

Efficacy of Restorative Primers on the Shear Bond Strength of Polymer-infiltrated Ceramic Network to Resin Cement

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Abstract

This study investigated the shear bond strength (SBS) of polymer-infiltrated ceramic network (PICN) to resin cement by application of different restorative primers which are commonly available in dental clinic. Fifty square-shaped PICN specimens were prepared and treated with 5% hydrofluoric acid. Then, the specimens were randomly divided into five groups with different surface treatments as follows: non-chemical surface treatment as control group (C), surface treatment with Monobond N (MN), Alloy Primer (AP), Super-Bond Universal Ceramic Primer (SB), and Clearfil™ Ceramic Primer Plus (CF). The specimens were then bonded to cylindrical resin composite block with Panavia™ V5. The SBS test was performed with universal testing machine. Data were recorded and statistically analyzed by One-way ANOVA and Tamhane's T2. The mode of failure was also evaluated under a stereomicroscope at 35x. The results showed that the mean SBS values of MN, CF, and SB groups were significantly higher than that of the C group ($p < 0.05$), but not significantly different to each other ($p > 0.05$). In contrast, AP group provided the lowest SBS value among all the groups ($p < 0.05$). The modes of failure of MN, CF, and SB groups were much preferable than that of C and AP groups. Thus, selection of restorative primers prior to cementation should be thoroughly considered due to the positive and negative effect on the SBS value of the PICN restorative material.

Keywords: Polymer-infiltrated ceramic network, Restorative primer, Shear bond strength, Surface treatment.

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Abstract

The polymer-infiltrated ceramic network (PICN) is a CAD/CAM resin-ceramic that has been developed to overcome the disadvantages of conventional dental ceramics. PICN is a structure with a sintered ceramic matrix infiltrated with a polymer matrix that provides the combination of

more flexibility, less stiffness, increased softness with satisfactory flexural and fracture strength values, and decreased wear of the occluded tooth.^{1,2} In the modern days, PICN has been used for the same purpose as other all-ceramic restorations due to its physical properties,

tooth-like appearance, and its ability to bond with various resin cements. Many studies have also shown that the PICN can be repaired with resin composite which is convenient for the patient and dentist in routine dental practice.^{3,4}

For PICN in this study, we used Vita Enamic[®] which is composed of ceramic 86% by weight (silica-based ceramic), and polymer 14% by weight (mostly UDMA). Thus, the bonding of PICN restorations utilizes the similar procedure as other silica-based dental ceramic by surface treatment and resin cement for the cementation. Surface treatment provides the mechanical and chemical retention, which significantly increases the bond ability of the restoration.³⁻⁶ Surface treatment with hydrofluoric acid (HF) creates mechanical retention by increasing the surface roughness of restoration, which in turn increases the surface area. Subsequently, the application of silane coupling agent promotes the chemical retention of the restoration to resin cement.³⁻⁸

In the present dental market, there are various commercially restorative primers available to select from, for instance, metal primer, ceramic primer, and universal restorative primer. Each primer is different in composition, ratio, or even method of application. The 10-MDP (10-Methacryloyloxydecyl dihydrogen phosphate) is commonly recommended for use with base metal alloy, alumina, and zirconia restorations. The sulfuric derivative primers, such as VBATDT (6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione), are recommended for use with noble metal alloy restoration. The silane coupling agent, 3-MPS (3-methacryloxypropyl-trimethoxysilane or γ -MPS) is recommended for use with silica-based restoration. The different types of restorative primers may be available in separated bottles or a mixture in one bottle. The component of ceramic primers could be only silane coupling agent (i.e., RelyX[™] Ceramic Primer and Porcelain liner M), or silane coupling agent with 10-MDP (i.e. Clearfil[™] Ceramic Primer Plus and Super-Bond Universal Ceramic Primer). Furthermore, the universal restorative primers which are composed of silane coupling agent, 10-MDP, and sulfuric derivative are also available. The

advantages of universal restorative primer are the ease of use and the lower cost than buying separate bottles.

Silane coupling agent increases the wettability of the restorative surface, and Si-OH group bonds with silicon oxide on silica-based material and its C=C chain has chemical reaction with the organic matrix of resin^{9,10}, while 10-MDP increases bond ability on tooth structures, resin composites, and zirconia based materials.¹¹⁻¹³

As the popularity of PICN is increasing, and thus inevitably becoming widely used in the routine dental practice, it's interesting to explore how different restorative primers in the daily dental practice affect the bond ability of PICN restoration. The purpose of this study was to investigate and to compare the effect of the restorative primers on the shear bond strength between PICN and resin cement. The null hypothesis was the shear bond strength between PICN and resin cement would not be affected by the different restorative primers.

Materials and Methods

The 50 square-shaped PICN specimens (VITA Enamic[®], VITA Zahnfabrik, Bad Säckingen, Germany) sized 6x7x3 mm³ were prepared by low speed cutting machine (IsoMet[™], Buehler, Illinois, USA). The specimens were invested into the PVC mold with acrylic resin (Unifast[™] Trad, GC cooperation, Tokyo, Japan) at the same level of acrylic resin. Then, the specimens were polished with metallographic paper P400, P600, P800 and P1000 respectively, using polishing machine (Nano 2000 grinder-polisher, Pace Technologies, Tucson, USA) under pressure of 4 kg/cm³, 200 rpm for 15 minutes. All specimens were subsequently cleaned with deionized water in ultrasonic bath for 2 minutes and dried with oil-free air. The 80- μ m thickness tape (ScotchBlue[™] Painter's Tape, 3M ESPE, Minnesota, USA), sized 10x10 mm² with the hole of 3 mm in diameter were placed on to the specimens prior to surface treatment of each specimen. The surface of the prepared specimens was treated with 5% hydrofluoric acid (VITA Ceramics Etch, VITA Zahnfabrik, Bad Säckingen, Germany) for 60 seconds and cleaned with deionized water for 60 seconds and dried with oil-free air

for 10 seconds. The PICN specimens were randomly divided into five groups (n=10), one control group and four different restorative primers, as follows:

Group 1: The surface was left untreated with any restorative primer as a control group (C).

Group 2: The surface was treated with Monobond N (MN).

Group 3: The surface was treated with Super-Bond Universal Ceramic Primer (SB).

Group 4: The surface was treated with Clearfil™ Ceramic Primer Plus (CF).

Group 5: The surface was treated with Alloy Primer (AP).

The restorative primers were applied on the prepared surface according to the manufacturer's instruction as shown in table 1. Then, the 3-mm in diameter and height of cylindrical composite resin specimens (Clearfil™ DC core, Kuraray Noritake Dental Co., Ltd., Tokyo, Japan) were fabricated and cemented to PICN specimens with Panavia™ V5 (Kuraray Noritake Dental Co., Ltd., Tokyo, Japan). The bonded specimens were placed under pressure of 1 kg using Durometer (PTC Instruments, California, USA). The excessive cement was removed, and light cured by light curing unit (Elipar™ FreeLight 2, 3M ESPE, Minnesota, USA) for 20 seconds at four sides and kept under pressure for

10 minutes. The bonded specimens were incubated in 37°C water for 24±2 hours in the dark environment. The SBS test was performed by universal testing machine (Shimadzu EZ-S, Shimadzu, Kyoto, Japan) with load cell 500 N and crosshead speed of 0.5 mm/min where the blade of testing machine was 1 mm in vertical dimension away from the bonded interface. The SBS values were collected and statistically analyzed by IBM SPSS Statistics 22 (IBM, New York, USA). The surfaces of fractured specimens, both on PICN and resin composite side, were investigated under the stereomicroscope at 35x magnification level which could be classified into 3 categories as follows:¹⁴

1) Adhesive failure: the failure occurred at the interface between composite resin and resin cement (Adh CR) or between resin cement and VITA Enamic® (Adh RE) more than 75 % of all areas

2) Cohesive failure: the failure occurred in the substrates of resin composite (Co C), resin cement (Co R) or VITA Enamic® (Co E) more than 75 % of all areas

3) Mixed failure: the failure which combined adhesive and cohesive failures, occurring at the interface and in the substrates where more than 25 % of both Adh RE and Co E were observed.

Table 1 The manufacturer's instruction for each primer

Primer	Instruction
MN: Monobond N (Ivoclar Vivadent AG, Liechtenstein)	Apply a thin coat of Monobond N with a microbrush to the pre-treated surfaces. Allow the material to react for 60 seconds. Subsequently, disperse any remaining excess with a strong stream of air.
AP: Alloy Primer (KURARAY CO., LTD.m)	Apply the Alloy Primer to specimens with brush and leave it dry.
SB: Super-Bond Universal Ceramic Primer (Sun medical, Japan)	1. Dispense an equal number of drops of UNIVERSAL PRIMER Parts A & B (1:1 ratio) into a clean mixing well and mix with a brush. 2. Apply the mixture to the surface to be bonded. 3. Blow lightly if the liquid remained.
CF: Clearfil Ceramic Primer Plus	1. Dispense the necessary amount of Clearfil Ceramic Primer Plus into a well of the mixing dish immediately before application. 2. Apply Clearfil Ceramic Primer Plus to the adherent surface of the restoration with an applicator brush. 3. Dry the entire adherent surface sufficiently using mild, oil-free air flow.

Results

The data of each group were analyzed by Kolmogorov Smirnov test and showed normal distribution in all groups. However, the Levene's test showed unequally homogeneity of variances. Thus, One-way ANOVA and post hoc Tamhane's T2 tests were chosen for the statistical analysis at 95% confidence level.

The mean SBS value and standard deviation of each group are shown in Table 2.

Table 2 The mean SBS value (MPa) and standard deviation of each group (mean \pm SD)

Group	Mean SBS \pm SD
C	13.10 \pm 1.40 ^B
MN	16.33 \pm 1.67 ^A
SB	14.81 \pm 0.52 ^A
CF	15.07 \pm 0.78 ^A
AP	11.42 \pm 0.66 ^C

* The same superscript capital letter means there was no significant difference at 95% confidence level

The results showed that the mean SBS value of MN, CF and SB groups were significantly higher than that of the C and AP groups ($p < 0.05$). Among these three groups, the MN group showed the highest SBS values followed by the CF and SB groups, but not significantly different ($p > 0.05$). Only the AP group showed significantly lower mean SBS value than that of the others ($p < 0.05$).



Figure 1 The mode of failures of each group

The mode of failure (Figure 1) showed that the C and AP groups exhibited only adhesive failure between the resin cement and VITA Enamic® (Adh RE) in all specimens. The others showed Adh RE combined with mixed failure (Adh RE with Co E). The MN group showed the most mixed failure followed by the CF and SB groups.

Discussion

The bond strength of the PICN restorations could be improved by mechanical and chemical surface treatments. The mechanical method, roughening the surface, was performed by grinding, etching or sandblasting on the inner surface of restoration, while using various types of primer according to the chemical structure of restorative materials could provide proper chemical bond of the ceramic restoration to resin cement. According to the manufacturer's specification, the dominant composition of VITA Enamic® is alumina-enriched feldspar ceramic network (86% by weight) which is composed of SiO₂ 58-63% by weight and Al₂O₃ 20-23% by weight. The ceramic network is strengthened by an acrylate polymer network, with both networks fully integrated with one another. The polymer is a mixture of two dimethacrylates: UDMA and TEGDMA (14% by weight). The manufacturer's recommendation for surface treatment of VITA Enamic® is the application of 5% hydrofluoric acid for 60 seconds, rinse for 60 seconds and dry with oil-free air as mechanical surface treatment. Then, applying the silane coupling agent containing restorative primer for chemical surface treatment similar to the surface treatment for the silica-based ceramic restorative material.

Previous studies showed that surface treatment of PICN restoration significantly increased the bond strength of PICN and resin composite in various resin cements.³⁻⁸ In 2016, Campos found that hydrofluoric acid etching achieved the highest bond strength compared to air-abrasion and no treatment groups.⁶ Bello *et al.*, reported that mechanical retention from sandblasting technique provided more surface roughness than 5% hydrofluoric acid etching, but there was not significantly different in term of bond strength when applying the silane-coupling agent.³ Conejo *et al.* in 2020, also reported that 5% hydrofluoric acid etching for 60 seconds or 120 seconds followed by silanization provided the highest bond strengths.⁸

In this study, the mechanical surface treatment was performed by application of 5% hydrofluoric acid according to manufacturer's recommendation. After that, 4 different restorative primers, commonly available in dental clinic, were chosen according to the different types

of compositions and the number of primer bottles. Alloy Primer is composed of VBATDT and 10-MDP. Monobond N is composed of silane coupling agent, 10-MDP and 10-MDDT (10-methacryloyloxydecyl 6,8-dithiooctanoate). Clearfil™ Ceramic Primer Plus is composed of 10-MDP

and silane coupling agent. Super-Bond Universal Ceramic Primer is also composed of 10-MDP and silane coupling agent, but in separate bottles. The composition of restorative primers are shown in Table 3.

Table 3 Compositions of restorative primers in this study

Trade name	Manufacturer	Compositions	Lot No.
Monobond N	Ivoclar Vivadent, Liechtenstein	Ethanol, water, 3-MPS, 10-MDP and 10-MDDT	X17917
Super-Bond Universal Ceramic Primer	Sun medical, Japan	Liquid A: MMA and 10-MDP Liquid B: MMA and 3-MPS	RR1
Clearfil™ Ceramic Primer Plus	Kuraray Noritake dental, Japan	Ethanol, 3-MPS and 10-MDP	8T0038
Alloy Primer	Kuraray Noritake dental, Japan	Acetone, 10-MDP and VBATDT	870094

* 3-MPS = 3-Trimethoxysilylpropyl methacrylate, 10-MDP = 10-Methacryloyloxydecyl dihydrogen phosphate, 10-MDDT = 10-methacryloyloxydecyl 6,8-dithiooctanoate and VBATDT = 6-(4-Vinylbenzyl-n-propyl)amino-1,3,5-triazine-s,4dithiol

The resin cement used in this study was Panavia™ V5 which is a novel resin cement. Previous studies showed higher bond strength of Panavia™ V5 compared with Panavia™ F2.0.^{15,16} It also showed higher color stability than Panavia™ SA due to the amine-free composition.¹⁷ The Panavia™ V5 is MDP-free in cement paste, but 10-MDP is still available in adhesive. Thus, Panavia™ V5 was chosen in this study to eliminate the effect of 10-MDP in resin cement, which may confound the effect of 10-MDP in the restorative primer.

The results of our study showed that MN, CF and SB groups which are silane containing restorative primer could significantly improve the SBS value compared with the C group. This is in agreement with previous recent studies.^{5,6,8,18,19} Hence, the null hypotheses of this study was rejected. The use of silane coupling agent is recommended for promoting the surface wettability of the restoration and the chemical bond between resin cement and silica-based materials.²⁰ The siloxane group (inorgano-functional group) of the silane coupling agent could react with the silica substrate on the surface, while the organo-functional group such as methacrylate could co-polymerize with the resin cement.²¹⁻²³ In 2016, Schwenter et al investigate the etching of VITA Enamic® with 5% HF followed by application of silane coupling agent, increased the shear bond strength between VITA Enamic® and resin composite cement.¹⁸

Tanapon *et al* subsequently reported the shear bond strength between lithium disilicate glass ceramic and resin cement. They found that the application of any type of silane coupling agent significantly increased the bond strength when compared with the untreated group.²⁴ These confirm the important role of silane coupling in promoting the chemical bond; covalent Si-O-Si bonds, of the silica component in VITA Enamic® and even in lithium disilicate ceramic.

Although MN, CF, and SB groups all contain silane coupling agent, they are different in their compositions and the number of primer bottles. This might affect the bond strength. Monobond N primer and Clearfil™ Ceramic Primer Plus are single bottle restorative primer. In contrast, Super-Bond Universal Ceramic Primer is the two-bottle system. Due to the manufacturer's data which are shown in Table 3, the mixing ratio of liquid A and liquid B of Super-Bond Universal Ceramic Primer might affect the shear bond strength. The among of the silane coupling agent may be different in concentration when mixing with 10-MDP. Furthermore, the homogeneity of the mixture may cause the distribution of silane coupling agent which leads to lower SBS value of the SB group compared with the MN and CF groups, but not significantly different. Thus, further studies may observe the effect of different ratio of liquid A and liquid B on the bonding ability between silica-based

restoration to resin cement. Including, one-bottle primer compared with mixing of two-bottle system of Super-Bond Universal Ceramic Primer.

According to Cui *et al*, the degree of conversion of polymer matrix in PICN was 82.17%,²⁵ hence the chemical bond between resin cement and polymer matrix in PICN seems to be a minor part to create the chemical bond. From previous studies, 10-MDP could improve surface wettability and enhance mechanical bonding in zirconia restoration^{11,13,26}, but the number of studies in silica-based and polymer-based restoration is still limited. The increase of surface wettability may improve the bond strength of restoration via mechanical retention.²⁶ However, the result showed the lowest mean SBS values in the AP group, which contains 10-MDP and VBATDT, hence 10-MDP might not enhance the bonding to PICN.

The VBATDT in Alloy primer and the 10-MDDT in Monobond N are the sulfur containing component, but the results in this study showed the significantly contrast SBS values. One possible reason might be the chemical structure differences, specifically sulfur containing component. VBATDT monomer consists of thiol group, while 10-MDDT monomer consists of disulfide group. The VBATDT is a restorative primer that contains thiol group and vinyl group on the end chain of each side. The coupling mechanism occurs by transforming thione into thiol and formation of the bond on the precious metal surface, and copolymerization of the vinyl groups with the resin cement. The thiol group-containing monomers are known to inhibit polymerization as they act as a substrate causing chain reaction movement in radical polymerization.²⁷ In our study, the AP group showed the significantly lowest mean SBS value and even significantly lower than that of the control group. This might be the negative effect of the unreacted thiol groups on bond strength. Since there is no metal oxide layer on the bonding surfaces, hence, the coupling mechanism cannot occur. Therefore the excess unreacted thiol group in VBATDT might cause the inhibition of the propagation of vinyl or acrylic free radicals, leading to inhibition of the resin cement polymerization and bond

strength may deteriorate at the primer-applied surface.²⁷ For this reason, the Monobond N that contains 10-MDDT (disulfide groups) had no negative effect on the SBS value in this study.

The mode of failure investigation in this study showed all adhesive failure between resin cement and PICN (Adh RE) in the C and AP groups. This could imply that the bond is the weakest point of the bonded interface,¹⁸ while the MN, CF, and SB groups showed Adh RE and mixed failure (Adh RE with Co E). This result is in agreement with the SBS value which revealed that the C, and AP groups showed significantly lower SBS values than those of the MN, CF, and SB groups. Thus, it might be concluded that the bond strength between resin cement and PICN is the weakest point and could be improved by applying the silane containing restorative primer which is in accordance with the mean SBS value observed in the study.

Conclusion

From the result of this study, it could be concluded as follows:

1. Restorative primers that contain Silane coupling agent increase the SBS between VITA Enamic[®] and resin composite via PanaviaTM V5 by the chemical bond with silica-component in VITA Enamic[®].
2. Restorative primers that contain VBATDT decrease the SBS between VITA Enamic[®] and resin composite via PanaviaTM V5 due to the unreacted VBATDT which may inhibit the polymerization of resin cement.
3. 10-MDP in restorative primers does not improve the SBS between VITA Enamic[®] and PanaviaTM V5.

Although many types of restorative primers are available in dental clinic, selection of the proper restorative primer for use with the PICN restoration should be thoroughly considered. The restorative primer may positively or negatively affect the bond strength. Thus, dentist should properly select the restorative primer to enhance the bond strength of the restoration and to avoid the negative effect which decreases the SBS of the restoration to resin cement.

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Conflict of interest

There is no conflict of interest in this study.

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