A Comparison of Shear Bond Strength between Different Type of Resin Cements with Additional Universal Adhesives on Human Dentin

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Abstract

This is the study in vitro compared the shear bond strength between acrylic resin cement or self-adhesive resin cement with and without prior universal adhesive application to human dentin. Thirty-five extracted permanent third molars were collected and embedded in self-cured acrylic molds exposing the buccal surface of superficial dentin. Resin composite cylinders (Filtek Z350XT) with 2.38 mm diameter were prepared and cemented using the following combination of resin cement and universal adhesives; group 1: Rely X U200 (RU), group 2: Single Bond Universal Adhesive and Rely X U200 (SBU-RU), group 3: Panavia SA Luting Multi (PSA), group 4: Clearfil Tri-S Bond Universal Quick and Panavia SA Luting Multi (CBQ-PSA), and group 5 Super Bond C&B (SB). All the specimens were then stored in an incubator at 37ºC and subjected to 5,000 cycles of thermocycling. After that, the shear bond strength test was evaluated using the Universal testing machine (EZ-S, SHIMADZU). One-way ANOVA and Tukey's post hoc test were used to statistically analyze the shear bond strength values. SBU-RU had the highest shear bond strength, followed by SB which was higher than the remaining groups. The shear bond strength of RU was not different from CBQ-PSA but greater than PSA. However, the shear bond strength of CBQ-PSA was not different from PSA. Overall, applying universal adhesives before self-adhesive resin cement showed significantly higher shear bond strength only when applied with Rely X U200 ($p < 0.05$) and Rely X U200 demonstrated higher bond strength than Panavia SA Luting Multi ($p < 0.05$). In Conclusion, Super Bond C&B had the highest shear bond strength compared to non-universal adhesive-coating self-adhesive resin cement. However, with universal adhesive coating, Single Bond Universal Adhesive and Rely X U200 combined yielded the highest shear bond strength.

Keywords: *universal adhesive, self-adhesive resin cement, acrylic resin cement, shear bond strength*

1. Introduction

Bonding to dentin has always been an issue in dentistry. Unlike enamel, dentin is considered a vital organ, comprising 50 vol% of dentin mineral, 30 vol% of type I collagen, and 20 vol% of water. The structure of dentinal tubules is permeable to water which negatively affects the bonding process (Tjäderhane, 2015). On the other hand, resin cement is hydrophobic. Traditionally, resin cement has an etch-and-rinse protocol, consisting of the steps of dentin substrate etching, conditioning the dentin substrate from hydrophilic to hydrophobic with primer, and then infiltrating the resin monomer (Mushashe et al., 2016; Tjäderhane, 2015). However, with the complexity of procedures and technical sensitivity, novel resin cement systems have been developed to simplify their clinical use (Maaßen et al., 2020).

Self-adhesive resin cement is convenient and user-friendly since it requires no dentin pretreatment steps. It was applied to replace conventional cement such as zinc phosphate cement (Ferracane et al., 2011) in the first place. Self-adhesive resin cement combines the simple application of conventional cement and improved mechanical properties and bonding potential of conventional resin cement. It acts as self-etching at the initial stages of chemical reaction, encouraging dental substrate to be demineralized. Due to its simplicity, self-adhesive resin cement has become a widely used material globally. Even though it is user-friendly, its shortcoming is the unpromising bond strength and inconsistent durability, compared to conventional resin cement (Holderegger et al., 2008; Radovic et al., 2008). However, a manufacturer tried to address this issue

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by adding some components. A study revealed that the dissolved hydroxyapatite are able to chemically bond with functional monomers such as 10-MDP, 4-META and phenyl-P. These carboxylic or phosphate group-containing monomers are able to form an ionic bond with calcium in hydroxyapatite (Miyazaki et al., 2012). Toshi Hidari et al found a significant increase in bond durability in 10-MDP-containing dental adhesive compared to the same ingredient adhesive without 10-MDP and propose that 10-MDP might play a key role in enhancing dentin bond quality(Hidari et al., 2020). Apart from 10-MDP, other functional acidic monomers such as MDPB, 4-MET and phosphoric methacrylate esters are able to chemical interacts with hydroxyapatite, hence promoting adhesion to tooth structure (Giannini et al., 2015).

The modification of the dentin surface is also a method of enhancing bond stability since the interaction of self-adhesive luting agents to dentin exhibits a non-authentic hybrid layer (Vaz et al., 2012). This phenomenon occurs due to the high viscosity of the resin cement which brings down the ability to penetrate the dentin structure (Manso & Carvalho, 2017). Several methods have been proposed to establish better adhesion between the tooth and self-adhesive resin cement such as selective enamel etching, adhesive bonding coating or using mild etchant as a cavity cleanser (Son, 2015).

The universal adhesive is a novel dental adhesive that can be employed as etch and rinse mode, self-etch mode and selective etching (Perdigão & Loguercio, 2014). Apart from a dental substrate, it also can be applied to bond to various materials (Tsujimoto et al., 2017). The universal adhesives contain mild to moderate acidity functional monomers (mostly acidic phosphate monomers), conventional dimethacrylate cross-linkers, non-acidic emulsifying monomers, curing initiators and solvents (Papadogiannis et al., 2019). A study found that the use of Nd: YAG and femtosecond lasers combined with applying 10-MDP containing universal adhesive drastically improved the adhesion between dentin and self-adhesive resin cement, reported by Mahmut Sertaç Özdoğan et al (Özdoğan et al., 2021).

Super Bond C&B (SB) is a self-cure acrylic resin cement. It has 4-META to promote diffusion and TBB to initiate polymerization (Taira & Imai, 2014). SB has no inorganic fillers such as silica or Bis-GMA that differ from composite resin cement. Therefore, the resin structure is relatively low in hardness and flexural modulus when compared to composite resin cement which gives high plastic deformation properties. A green activator or 10% citric acid with ferric chloride is used as a dentin pretreatment agent which helps minimize the degradation of the collagen in the decalcification zone. TBB also plays a vital role in initiating the polymerization in rich oxygen and water conditions, which promotes the penetration of resin monomer forming a uniform hybrid layer (Shinagawa et al., 2019; Takagaki et al., 2009).

Even though there were studies regarding the technique of increased adhesion between dentin and self-adhesive resin cement, the use of universal adhesive as dentin coating is scarce. The comparison of different phosphate-based resin cement and acrylic resin cement is still lacking. This study was the comparison of the shear bond strength between two self-adhesive resin cement that contain different phosphate functional monomers and acrylic resin cement with the shear bond strength between universal adhesive-coated and uncoated dentin. The null hypotheses were that (1) there is no difference in shear bond strength between the three resin cement; (2) there is no difference in shear bond strength between dentin treated with universal adhesive and untreated dentin of the same resin cement.

2. Objectives

To evaluate the shear bond strength between acrylic resin cement and self-adhesive resin cement with and without prior universal adhesive application to human dentin.

3. Materials and methods

3.1 Sample preparation

Thirty-five extracted human maxillary and mandibular third molars were collected and stored in a 1% chloramine-T solution (TCI, Tokyo Chemical Industry, Japan). The selection criteria included teeth that were free from caries, restorations, and previous endodontic treatment. The teeth were used within 6 months after extraction. The root was cut with the fine diamond bur with coolant below the cementoenamel junction and the buccal surface was ground within the enamel and placed down and potted into a polyvinyl chloride

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mold (PVC) with 18 mm internal diameter, 22 mm external diameter, and 15 mm height, with the thin plastic film underneath. Then, the self-cure acrylic resin was poured into the mold. During the self-cure acrylic setting, the samples were under running water until the acrylic was completely settling. Then, the thin film remove (Figure 1).

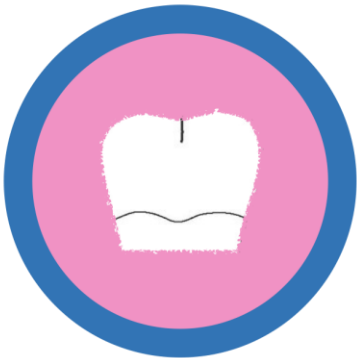

Figure 1 Root-cut third molar was potted and poured with self-cure acrylic resin in a PVC mold.

The dentin surface was prepared according to ISO 29022:2013 (International Organization for Standardization Technical Committee ISO TC 106 ISO 29022, 2013) by a two-step sequential planning process using P100 silicon carbide paper (TOA, Japan) at the speed of 250 rpm (Minitech 233, France) to expose the superficial dentin, followed by P400 silicon carbide paper (TOA, Japan) at the speed of 250 rpm until the surface was even and smooth when visually inspected. The planned surface was rinsed in the water and then cleaned with the ultrasonic cleaner in distilled water for 10 minutes. Then, the samples was stored in the water at room temperature and performed the cementation steps within 4 hours.

3.2 Cementation

The specimens were divided into five groups (n = 7);
Group 1 (RU) cemented with Rely X U200 (3M ESPE, USA),
Group 2 (SBU-RU) cemented with Single Bond Universal Adhesive (3M ESPE) and Rely X U200,
Group 3 (PSA) cemented with Panavia SA Luting Multi (Kuraray Noritake, Japan),
Group 4 (CBQ-PSA) cemented with Clearfil Tri-S Bond Universal Quick (Kuraray Noritake, Japan) and Panavia SA Luting Multi,
and group 5 (SB) cemented with Super Bond C&B (Sun Medical Co., Japan). Composite cylinders (Filtek Z350XT A1B, 3M ESPE, USA), 2.38mm diameter and 4mm height, received 27µm aluminum-oxide air abrasion at the cementation area, were prepared. The dentin has absorbed the water with gauze and then blown with triple syringe air for 5 seconds before the cementation steps. The middle one-third of the dentin was cemented to the composite cylinder under the loupes with 2.5X magnification.

Group 1 and 3, the mentioned luting agent was applied to the dentin with a micro brush. The resin composite cylinder was placed and pressed by the durometer (ASTM D 2240 Type A, USA) with a one-kilogram force (Figure 2). The excessive cement was removed with a micro brush, then was performed photoactivation with the light-curing unit (Demi Plus, Kerr, USA) with 1,000 mW/cm^3 for 40 seconds and another 20 seconds on the top of the composite cylinder after the loading device was unloaded.

Group 2 and 4, the procedures were similar to group 1 and 3 but required additional pretreatment steps. The mentioned universal adhesive was applied on the dentin with a micro brush in self-etch mode with rubbing motion for 20 seconds. Then the adhesive layer was blown gently until no movement of the liquid was observed. Assume the complete evaporation of the solvent. Then, following the protocols of group 1 and 3 depending on the systems used.

Group 5, the dentin surface was treated with the green activator in Super Bond C&B kits using a sponge pledget for 10 seconds and then rinsed with water and blown with air for 10 seconds. The catalyst and

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monomer were mixed according to the manufacturer's instructions. The small scoop of opaque type polymer powder was mixed with the monomer using the bulk-mix technique. Then, the cement was applied to the dentin and left for complete polymerization for 8 minutes under 1-kilogram loading.

All the specimens were stored in water at 37ºC for 24 hours in the incubator (Contherm 160M, Contherm Scientific Ltd., New Zealand), then subjected to the thermocycling process for 5,000 cycles at 5ºC and 55ºC with the dwell time of 30 seconds and the transfer time 5 seconds.

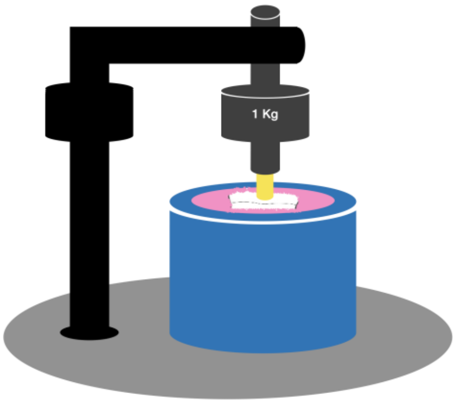

Figure 2 The composite cylinder was loaded with a 1-kg force

3.4 Shear bond strength test and fractography analysis

The samples were mounted on the Universal testing machine (EZ-S, SHIMADZU, Japan) and subjected to the shear bond test with a notched-edge crosshead. The notch of the crosshead was aligned directly over the composite rod and then flushed against the tooth surface (Figure 3). The crosshead was activated at the speed of 1mm/min until the debonding detect. The result recorded was megapascal (MPa), which calculate by force (N) divided by bonding area (mm^2).

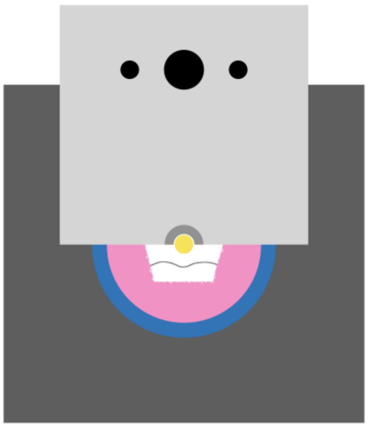

Figure 3 The notched-edge crosshead was aligned over the composite cylinder

The debonded dentin was then evaluated in the failure mode under the stereomicroscope (SZ 61, OLYMPUS, Japan) at 40X magnification. Adhesive failure marked if the debonded dentin showed no remnant of resin cement. Cohesive failure in this study had only in the resin cement layer that noted if the remnant of resin cement covered all the debonded area. When the mixture of dentin and resin cement in the debonded area was made visible by the eye, the specimen was marked as mixed failure.

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3.5 Statistical analysis

The data were analyzed using statistical software (IBM® SPSS 20, SPSS, Chicago, IL, USA). The Shapiro-Wilk test was carried out to test whether the data were normally distributed. One-way ANOVA and Tukey's HSD post hoc analysis were used to compare the mean shear bond strength values statistically. A P-value less than 0.05 was considered significant in all comparisons. The failure mode was analyzed using descriptive statistics.

4. Results and discussion

4.1. Results

The mean shear bond strength values (MPa) and the standard deviations were depicted in the graph (Figure 4). The highest value was obtained from the SBU-RU group (22.583 ± 3.419 MPa), which is significantly higher than the RU group (12.135 ± 2.527 MPa) (p-value <0.05). On the other hand, CBQ-PSA (9.233 ± 1.684 MPa) demonstrated higher shear bond strength than PSA (7.909 ± 1.721 MPa) but was not statistically different (p-value >0.05). PSA also exhibited significantly lower bond strength than RU (p-value<0.05). The number of SB (17.920 ± 2.011 MPa) was statistically lower than SBU-RU (p-value <0.05) but higher than the others (p-value >0.05).

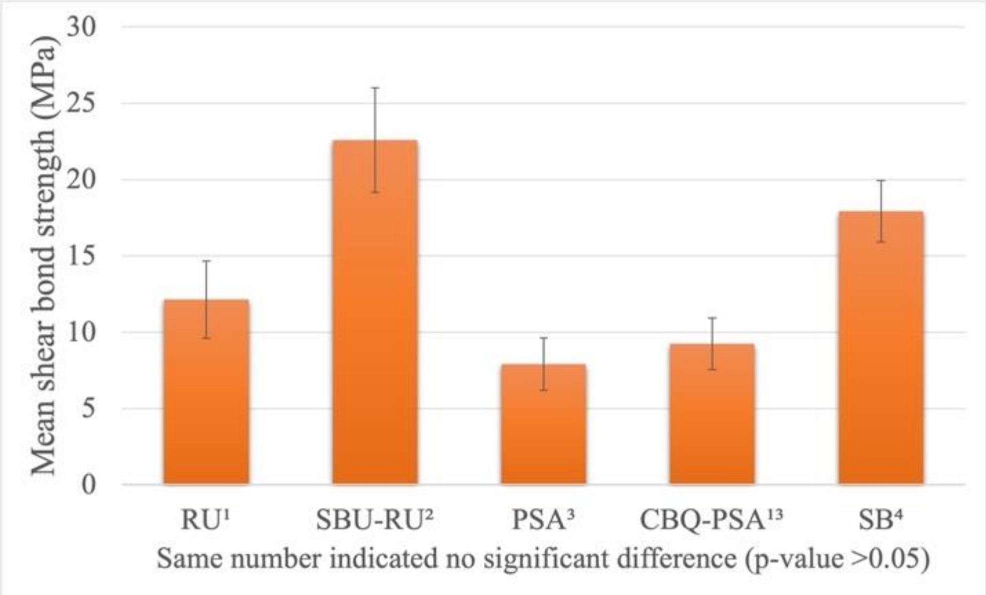

Figure 4 Mean shear bond strength of 5 different cementation groups

The failure mode of the luting agent was visualized by the stereomicroscope and shown in the graph (Figure 5). RU and PSA predominantly showed adhesive and mixed failure whereas CBQ-PSA and SB mostly showed cohesive failure in resin cement and mixed failure. SBU-RU found only the cohesive failure in resin cement.

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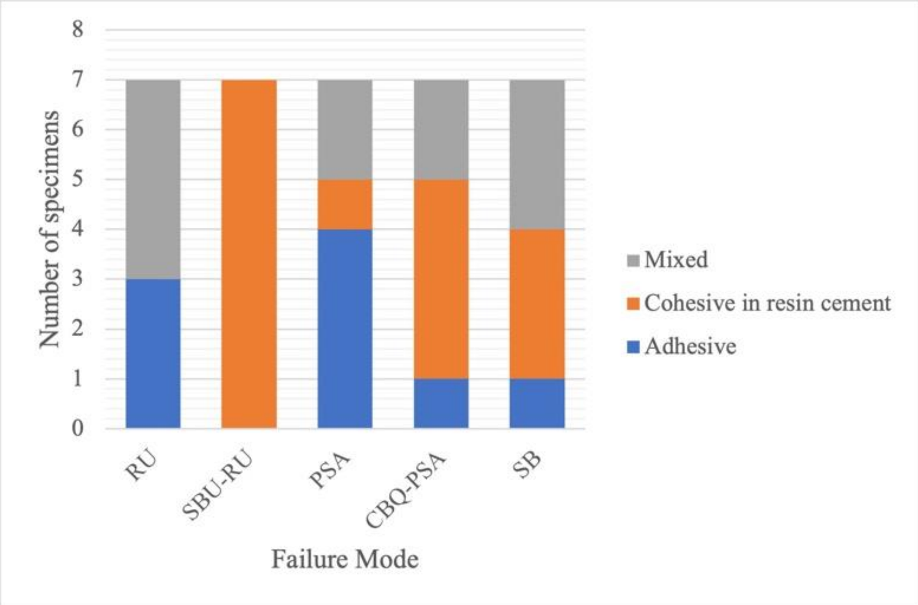

Figure 5 The failure mode of the 5 groups

4.2. Discussion

This study was conducted to investigate the shear bond strength between an acrylic resin cement (4-META resin cement) and two different phosphate-based self-adhesive resin cement with human dentin, and the effect of applying universal adhesives on human dentin before the application of self-adhesive resin cement. The first null hypothesis was accepted as three different cements exhibited different shear bond strengths. However, the second null hypothesis was rejected because Clearfil Tri-S Bond Universal Quick coating failed to exhibit significantly different shear bond strength compared to using Panavia SA Luting Multi alone. However, there was a significant improvement in shear bond strength value when applying Single Bond Universal Adhesive before the load of Rely X U200. Therefore, it seems that the different phosphate monomers in the resin cement may provide different tooth adhesion capabilities and the surged bond strength observed in the universal adhesive coating before loading the luting agent could be product dependent.

The resin cement used in this study was Rely X U200, Panavia SA Luting Multi and Super Bond C&B. The reason for choosing these three resin cements is that they are widely used in dentistry and a comparison of acrylic resin cement and composite resin cement is also rare. The result of this study showed that Super Bond C&B demonstrated the highest bond strength, followed by self-adhesive resin cement which is in line with previous studies (Doğanay Yıldız et al., 2019; Özcan & Mese, 2012). This could be attributed to the property of ferric chloride in the green activator that can prevent the degradation of the collagen fiber at the decalcification layer. The bond strength between MMA resin and ferric ion-absorbed dentin was significantly improved as reported in this study (Takagaki et al., 2009). However, self-adhesive resin cement cannot encapsulate the collagen mesh and form an authentic hybrid layer (Vaz et al., 2012). Thus, these resin cement adhere to the dentin weaker than Super Bond C&B does. Rely X U200 performs better bond strength compared to Panavia SA Luting Multi is accordant with the other study that compared Rely X U200 with Panavia SA (Silva et al., 2019). It could suggest that HEMA monomers incorporated into the Panavia SA Luting Multi which is hydrophilic could lead to poor bonding (Ikemura et al., 2011).

This study showed that Single Bond Universal Adhesive coating almost doubled the shear bond strength value. Even though a phosphoric methacrylate ester which is a functional monomer of this luting cement can chemically react to the tooth substrate (Giannini et al., 2015), the high viscosity of the material might limit the encapsulation of the resin on the dentin surface which results in poor hybrid layer formation

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(Manso & Carvalho, 2017). On the other hand, a Single Bond Universal Adhesive coating can promote a dense uniform hybrid layer with long resin tags regardless of adhesive strategy. Besides, 10-MDP in this adhesive can ironically bond to the Ca^{2+} in hydroxyapatite and form a nano-layer calcium salt on hydroxyapatite (Hass et al., 2019). The strong hybrid layer formation and the stable nano-layer calcium salt may contribute to noticeably higher bond strength in this circumstance. However, when applying Single Bond Universal Adhesive before Rely X U200, it is interesting that the bond strength exceeds the number of Super Bond C&B. Even though 4-META resin cement provides a strong hybrid layer. The relatively low flexural strength might lead to early cohesive breakdown in the resin cement layer as it is a weak point when the cement expose to the environment (Shinagawa et al., 2019).

The result of Panavia SA Luting Multi and its universal adhesive failed to demonstrate a significantly higher bond strength compared to without adhesive-coating conditions. This is interesting because the low viscosity of the universal adhesive should interact with dentin more efficiently when compared to the high-viscosity resin cement. It could be that the lower pH of Clearfil Universal Bond Quick (pH = 2.3) compared to Single Bond Universal (pH = 2.7) negatively affects the chemical polymerization of the resin cement which results in a low degree of conversion. André Mallmann and colleagues stated that the low pH of the adhesive system negatively affects the bond strength of dentin when using chemically activated resin composite but not in light-activated conditions (Mallmann et al., 2007). Although this study is designed to perform photoactivation, the composite cylinder height can be an issue. The 4 mm thickness was chosen to simulate indirect restoration cementation since the distance from the central groove to the roof of the pulp chamber in the mandibular first molar is around 4 mm (Reuben et al., 2008). This could be the limitation of the light being able to pass through the 4 mm thickness and reach the resin cement at the bottom. A study found that resin composite has the capacity of reducing light penetration and consequently, dramatically affects the polymerization effectiveness at the bottom of the specimen (Aguiar et al., 2009). Tilt-angled light activation is also considered a compromised method of photocuring (Konerding et al., 2016). Therefore, the polymerization of the resin cement in this study also relies on chemical activation which is impaired by the high acidity of the resin adhesive. This could be the reason why the negligibly higher bond strength is observed in this study.

The present study showed that Rely X U200 had significantly higher bond strength than Panavia SA Luting Multi. On the other hand, when coating with Single Bond Universal Adhesive containing 10-MDP, Rely X U200 demonstrated almost doubled bond strength. Therefore, it cannot conclude that the phosphoric methacrylate ester in Rely X U200 played a key role and had better adhesion to dentin over 10-MDP in Panavia SA Luting Multi. The acidic monomer is only one of many factors that strengthen the dentin bond; however, the heart of strong tooth adhesion is to achieve the uniform durable hybrid layer which is contributed to both mechanical and chemical properties of the resin cement (Nakabayashi et al., 1991).

The failure modes were associated with the bond strength in this study. The high shear bond groups predominantly showed cohesive failure in resin cement and mixed failure, whereas the low shear bond groups predominantly showed adhesive and mixed failure, except for group CBQ-PSA where cohesive failure in resin cement was highly observed. It could be speculated that the low degree of conversion of the resin cement in this group caused the weakness of the cement layer which in turn, is prone to fracture at the cement layer (Pereira et al., 2010). Group SBU-RU displaying only cohesive failure in resin cement with high bond strength could be the success of achieving a strong dentin-adhesive bond interface. Therefore, even though CBQ-PSA failed to exhibit high bond strength, applying universal adhesives in both self-adhesive resin cement could strengthen the dentin bond since adhesive failure dramatically decreased as compared to without-adhesive-coating groups.

According to the results of this study, it is suggested that applying universal adhesive on dentin before loading self-adhesive resin cement does not have a negative effect on the shear bond strength. Besides, one product produces high bond strength after coating with a universal adhesive agent. Although Super Bond C&B benefits the dentin adhesion due to ferric chloride in the green activator, the low flexural strength property could lead to a cohesive breakdown in the cement layer.

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This study had some limitations. Being an in-vitro study might not simulate the actual clinical service. Due to time limitations, it was designed to perform a thermocycling of 5,000 cycles that claimed to be equivalent to 6 months of the intraoral situation (Gale & Darvell, 1999). The use of a 4 mm thickness of composite cylinder could be an issue for light penetration. Also, the present study only compared two brands of self-adhesive resin cement. More studies should be carried out to test the shear bond strength of various resin cement with the longer ageing condition.

5. Conclusions

Based on the findings of this present study under limitations, it can conclude that:

1. Two different phosphate-based self-adhesive resin cement produced a statistical difference in shear bond strength.

2. Applying universal adhesive prior to self-adhesive resin cement showed no negative effect on shear bond strength.

3. Without universal adhesive coating, Super Bond C&B had the highest shear bond strength compared to self-adhesive resin cement. However, with universal adhesive coating, Single Bond Universal Adhesive and Rely X U200 combined yielded the highest shear bond strength.

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