbon-carbon double bond in the resin composite, and the other side, the hydrolysable group reacting with the hydroxyl group on the ceramic surface.[22-23] The incorporation of silane is found in Single BondTM Universal as claimed by the manufacturer. However, there are some studies reported that universal bonding systems containing water and acidic agent caused dehydration condensation of silane [17,23-24], which did not bond with the glass phase of the ceramic surface, causing bond strength reduction.[14,22] Similarly to the study from Kim R. *et al.* (2015) which found that the microshear bond strength of Single Bond™ Universal was not significantly different from that of All-Bond Universal (Bisco, USA), despite the fact that the latter does not contain silane. The microshear bond strength of the two universal adhesives was also lower when compared with conventional adhesive.[17] It was also supported by Yoshihara *et al.* (2016), reporting that when using universal adhesive, the silica glass plate showed lower shear bond strength compared to the fresh silane and bonding agent group.[23] They noted that a suitable pH for silane was 4.6, but the pH of the uni-

versal bonding agent was acidic at 2.7 and possibly caused the silane solution to become unstable and inactive faster with a correspondingly shorter shelf life.[23] A stable siloxane bond requires water for condensation between silanol and the OH group.[22] However, Chen *et al.* (2013) determined that the condensation reaction was inhibited by Bis-GMA in the universal adhesive which slowed down water evaporation. Thus, bond strength and stability of the universal adhesive were inferior compared to conventional adhesive.[25]

It was well-established in many previous studies that surface treatment with 9.5 % hydrofluoric acid following by applying silane when repairing ceramic with resin composite provided the most effective result. The interest of this study was focused on the performance of Single Bond™ Universal, which claimed to have included silane in one bottle, compared with gold standard technique using separated silane and hydrophobic adhesive. Applying only 9.5 % hydrofluoric acid or only silane were not included as negative control groups since the effect of hydrofluoric acid or silane alone was not considered in this study.

An aging process utilizing thermocycling affected the μTBS of ceramics repaired by resin composite.[26-29] Some studies indicated no significant difference of μTBS between testing groups aged using thermocycling.[30-31] ISO TR 11450 standard (1994) states 500 cycles of thermocycling in water between 5°C and 55°C as a suitable condition for the aging test. Gale and Darvell (1999) found that 10,000 cycles of thermocycling were comparable to one year of function *in vivo*.[32] In this study, 10,000 cycles of thermocycling with dwell time of 60 seconds (5°C, 35°C, 55°C, and 35°C for 5, 25, 5, and 25 seconds, respectively) were used to test the performance of the two adhesive systems. Results showed no significant differences from thermocycling on μTBS between the "immediate" and "aging" groups (p=0.083). Therefore, the second hypothesis stating there was no difference in μTBS between aged and non-aged groups of repaired ceramics using different adhesive systems was accepted. Moreover, Foxton *et al.* (2002) stated that hydrolytic degradation weakened the bonding interface after water storage for six weeks [33]; therefore, the aging process used here may not be adequate since the actual storage time was only 10 days.[34]

The EMC group recorded a statistically significant higher μTBS than the EEC group (p<0.001) for repaired ceramics using conventional adhesive; however, this trend was not found in the universal adhesive system. Della Bona *et al.* (2003) reported that the μTBS of lithium disilicate treated with 9.6 % hydrofluoric acid followed with silane was higher compared with leucite-reinforced ceramic under the same conditions as HF did more damage to leucite-reinforced ceramic surface than that of lithium disilcate. As hydrofluoric acid targeted more at the interface between leucite and glass phase, leaving the remaining glass phase impaired, causing μTBS of leucite-reinforced ceramic to be lower.[35]

Shear and μTBS tests are common techniques used for measuring adhesive materials. But the advantages of microtensile bond strength test over shear bond strength test is that stress distribution is more focused in adhesive interface, causing adhesive failure, due to its smaller bonding area[36] which is also supported by Della Bona and Van Noort (1995). Their study found that tensile stress occurring near the adhesive interface initiated cracks or fractures at the base of the specimen when conducting the shear bond strength test and these caused misinterpretation as a cohesive failure. Moreover, finite element analysis (FEA) revealed an uneven distribution of force applied by the shear bond strength test.[37] The preparation of specimens for microtensile testing is very technique-sensitive requiring an experienced investigator, meaning they need to be cut into slabs with thicknesses ranging 0.5-1.5 mm in order to have small bonding area. Shape also affected the testing results which hourglass shapes provide more accurate μTBS values than those with non-trimmed bar-shaped but require a more complex

preparation method.[36]

Results showed that the most common modes of failure of both ceramics repaired by conventional and universal adhesive systems were adhesive failures (83.34 %). Thus, the mean values of μTBS for ceramics repaired by both adhesive systems were lower than the tensile strengths of the ceramics and resin composites. Specimens of ceramics repaired by the conventional adhesive system showed cohesive and mixed failure in greater numbers than those repaired by the universal adhesive system. The mean values of μTBS for the conventional adhesive system were higher than those of the universal adhesive system. Therefore, repairing ceramics using the former was considered to be more effective than using the latter.

Aging processes had no effect on mode of failure in the experimental groups, except for the EEU group. Before aging, the EEUI group showed dominant cohesive and mixed failure of the specimens. After aging, specimens in the EEUA group broke at the adhesive layer, indicating that the immediate bond strength of repaired EE ceramics using universal adhesive systems was effective; however, the thermocycling process reduced the bonding ability resulting in adhesive failure.

Pretest failures could be dealt with in many ways. When pretest failures were excluded from sum of bond strength value, the mean μTBS would be overvalued.[38] On the other hand, assigning them the value of zero would minimize the mean μTBS.[39-40] In this study, they were assigned as minimum μTBS of each group, so the mean μTBS stayed in normal level.[41]

Thermocycling at 10,000 cycles did not produce any difference in the repair performance between the two adhesive systems, therefore, alternative aging processes are suggested, such as increased numbers of thermocycling cycles and longer water storage. Other aging techniques including cyclic loading may provide useful data, and the ability of both adhesive systems to repair recently launched ceramics should also be trialed.

Conclusions

Within the study limitations, the following conclusions can be drawn. Higher μTBS between a resin composite and ceramic was achieved using a conventional adhesive system, compared to a universal adhesive system. This advantage was most distinct in the lithium-disilicate group. Aging process utilizing 10,000 cycles of thermocycling had no effect on the μTBS of repaired ceramics using resin composite and adhesive systems.

Clinical implications

After a period of aging, both universal and conventional adhesive systems demonstrated acceptable reparability. However, ceramic repaired with resin composite and conventional adhesive technique, using 9.5 % hydrofluoric acid as surface treatment, may provide favorable results, especially for lithium-disilicate ceramic.

Declaration of Conflicting Interest

The authors declare no conflict of interest.

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