Adhesive bonding to polymer infiltrated ceramic

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Aim of this study was to investigate the mechanism of adhesive bonding to the polymer-infiltrated ceramic VITA Enamic [VE]. Shear bond strength was measured with three resin composite cements: RelyX Unicem 2 Automix, Clearfil SA and Variolink II on polished surfaces of VE and its components silicate ceramic [SC] and polymer [PM] (n=12). Further, the effect of etching VE with 5% HF for 15–240 s and the application of silane coupling agents was analyzed in a screening test (n=6). Shear bond strength measurements were performed after 24 h of water storage at 37°C. Significant bonding to polished substrates could only be achieved on VE and SC when silane coupling agents were used. Etching of VE with 5% HF increased shear bond strength. Following silanization of etched VE, a further increase in shear bond strength could be established. Etching for more than 30 s did not improve shear bond strength.

Keywords: Polymer infiltrated ceramic, Hybrid ceramic, Shear bond strength, Cement, Surface pre-treatment

INTRODUCTION

Silicate ceramics and resin composite materials are commonly used in dental CAD/CAM technology. Recently, a polymer infiltrated ceramic was developed as a completely new type of CAD/CAM material10. The polymer infiltrated ceramic was designed to combine the benefits of both ceramic and composite materials. According to the manufacturer, it contains in weight 86% silicate ceramic and 14% polymer. The polymer composition includes UDMA and TEGDMA11. Contrary to composite materials, where ceramic fillers are embedded in a continuous resin matrix, the polymer infiltrated ceramic is composed of two interpenetrating networks, resulting in a reduced fracture susceptibility of the material10.

An important factor for clinical success of a tooth restoration is cementation29. Luting cements link restorations with the supporting tooth structure30. Different luting cements such as conventional luting cements, adhesive resin composite cements and self-adhesive resin composite cements are available. Thereby, the link may be of mechanical and/or chemical nature on both restoration and tooth structure23. Restorations made of silicate ceramic benefit from luting with resin composite cement because of increased fracture resistance due to adhesive bonding23,30. However, luting with adhesive resin composite cement requires technique-sensitive pre-treatment of the tooth structure23,30. Compared with adhesive resin composite cements, conventional cements such as glass ionomer cements are easy to apply, but their mechanical properties are inferior. In terms of improvement, self-adhesive resin composite cements were developed with the aim of combining the benefits of adhesive luting with the simple handling of conventional cements. Self-adhesive resin composite cements are not only self-adhesive but also self-conditioning, meaning, they do not need any additional pre-treatment of enamel or dentine6. Self-conditioning and self-adhesion is both provided either by phosphoric acid groups incorporated in the resin monomer or by special coupling monomers such as 10-methacryloyloxydecolyl dihydrogen phosphate (MDP).

Bonding to ceramic surface requires pre-treatment to achieve high bond strength30. Usually, ceramic restorations are pre-treated by a combination of physical and chemical measures35. Physical methods include roughening of the surface with a diamond bur, sandblasting with alumina or etching with hydrofluoric acid (HF)5,6. During HF etching, parts of the silicate ceramic surface are removed, resulting in surface roughness5,7. A chemical approach to adhere to silicate ceramic is the application of a silane coupling agent, which provides a chemical link between resin composite cement and silicate ceramic5. Silane coupling agent contains a bifunctional molecule, which is able to bind covalently to silicon dioxide as well as to copolymerize with the organic matrix of resin composite cement5,7. In a study on feldspathic ceramic, etching with HF followed by an application of silane coupling agent turned out to be the most efficient and reliable combination of conditioning methods6.

To achieve a sufficient bond to resin surface, pre-treatment is important, too6. The bond to the resin surface may be enhanced by mechanical and chemical means6. Theoretically, chemical bonding between methacrylate monomers is a possible bonding mechanism between resin composite cement and resin based restorative material23,9. Methacrylate monomers...
from the resin composite cement may copolymerize with unreacted vinyl groups of the polymeric material. Wetting the polymeric resin surface with adhesive resin or monomer liquid leads to a swelling of the polymer, which is reported to enhance adhesive bonding. However, if there is a high degree of conversion in the resin material, bond can mainly be attributed to micromechanical retention achieved through mechanical roughening of the resin surface. Micromechanical retention on polymeric surfaces is commonly gained by air particle abrasion, which increases surface area resulting in higher bond strength.

The bonding mechanism of resin composite cements to silicate ceramics or resin composite materials is widely understood. However, there is no data available on the bond characteristics of resin composite cements to the polymer infiltrated ceramic. Therefore, the aim of the present study was to evaluate the bonding mechanism of resin composite cements to the polymer infiltrated ceramic in order to provide a recommendation for the clinical procedure.

**MATERIAL AND METHODS**

**Preparation of specimens**

Shear bond strength measurements were performed on three different materials: polymer infiltrated ceramic (VITA Enamic, VITA Zahnfabrik, Bad Säckingen, Germany) [VE] and its components silicate ceramic [SC] and polymer [PM]. According to the manufacturer’s specification VE is composed of a fine structure alumina-enriched feldspar ceramic network (86% by weight), while the polymer is a mixture of two dimethacrylates: UDMA and TEGDMA (14% by weight). Dense blanks of SC were obtained by complete sintering of porous ceramic blanks without any polymer infiltration. PM was obtained by polymerizing the monomer mixture of UDMA and TEGDMA under the same conditions as used for the production of VE. All blanks were provided by the manufacturer (VITA). Specimens were produced by fixing an acrylic cylinder (D+R Tec, Birmensdorf, Switzerland) on the specimen’s surface. The acrylic cylinder had an inner diameter of 2.9 mm. Resin composite cements were mixed according to the manufacturer’s instructions, filled into the cylinder and cured for 2 min respectively, first in distilled water, then in 70% ethanol using an ultrasonic bath (TPC-15, Telesonic, Bronschhofen, Switzerland).

Shear bond strength tests were performed on polished surfaces (n=12). To assess the effect of mechanical bonding in a screening test, polished polymer infiltrated ceramic specimens were etched with 5% HF (VITA Ceramics Etch, VITA Zahnfabrik) for 15, 30, 60, 120 or 240 s prior to bonding (n=6).

Three resin composite cements were chosen for the bond strength tests (Table 1): two self-adhesive resin composite cements (RelyX Unicem 2 Automix [RXU], 3M ESPE, Seefeld, Germany and Clearfil SA [CSA], Kuraray-Noritake, Kurashiki, Japan) as well as one multistep adhesive resin composite cement (Variolink II [VL2], Ivoclar Vivadent, Schaan, Liechtenstein). Each material combination was analyzed with or without use of the corresponding silane coupling agent (Table 1). Silane coupling agents were applied to the specimen’s surface with a micro-brush and then dried with oil-free air as recommended by the manufacturers.

The shear test design used in this study was already specified in previous publications. Resin composite cement was applied to the specimen’s surface in a standardized way, using a special device fixing an acrylic cylinder (D+R Tec, Birmensdorf, Switzerland) on the specimen’s surface. The acrylic cylinder had an inner diameter of 2.9 mm. Resin composite cements were mixed according to the manufacturer’s instructions, filled into the cylinder and loaded with 10 N by means of a steel set screw (BN 617, Bossard, Zug, Switzerland). Light polymerization with a LED lamp (Elipar S10, 3M ESPE, 1,200 mW/cm²) was performed for 20 s (RXU and CSA) or 40 s (VL2) from two opposing sides and the light source was held as near to the specimen as possible. Specimens were carefully removed from the device 10 min after light curing and stored in water at 37°C for 24 h.

**Shear bond strength measurements**

Shear bond strength was measured with a universal testing machine (3344, Instron, Canton, MA, USA). Specimens were fixed in a special device, where the

| Sample material                | Code  | Manufacturer                              | Lot. No. |
|-------------------------------|-------|-------------------------------------------|----------|
| RelyX Unicem 2 Automix        | RXU   | 3M ESPE, Seefeld, Germany                 | 529196   |
| +RelyX Ceramic Primer         | RXCP  | 3M ESPE                                   | N453546  |
| Clearfil SA                   | CSA   | Kuraray-Noritake Dental, Kurashiki, Japan | AR0020   |
| +Clearfil Ceramic Primer      | CFCP  | Kuraray-Noritake Dental                   | 00024D   |
| Variolink II                  | VL2   | Ivoclar Vivadent, Schaan, Liechtenstein  | S10280   |
| +Monobond Plus                | MBPL  | Ivoclar Vivadent                          | R03105   |
acrylic cylinder was sheared parallel to the substrate surface by a loading piston. Load was applied in a distance of 0.2 mm from the substrate surface and with a cross-head speed of 1 mm/min. The load at de-bonding was registered and shear bond strength values were calculated.

**Classification of failure type**

Scanning electron microscopy (ESEM Philips XL Series XL30 FEG, Philips Electron Optics, Eindhoven, The Netherlands) was used to document the surface topography after polishing, etching and de-bonding and to identify the different types of failure: (a) adhesive (failure occurred in the interface), (c) cohesive (failure occurred in the specimen material or in the cement), (m) mixed (failure occurred partially in the specimen material, partially at the interface).

**Statistical analysis**

A one-way analysis of variance (ANOVA) was used to assess significant differences between the tested resin composite cements and between substrates. Mean values were compared at a level of significance of $p=0.05$.

**RESULTS**

**Shear bond strength to polished substrates**

The two self-adhesive resin composite cements RXU and CSA were not able to establish adhesion to polished polymer surface, neither in untreated nor in silanized condition (Fig. 1). After water storage, all specimens had de-bonded and bond strength was set to 0 MPa. In case of the multistep adhesive resin composite cement VL2, low adhesion to untreated polymer surface could be detected: Two specimens bonded without silane coupling agent exhibited moderate bond strength of 4.6 MPa and 5.8 MPa, respectively. As the remaining 10 specimens spontaneously debonded during water storage, their shear bond strength was set to 0 MPa, resulting in an overall average bond strength value of 0.9±2.0 MPa. With application of silane coupling agent, no bonding was observed.

To untreated polished silicate ceramic, only CSA showed low adhesion (0.3±0.6 MPa) (Fig. 1). Four out of 12 specimens outlived water storage. But when a silane coupling agent was used, all of the tested resin composite cements established adhesion to polished silicate ceramic surface. The shear bond strength values of RXU and CSA did not significantly differ on silanized silicate ceramic (RXU: 8.1±3.4 MPa; CSA: 9.3±2.4 MPa; $p=0.348$). Highest mean shear bond strength on silanized silicate ceramic was found with VL2 in combination with MBPL (19.0±3.7 MPa, $p=0.000$ vs. RXU and $p=0.000$ vs. CSA).

Similar to the polished silicate ceramic substrate, only CSA showed low adhesion to the untreated polished polymer infiltrated ceramic surface (0.7±0.7 MPa) (Fig. 1). On silanized polymer infiltrated ceramic surface, shear bond strength values of RXU and CSA were again statistically similar (RXU: 6.4±4.5 MPa; CSA: 9.1±2.0 MPa; $p=0.087$). Significantly higher mean shear bond strength was shown by VL2 in combination with MBPL (13.3±4.7 MPa; $p=0.002$ vs. RXU and $p=0.012$ vs. CSA).

The patterns of the results are similar for polished silicate ceramic and polished polymer infiltrated ceramic (Fig. 1). VL2 exhibits statistically higher shear bond strength on SC than on VE ($p=0.004$), whereas shear bond strength of RXU and CSA did not differ on SC and VE ($p=0.338$ and $p=0.833$, respectively) (Fig. 1).

The failure type on polished surfaces (Fig. 2) differed according to the specimen material and the
Table 2  Frequency of failure types on polished specimens (a: adhesive, m: mixed, c: cohesive)

| Specimen                  | Polymer                | Silicate ceramic         | Polymer infiltrated ceramic |
|---------------------------|------------------------|--------------------------|-----------------------------|
|                           | a   | m   | c   | a   | m   | c   | a   | m   | c   |
| Resin composite cement    |     |     |     | 12  |     |     | 12  |     |     |
| RXU                       |     |     |     |     |     |     |     |     |     |
| CSA                       |     |     |     | 12  |     |     | 10  | 2   |     |
| VL2                       |     |     |     | 12  |     |     | 12  |     |     |
| Resin composite cement    | w/o silanization       |                          |                             |
| RXU                       |     |     |     | 12  |     |     | 12  |     |     |
| CSA                       |     |     |     | 12  |     |     | 10  | 2   |     |
| VL2                       |     |     |     | 12  |     |     | 12  |     |     |
| Resin composite cement    | w silanization         |                          |                             |
| RXU                       | 12  | 5   | 6   | 12  |     |     |     |     |     |
| CSA                       | 12  | 9   | 3   | 12  |     |     |     |     |     |
| VL2                       | 12  | 9   | 3   | 12  |     |     |     |     |     |

Fig. 3  SEM of polymer infiltrated ceramic surfaces after different etching times (×1,000).
1: ceramic fraction, 2: polymer fraction.

Fig. 4  Shear bond strength to etched polymer infiltrated ceramic surfaces (means and standard deviations).

Compared with bonding to polished polymer infiltrated ceramic, etching with 5% HF increased shear bond strength (Fig. 4). Without the use of silane coupling agent RXU and VL2 reached similar levels of shear bond strength, whereas shear bond strength of CSA remained on a lower level. Etching for more than 30 s was not beneficial.

The additional use of silane coupling agent initially led to higher levels of shear bond strength (Fig. 4). Highest shear bond strength values could be found among briefly etched (15–30 s) and silanized polymer infiltrated ceramic. The peak of shear bond strength was at 15 s of surface etching for resin composite cement RXU (26.5±3.3 MPa) and at 30 s of surface etching for resin composite cements CSA (19.8±3.4 MPa) and VL2 (22.4±1.8 MPa) (Fig. 4). By trend, with a longer duration of the surface etching, the benefit of additional silanization became smaller and the shear bond strength values decreased (Fig. 4). Pooling the values measured on etched surfaces revealed a significant difference between the shear bond strength obtained without pre-treatment. On polished specimens, mostly adhesive type of failure occurred, with the exception of silanized silicate ceramic, where predominantly cohesive or mixed failure type was found (Table 2).

Shear bond strength to etched polymer infiltrated ceramic
Scanning electron microscopy of the etched polymer infiltrated ceramic surfaces showed progressive dissolution of the ceramic fraction with the degree of acid attack. Subsequently, the formation of undercuts increased due to the increasing exposure of the polymer network (Fig. 3).
Fig. 5 SEM of different failure types on etched polymer infiltrated ceramic (VE).
a: adhesive failure type (RXU 15 s w/o silanization), m: mixed failure type (VL2 120 s with silanization), c: cohesive failure type (RXU 240 s with silanization) ×35.

Table 3 Frequency of failure types on etched specimens (a: adhesive, m: mixed, c: cohesive)

| Etching duration | Failure type | 15 s | 30 s | 60 s | 120 s | 240 s |
|------------------|--------------|------|------|------|-------|-------|
|                   | Resin composite cement w/o silanization |      |      |      |       |       |
| RXU               | a            | 3    | 3    | 6    | 5     | 1     |
|                  | m            | 6    | 6    | 6    | 6     | 1     |
|                  | c            | 6    | 6    | 6    | 6     | 6     |
| CSA               | a            | 6    | 6    | 6    | 6     | 6     |
|                  | m            | 6    | 6    | 6    | 6     | 6     |
|                  | c            | 6    | 6    | 6    | 6     | 6     |
| VL2               | a            | 6    | 6    | 6    | 6     | 6     |
|                  | m            | 6    | 6    | 6    | 6     | 6     |
|                  | c            | 6    | 6    | 6    | 6     | 6     |
| Resin composite cement w silanization |      |      |      |       |       |       |
| RXU               | a            | 1    | 1    | 4    | 3     | 2     |
|                  | m            | 6    | 6    | 6    | 6     | 6     |
|                  | c            | 4    | 4    | 4    | 4     | 4     |
| CSA               | a            | 6    | 6    | 6    | 6     | 6     |
|                  | m            | 3    | 3    | 3    | 3     | 3     |
|                  | c            | 1    | 1    | 1    | 1     | 1     |
| VL2               | a            | 2    | 2    | 2    | 2     | 2     |
|                  | m            | 4    | 4    | 4    | 4     | 4     |
|                  | c            | 1    | 1    | 1    | 1     | 1     |

application of silane coupling agent (15.4±4.1 MPa) and that obtained with application of silane coupling agent (19.9±4.8 MPa) (p<0.001).

Failure types on etched polymer infiltrated ceramic (Fig. 5) depended on silanization more than on the duration of the surface etching (Table 3). Resin composite cement did not have any influence on failure type. Adhesive failure was rare, with the exception of 15 s etched non-silanized specimens. On non-silanized specimens, mixed type of failure was predominant, whereas on silanized specimens cohesive type of failure was frequently seen. No correlation between failure types and shear bond strength measurements could be found.

**DISCUSSION**

Shear bond strength to polished substrates
In a first step, bonding to polished substrates was investigated in order to exclude micromechanical interlocking. Thus the extent of chemical adhesion was measured. In general, without silanization, no or minor adhesion was obtained, even on the polymer substrate. It is assumed that as a result of the industrial polymerization process of the polymer the number of reactive vinyl groups available on the surface of the resin substrate was quite limited and therefore no or insignificant chemical bonding occurred. In no case the respective silane coupling agent was able to improve the bonding of resin composite cement to polymer. These findings correlate with results of a study, where the bonding of self-adhesive resin composite cements, among them RXU, to resin composite restorative materials was
investigated under different pre-treatment protocols, of which one was silanization with RXCP. In that study the use of silane coupling agent was unable to improve the bonding of self-adhesive resin composite cements to resin composite restorative materials. A further study reported no adhesion of resin composite cement to untreated polymeric surface when the effect of surface conditioning on the tensile stress of polymeric CAD/CAM crowns with resin composite cements RXU and VL2 was examined. Regarding the chemistry of silane coupling agents, it is obvious that the bifunctional molecule is not capable of simultaneously bonding to the polymer substrate and to the resin composite cement.

Only CSA showed low adhesion to non-silanized polished silicate ceramic (0.3±0.6 MPa) and polymer infiltrated ceramic (0.7±0.7 MPa). The reason for this may be the MDP content of CSA cement, which enables bonding to silicate ceramic and the ceramic part of polymer infiltrated ceramic. The MDP-monomer, with its phosphate ester group, binds directly to surface oxides, and its methacrylate group enables copolymerization with the resin matrix of the luting cement.

Silane coupling agents provided considerable bond strength both on SC and VE, since silicate surfaces such as SC or VE allow for covalent Si-O-Si bonds between ceramic and silane coupling molecules. As on silanized SC nearly no adhesive, but cohesive or mixed fractures were observed, it can be concluded, that the bond strength between resin composite cement and substrate, established by silane coupling molecules, exceeds the intrinsic strength of the substrate material itself. As no interfacial shearing occurred, the results may not be considered as shear bond strength data. On silanized VE, only adhesive failure was observed. The effect may be explained by the fact that the polished polymer infiltrated ceramic surface is a mosaic of polymer and ceramic in the micrometer scale. As was seen before, the silane coupling agent did not bind to the polymer surface. Cracks were not able to progress into the ceramic because they would be directed to the nearest polymer area, which acts as a predetermined breaking point.

All three silane coupling agents used contain MPTS. However, MBPL was the most efficient agent. This could be explained by the presence of methacrylate phosphoric acid ester, which might provide additional bonding capacity.

Shear bond strength to etched polymer infiltrated ceramic

SEM analysis revealed that HF etching removed the ceramic part of the polymer infiltrated ceramic and exposed the polymer network. Consequently, HF etching increased surface roughness and provided undercuts, which are beneficial for micromechanical retention.

When the polymer infiltrated ceramic was etched with HF, a significant increase in shear bond strength was detected with all of the tested resin composite cements, compared to the results obtained with polished surfaces. This can be attributed to micromechanical retention created by roughening the polymer infiltrated ceramic surface and the resin composite cement flowing into undercuts. Roughening of the polymer infiltrated ceramic surface seems to be sufficient even when the polymer infiltrated ceramic was etched for only 30 s, since shear bond strength remained constant with further etching.

When a silane coupling agent was used on the etched polymer infiltrated ceramic, higher values in shear bond strength could be observed. The increase in shear bond strength can be explained by additional chemical linkage of the silane coupling agents to the silicate ceramic phase of the polymer infiltrated ceramic.

Excessive etching led to an increased bonding area, which is beneficial for micromechanical retention. On the other hand, it reduced the ceramic surface and thereby the surface available for a chemical linkage by silane coupling molecules. Hence, the more polymer infiltrated ceramic is etched, the less chemical bonding is possible and the more shear bond strength is provided by the micromechanical interlocking between the resin framework and the adhesive cement. Moreover, when polymer infiltrated ceramic is strongly etched, the silane coupling agent may also form puddles in deeper cavities, where it cannot be dispersed completely and consequently acts as a barrier because of the remaining solvent. These two aspects might explain the merging of the shear bond strength values of silanized specimens towards those values obtained by non-silanized specimens after prolonged etching.

The low level of shear bond strength values of CSA on non-silanized polymer infiltrated ceramic is striking. Due to the presence of MDP it may be supposed that CSA is slightly more hydrophilic and shows less wetting of the resin matrix of the polymer infiltrated ceramic than the other two cements. At least, it was shown that the contact angle of water on a silicate ceramic, with a composition very near to that of the ceramic matrix of the polymer infiltrated ceramic, is lower than the contact angle on the etched polymer infiltrated ceramic. In the present study, CSA was the only cement, which slightly bonded to untreated SC and VE. That effect underlines the more hydrophilic character of CSA in comparison with RXU and VL2.

The shear bond strength values of the different resin composite cements did not considerably differ when polymer infiltrated ceramic was etched for 30 s and silanized. This indicates that the choice of the resin composite cement is of an inferior importance when bonding to the polymer infiltrated ceramic. More important is that the polymer infiltrated ceramic surface is being etched and silanized. A high prevalence of cohesive and mixed failure types implies a strong bond to etched and silanized polymer infiltrated ceramic, which even exceeded the fracture resistance of the material itself.

In a recently published investigation, bond strength to the polymer infiltrated ceramic was measured by micro-tensile bond strength. Specimens were cut from blanks, the surfaces ground with 600 grit silicon
carbide paper and HF etched for 60 s. After bonding and light curing, specimens were stored for 24 h at 37°C in a water bath and subsequently subjected to 10,000 thermal cycles. Among others, bonding was performed with RXU. As silane coupling agent, MBPL was used. Bonding without silanization resulted in a bond strength of 16.1±4.7 MPa, with silanization, a value of 20.2±4.9 MPa was obtained. Both values are very close to the results we obtained (18.0±3.3 MPa and 23.5±2.3 MPa, respectively), although different test methods were employed.

Limitations of the study and future investigations
The number of 6 specimens per measuring point for assessing the effect of surface etching and micromechanical retention on bond strength may be considered as too low. With a higher number of specimens, probably more precise results would have been obtained. However, the measuring curves are quite smooth, indicating that the obtained results, at least fundamentally, represent the actual conditions.

On etched surfaces, most of the specimens showed mixed or cohesive failure types. Therefore, the applied test is not a shear test in the proper sense. The finding, however, that chemical bonding and interlocking of the resin composite cement to the substrate material are stronger together than the substrate material itself, is a valuable information on the performance of resin composite cements, justifying the use of the applied test design.

The present results were obtained after 24 h water storage with only 3 different resin composite cements. The effect of aging by thermal cycling was not investigated. Future tests must include additional resin composite cements and thermal cycling.

An interesting question for further research would be the comparison of VE with conventional resin composite restorative materials, considering the composition of the two materials being fundamentally the same, but differing crucially in micro-structure.

CONCLUSION
In order to adhesively bond to the polymer infiltrated ceramic VITA Enamic, it is recommended to etch with 5% HF for 30–60 s, to then thoroughly wash and dry the etched surface and apply a silane coupling agent corresponding to the resin composite cement.

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