The effects of dentin and intaglio indirect ceramic optimized polymer restoration surface treatment on the shear bond strength of resin cement

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Abstract. Ceramic optimized polymer (ceromer) bonds to the tooth substrate through resin cements. The bond strength between dentin, resin cement, and ceromer depends on the applied surface treatment. To analyze the effects of dentin and intaglio ceromer surface treatment on the shear bond strength self-adhesive resin cement. Forty-five dentin premolar and ceromer specimens were bonded with resin cement and divided into three groups as follows: in group 1, no treatment was applied; in group 2, dentin surface treatment was carried out with acid etching and a bonding agent; and in group 3, dentin surface treatment was carried out with acid etching, a bonding agent, and intaglio ceromer surface treatment with etching and silane. All specimens were incubated at 37 °C for 24 hours, and the shear bond strength was measured using a universal testing machine. Group 3 showed the highest shear bond strength, followed by group 2. The surface treatment of dentin and intaglio ceromer showed significantly improved shear bond strength in the group comparison. Dentin and intaglio ceromer surface treatment can improve the shear bond strength self-adhesive resin cement.

1. Introduction

The use of indirect tooth-colored restorations with composite and ceramic bases has been increasing. However, indirect composites have a weakness, as plaque could easily accumulate because of the rough material surface and the lack of full polymerization of the monomer [1]. Moreover, the usage of ceramics as a restoration material requires a large amount of tooth structure removal for its minimum width of 1.5–2 mm, and such material can cause tooth wear [2]. The weaknesses of composites and ceramics have triggered the emergence of a new material that combined ceramic and composite characteristics to preserve tooth structure; the restoration material is known as ceramic optimized polymer (ceromer), polymer-infiltrated ceramic network (PICN), organically modified ceramic (ormocer), or resin-matrix ceramic. Ceromer/representing a modification of the organic polymer matrix and ceramic filler, has been developed for flexural strength, modulus of elasticity, and compressive strength so that it more closely approximates the tooth structure. Thus, this material could fulfill the esthetic needs related to restorations with better plaque resistance and optimal physical properties [3, 4].

Indirect tooth-colored restorations require a strong bond with the tooth structure so that the restoration will not be easily detached. Such a bond can be achieved with the use of resin cement as the adhesive. With the technological development of adhesive materials, a resin cement product with a...
one-phase application known as self-adhesive resin cement has been generated. Self-adhesive resin cement can bind to the tooth structure without the application of etching, primer, and bonding agent; therefore, the application of this cement is easier and requires fewer clinical procedural stages. It can also bind to the tooth structure chemically and has many shade variations to produce good aesthetic results [5, 6].

Organic matrixes on self-adhesive resin cement contain modified phosphoric acid methacrylates. Phosphoric acid methacrylates react with main filler of the cement and hydroxyapatite from the tooth’s hard tissue. However, a low acidic level in the resin cement will weaken the bond strength, and this strength is fundamental for the long-term stability of a restoration. Clinical studies have shown that the lack of retention between indirect tooth-colored restorations and the tooth surface is the main reason for restorations to come off.

Retention can be improved to prevent restoration detachment by carrying out surface treatment, which means carrying out prior treatment on the dentin and inner part of the restoration facing tooth surface (intaglio). Some surface treatments that can be done for intaglio of an indirect restoration are sandblasting, hydrofluoric acid application, and using silane as a coupling agent. Sandblasting is a method that involves an additional tool; it is relatively impractical and expensive for daily clinical use. Thus, surface treatments using hydrofluoric acid application and silane are most common [7-9].

Various cementation methods for indirect tooth-colored restorations, whether resin- or ceramic-based, have been studied. D’Arcangelo studied the effect of the indirect composite restoration intaglio surface treatment method and found that the control group, which was not given any surface treatment, exhibited more frequent adhesive failure compared to the group with surface treatment using hydrofluoric acid application and silane [9]. Moreover, Pavanelli et al. evaluated the shear bond strength of self-etching, the etch-and-rinse approach, and self-adhesive for enamel and dentin using indirect ceramic composite material; they showed that the highest shear bond strength was found in the etch-and-rinse enamel–ceramic group [10]. Alves et al. revealed that cementation with self-adhesive resin cement combined with the intaglio ceramic surface treatment method using hydrofluoric acid application and silane gave higher shear bond strength compared to resin cement without any surface treatment [11]. In addition, Jetti et al. evaluated the shear bond strength of feldspathic Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) ceramic with 5% hydrofluoric acid combined with silane. The results indicated that the addition of silane application to intaglio ceramic after the etching process using hydrofluoric acid increased the strength of adhesion to the tooth surface [12]. Fernandes et al. tested the shear bond strength of ceromer intaglio restoration with dentin using three different kinds of cements. The results showed that total etch and self-etch cement provided higher shear bond strength scores compared to self-adhesive cement [13].

The bond strength between a restoration and the tooth structure can be analyzed via microtensile bond strength and shear bond strength tests. The measurement of shear bond strength is the most common measurement performed in the laboratory to evaluate the bonding system. Shear bond strength is the maximum shear stress that can be accepted or resisted by a material before it is sheared [14, 15]. This study was performed to test the effect of dentin and intaglio indirect ceromer restoration surface treatment to the shear bond strength of self-adhesive resin cement.

2. Materials and Methods

The specimens were dentin of premolar teeth and Ceromer (Ceramage™; Shofu Inc., Kyoto, Japan). Inclusion criteria for tooth specimens were maxillary and mandibular premolar teeth with no caries lesion, and the exclusion criteria was tooth that had been restored.

Forty-five teeth that had been extracted were stored in saline solution. One week prior to the laboratory procedure, all tooth specimens were cleaned of calculus and soft tissues and stored in distilled water. The root parts of the specimens were removed. Each tooth crown was cut mesiodistally into two parts using a low-speed carbide disc under water spray. Following this, the crown parts were dried and embedded in self-cured acrylic resin inside a plastic mold measuring 3 cm in height and 2 cm in diameter. The dentin surfaces were exposed without contamination of the acrylic material.
Then, grinding of the specimens was performed at a constant low pressure using carbide 600-grit sandpaper. The specimens were divided into three groups, with 15 specimens per group. A different surface treatment was administered to each group. Specimens from each group were stored in saline water and incubated inside incubator at 37 °C for 24 hours.

Forty-five ceromer specimens were placed into a mold measuring 3 mm in diameter and 3 cm in height. They were then light cured in a Solidilite™ (Shofu Inc, Kyoto, Japan) unit for 3 minutes. Next, the specimens were cleaned with ultrasonic cleaner for 10 minutes. In this study, Breeze™ (Pentron Clinical, Wallingford, CT, USA) self-adhesive resin cement was used.

Group 1
The restoration specimen surfaces were layered with self-adhesive cement that had been automatically mixed through a dispensing and mixing system, and the specimens were bonded to the dentin surfaces. The excess cement was removed before the light-curing process was carried out. The light-curing process was done for 1 minute from the front and back sides. Then, the specimens were stored in saline water and incubated inside incubator at 37 °C for 24 hours. Following, the shear bond strength of the specimens was tested, and one specimen was tested using scanning electron microscopy (SEM).

Group 2
The exposed dentin surfaces were etched with 36% phosphoric acid for 15 seconds, washed with water for 10 seconds, and dried using low-pressure air spray. Next, bonding agents were applied for 10 seconds using a microbrush, and the specimens dried using low-pressure air spray for 5 seconds; this followed by polymerization using a dental curing light for 15 seconds. The surfaces of specimens were layered with self-adhesive cement that had been automatically mixed through its dispensing and mixing system, then bonded to the dentin surfaces. The excess cement was removed, and the light-curing process was carried out for 1 minute from the front and back sides. Then, the specimens were stored in saline water and incubated inside incubator at 37 °C for 24 hours. Following this, the shear bond strength of the specimens was tested, and one specimen was tested using SEM.

Group 3
The exposed dentin surfaces were etched with 36% phosphoric acid for 15 seconds, washed with water for 10 seconds, and dried using a low-pressure air spray. Following this, bonding agents were applied for 10 seconds using a microbrush and dried using a low-pressure air spray for 5 seconds. This was followed by polymerization using a dental curing light for 15 seconds. The bottom surfaces of the restoration specimens were layered with 9.6% hydrofluoric acid for 1 minute, then washed with water for 10 seconds and dried using a low-pressure air spray. Next, the surfaces of specimens that had gone through the etching process and silane application were layered with self-adhesive cement that had been automatically mixed through its dispensing and mixing system, then bonded to the dentin surfaces. The excess cement was removed, and then the light-curing process was carried out for 1 minute from front and back sides. Then, the specimens were stored in saline water and incubated at 37 °C for 24 hours. Following this, the shear bond strength of the specimens was tested, and one specimen was tested using SEM.

Observation and Measurement
The shear bond strength was tested using a universal testing machine. A chisel-shaped blade was placed on the ceromer with a 0.5 mm/minute pressure ratio and 50 kgf loads were added until the bond failure of the specimens occurred. The shear bond strength was measured in Kgf/mm² and converted into megapascals. After the shear bond strength test was conducted, one specimen from each group was randomly selected and tested using SEM to observe the bond failure.
3. Results and Discussion

3.1 Results

The data on shear bond strength were statistically tested using SPSS 20.0. First, tests of normality and homogeneity of the data were performed using the Shapiro–Wilk and Levene tests; the results showed that the data were normal and homogeneous. The next test was carried out using one-way ANOVA to test the mean difference between each group, and the post-hoc Bonferroni test was used to determine the significance of the relationships between the groups (p < 0.05).

**Table 1.** Mean, Standard Deviation (SD), and Significance Value of Shear Bond Strength (MPa)

| Group       | n  | Mean | SD   | p     |
|-------------|----|------|------|-------|
| Group 1     | 15 | 6.30 | 1.70 |       |
| Group 2     | 15 | 11.13| 2.98 |       |
| Group 3     | 15 | 15.77| 2.91 | 0.000 |

Significance value: p < 0.05

- Group 1: Without surface treatment
- Group 2: Surface treatment on dentin
- Group 3: Surface treatment on dentin and ceromer

Table 1 shows that the group with the highest mean value was group 3, with a value of 15.77 MPa, followed by group 2, with a value of 11.13 MPa. The group with the lowest mean value was group 1, with 3.52 MPa. When all three groups were compared, the significance value was p < 0.05, which means that there were significant differences among the three groups. As illustrated in Table 2, when each group was compared, significant differences were observed (p < 0.05). This indicates that surface treatment on dentin and intaglio ceromer can increase the shear bond strength.

**Table 2.** Significance value for each group

| Group               | P       |
|---------------------|---------|
| Group 1 vs. Group 2 | 0.000*  |
| Group 2 vs. Group 3 | 0.000*  |
| Group 3 vs. Group 1 | 0.000*  |

* Significance value: p < 0.05

- Group 1: Without surface treatment
- Group 2: Surface treatment on dentin
- Group 3: Surface treatment on dentin and ceromer

One dentin specimen from each group was randomly selected for SEM imaging (Figure 1). The image from group 1 in Figure 1(a) shows the dentin surface over the whole specimen, without resin cement materials or ceromer. Figure 1(b) shows a specimen from group 2; the dentin has open dentin tubules, and resin cement is still attached to the dentin portion. Figure 1(c) shows a specimen from group 3; it can clearly be observed that part of the resin cement has infiltrated into the dentin tubules, and on the top surface of the resin cement, a topographic pattern has formed because of the resin cement infiltration into the ceromer surface.
Figure 1. Scanning electron microscopy (SEM) images of a specimen from each group with 900× magnification: (a) group 1, (b) group 2, (c) group 3.

D: dentin
SR: resin cement
CR: Ceromer

3.2 Discussion

Table 1 shows that the shear bond strength value for group 1, in which Ceramage was directly cemented using self-adhesive resin cement bonded to dentin surface; in this group, the average value was 6.30 ± 1.70 MPa. This is almost the same as the results reported by Chen et al. (2010), 5.30 MPa, and Leon et al. (2012), which was 8.28 MPa [13, 16]. This proves that the use of self-adhesive resin cement without pretreatment on dentin will result in a low shear bond strength. Although it has been stated that the cement can form a mechanism of adhesion through micromechanical retention and chemical interaction between the acid monomer group and hydroxyapatite, Chen et al. (2010) revealed from SEM images generated in their study that there were no resin tags extending from the resin cement and no interlocking structure was formed; this means that micromechanical retention does not occur on the self-adhesive resin cement bond [16]. Several studies have reported that self-adhesive resin cement can interact superficially with the dentin surface, so the smear layer will be demineralized by acid monomer and form short resin tags [17, 18]. De Munck et al. studied the limited attachment interaction (100–200 nm) between self-adhesive resin cement and dentin. The strength of dentin-cement bonding is determined by the expansion of monomer infiltration into the demineralized collagen. The low bond strength of self-adhesive resin cement may be caused by the cement’s lack of ability to demineralize and infiltrate dentin. Although this cement has low initial pH (pH < 2) in the first minute, but the high viscosity may explain why there is no hybrid layer that forms when it is applied to the dentin [19].

In group 2, dentin surface treatment with application of 36% phosphoric acid etching and a bonding agent significantly enhanced the value of the shear bond strength (11.13 ± 2.98 MPa) compared with the group without surface treatment (6.30 ± 1.70 MPa) with a mean difference of 4.83 MPa (Table 1). These result contrasts with De Munck’s finding that pre-treatment with phosphoric acid suppressed the bonding power of self-adhesive resin cement to dentin. This is because although the smear layer is removed, it still leaves thick collagen that blocks the cement from penetrating the dentin [19]. Some other studies have also been performed to assess the effect of pre-treatment on dentin with some of the acid solution and found that the removal of the smear layer increases the interaction between self-adhesive resin cement and dentin. However, the use of phosphoric acid resulted in a decrease in bond strength because the presence of collagen fibers reduces cement penetration [20]. This difference was found in testing dentin-smoothing methods using diamond burs and carbide sandpaper with different grits, resulting in different smear layer thicknesses. However, the findings of this study are consistent with results of a previous study by Chen et al., where the application of phosphoric acid and a dentin bonding agent increased the shear bond strength between the self-adhesive resin cement and dentin, with resulting shear bond strength of 10.01 MPa [16]. In line research conducted by Lisbon (2013), the use of self-adhesive resin cement Rely X-Unicem™ for surface treatment using etching applications
on dentin resulted in a shear bond strength of $14.2 \pm 2.7$ MPa, while Biscem™ self-adhesive resin cement resulted in a shear bond strength of $5.3 \pm 1.3$ MPa; the result for the control group was $3.6 \pm 1.1$ MPa [21]. This can be explained in that phosphoric acid can remove the smear layer and smear plug, demineralize superficial dentin surface, and expose dentin tubules. Then, the self-adhesive resin cement can flow into the demineralized surface along with the opening of dentin tubules and form resin tags that contribute to micromechanical retention [16]. The results of this study showed a statistically significant value ($p \leq 0.05$); thus, dentin surface treatment can increase the shear bond strength of indirect ceromer restoration.

The bond between indirect ceromer restoration and tooth structure involves two different interfaces— that between the dentin/enamel and resin cement and that between the resin cement and restoration material. The bonding that occurs in indirect ceromer material is based on mechanical retention and chemical adhesion. Some indirect ceromer surface treatments have been studied in effort to enhance the bond between indirect ceromer restorations and resin cement. Improving surface roughness via several techniques can provide better mechanical interlocking and increase the amount of free-carbon residue left on a larger surface. Some of these techniques include sandblasting, hydrofluoric acid, or coarsening mechanically with a carbide bur. The technique is selected based on the type of restoration material that will be used. Another surface treatment is using silane to increase the chemical bond between the resin cement and indirect ceromer restoration [22]. According to the Ceramage manufacturer recommendations, restoration treatment prior to cementation should involve sandblasting on the adhesion surface using an air spray of $0.1–0.2$ MPa [4]. However, this technique is complicated, expensive, and not commonly available in everyday practice.

In this study, in group 3, dentin specimens underwent surface treatment with $36\%$ phosphoric acid for etching and then a bonding agent, while Ceramage restorations underwent surface treatment in the form of application $9.6\%$ hydrofluoric acid and silane. This test group showed a higher shear bond strength value compared to groups 1 and 2, $p \leq 0.05$ (Table 1). Thus, the second hypothesis in this study—that surface treatment on dentin and indirect intaglio ceromer restorations results in the highest shear bond strength values—is accepted.

The result can be explained in that Ceramage compositions contain ceramic materials, namely zirconium and silica. Hydrofluoric acid etching on zirconium cannot produce effective retention, since it contains no glass. The difference in the microstructure and composition of the ceramic is a determinant factor in the micromechanical retention obtained with acid etching. In the case of silica, etching with hydrofluoric acid from 4 to $9.5\%$ has been shown to increase surface hardness for mechanical interlocking [23]. Application of hydrofluoric acid to the indirect ceramic restoration surface will produce a topographic pattern where the micromechanical bonding will occur; moreover, the glassy matrix will be removed and the crystalline structure will be exposed. Hydrofluoric acid application will also increase the surface energy; thus, the adhesive resin cement will nicely wet the surface of the restoration [24, 25]. This surface will also help in providing sufficient surface energy so that silane can be introduced into this area. There are no specific standards on the concentration and duration of application of hydrofluoric acid. Chen et al. evaluated $2.5\%$ and $5\%$ hydrofluoric acid concentrations with different application lengths. The results showed that etching with a duration of over 30 seconds effectively increased the bond strength of resin cement [26]. Guler et al. evaluated the effect of $9.6\%$ hydrofluoric acid with different durations on porcelain. Their findings showed that hydrofluoric acid etching application for 120 seconds provided adequate bond strength [27].

Della Bona et al. stated that the mechanism of etching changes based on the type of etching material and duration, as well as the restoration material microstructure and composition. Therefore, it is difficult to compare these results with previous studies using different etching materials and protocols [28]. However, in the study by Zogheib, it was found that ceramic material requires a duration of hydrofluoric acid etching of at least 60 seconds to produce an adequate retentive surface with resin bonding [24].

The high amount of silica in Ceramage is what makes the chemical bond between silane and resin cement possible. This occurs because of silane’s hydrolysis and adsorption on the etching surface, as
well as its covalent bond with resin cement. Silane also increases the restoration surface’s wettability so that the resin cement can spread more widely into irregular areas [29].

This study has a weakness in that the cementation method was not carried out with static pressure; thus, the thickness of the cement left between the dentin and ceromer was not uniform. According to Panavelli et al., the static weight that can be provided to measure the shear bond strength is 2 kg for 1 minute [10].

Based on SEM imaging, group 1 showed that the resin cement was completely separate in the dentin specimens, and a thick smear layer was visible. This was also found in Garcia’s study, which examined the bonding of self-adhesive resin cement to dentin [30]. It can be assumed that the methacrylate phosphoric acid contained in self-adhesive resin cement is not capable of reducing the smear layer; thus, it blocks resin and dentin from bonding. Moreover, the dentin is not demineralized enough, so that the resin cement cannot penetrate further into the dentin tubules. In group 2, dentin tubules were exposed by the phosphoric acid and resin cement bonded to the dentin surface, but the Ceromer material was entirely detached from the resin cement. This group showed results in accordance with those of Chen et al., who found that the surface treatment of dentin with 36% phosphoric acid etching applications could remove smear layer and smear plug; thus, the dentin surface was demineralized, so that the dentin tubules were open and the resin cement could form resin tags that contributed to enhancing retention [16]. In group 3, parts of the resin cement that infiltrate into the dentin tubules, and at the top surface of resin cement, a topographic pattern is formed because of resin infiltration into the Ceromer surface that had been etched with hydrofluoric acid. Surface treatment via intaglio ceromer dissolves the filler component of the restoration and produces a porous surface as a place to form retention.

4. Conclusion
Dentin surface treatment can increase the shear bond strength of self-adhesive resin cement. Surface treatment on dentin and indirect intaglio ceromer restoration had the highest shear bond strength of the treatments used in this study. Further research should be done using a static load during the cementation period to obtain more accurate results.

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