In vitro bonding effectiveness of new self-adhering flowable composite to calcium silicate-based material

Cemal YESILYURT¹, Kadir T. CEYHANLI², Cemile KEDİÇİ ALP¹, Tahsin YILDIRIM¹ and Tamer TASDEMİR²

¹ Department of Restorative Dentistry, Faculty of Dentistry, Karadeniz Technical University, Trabzon, Turkey
² Department of Endodontics, Faculty of Dentistry, Karadeniz Technical University, Trabzon, Turkey
Corresponding author, Kadir T. CEYHANLI; E-mail: tolgaceyhanli@hotmail.com

The purpose of this study was to determine the in vitro bonding effectiveness of Vertise Flow (VF), a new self-adhering flowable composite (FC), and Ultimate Flow (UF), a conventional FC, to BioAggregate (BA), and to investigate whether the use of self-etch or etch-and-rinse adhesive improved the bond strength of VF to BA. Shear bond strengths of FC to BA were measured for this reason. Data were analyzed using Wilcoxon and Mann-Whitney U-tests (p<0.05). There were significant differences of bond strengths after 24- and 72-h (p<0.05, p<0.001). After 72-h, self-adhering FC may be used successfully with BA and no other adhesive, as an alternative to the combined use of conventional FC and self-etch adhesives. The use of etch-and-rinse adhesives with self-adhering FC may improve adhesion to BA.

Keywords: Self adhering composite, BioAggregate, Bonding

INTRODUCTION

Mineral trioxide aggregate (MTA), the first retrograde filling material based on calcium silicate, was introduced in 1993¹². MTA is a powder consisting of hydrophilic particles of tricalcium aluminate, tricalcium silicate, and tricalcium oxide. It also contains small amounts of other mineral oxides that modify its chemical and physical properties¹³. Several calcium silicate-based materials, such as MTA Angelus, BioAggregate (BA, Innovative BioCeramix, Vancouver, Canada), and tricalcium silicate cement have been described recently⁴. Most constituents of these products are similar to those of white MTA⁶. Calcium silicate-based materials are currently used for a variety of applications in endodontics and restorative dentistry⁵, such as direct pulp capping⁶, apexification⁷, external root resorption repair⁸, partial pulpotomy⁹, and the treatment of large periapical lesions¹⁰.

In vital pulp therapy using MTA, the choice of restorative material to be applied over MTA is important. Previous studies have emphasized that glass-ionomer cement can be used as intermediate material and that composites can be used for final restoration over MTA¹¹-¹³. However, acid etching before composite placement reduces the compressive strength and surface microhardness of MTA¹⁴. The nature of the solvent (acetone, ethanol, or water) and the filler content of the adhesive may also influence the bond strength of MTA to resin¹⁵. Furthermore, materials used for coronal restoration over MTA should have low condensation forces¹⁵.

The FC with no compression force may be placed over calcium silicate-based materials with low condensation forces. Vertise Flow (VF; Kerr, Orange, CA, USA), a new-generation FC, was recently introduced in the market¹⁶. This self-adhering composite was designed to bond to tooth substrate without need for a separate adhesive or etching step¹⁷. The bonding mechanism of self-adhering composite relies on a monomer glycerol phosphate dimethacrylate (GPDM) adhesive¹⁸. The manufacturer of VF has claimed that its shear bond strengths to dentin and enamel are statistically similar to those of commercially available self-etch adhesive/FC systems. Vichi et al.¹⁸ reported that VF showed lower bond strengths to dentin and enamel, but superior marginal sealing ability in comparison with all-in-one adhesive systems. Studies examining the physical properties¹⁹-²¹, bond strengths, and marginal microleakage²² of self-adhering composites have been published, but no reported study has investigated the bonding effectiveness of this simplified restorative material to calcium silicate-based materials. The placement of a restorative material with no compression force over a calcium silicate-based material without a separate adhesive may eliminate the adverse effects of adhesive application and limit handling errors.

The purpose of this study was to determine the shear bond strength of VF to BA after 24- and 72-h storage periods in comparison with that of a conventional FC used in combination with dentin adhesives. The secondary objective was to investigate whether the use of an etch-and-rinse or self-etch adhesive improved the bond strength of VF to BA.

The tested null hypothesis was that there would be no difference between the bond strengths obtained by self-adhering FC and conventional FC to BA. Secondly,
the use of VF combined with self-etch or etch-and-rise adhesive would improve the VF/BA bond strengths.

MATERIALS AND METHODS

Table 1 lists the materials used in this study, including the calcium silicate-based material, self-adhering FC, conventional FC, and two adhesive systems (Clearfil SE Bond and Scotchbond Multi-Purpose).

Specimen preparation

BA specimens (n=144) were prepared using cylindrical acrylic blocks, each of which had a central hole with a 4-mm diameter and 2-mm depth. The BA was mixed according to the manufacturer’s instructions, poured into the holes in the acrylic blocks, and covered with a moist cotton pellet and temporary filling material (Cavit; ESPE America, Norristown, PA, USA). Before any adhesive and/or composite was placed, half of 144 prepared BA specimens were stored for 24 h at 37°C and 100% humidity and the remaining half of the specimens stored for 72 h in the same medium. After storage, the temporary material and cotton pellet were removed; the BA surface was not rinsed or polished. The specimens were then divided into the following groups (n=12) each:

Table 1  Manufacturers, composition, application methods and lot numbers of the tested materials

| Product name               | Composition                                                                 | Application                                                                                   | Manufacturer                  |
|----------------------------|----------------------------------------------------------------------------|---------------------------------------------------------------------------------------------|-------------------------------|
| BioAggregate (BA)          | Tricalcium silicate, Dicalcium silicate, Tantalum pentoxide, Calcium phosphate monobasic, Amorphous silicon oxide | • Mix BioA liquid and BioA powder for about 2 min                                              | BA; Innovative BioCeramix, Vancouver, Canada Lot: 0701BA |
| Vertise Flow (VF)          | GPDM, HEMA, Prepolymerized filler (20 μm), barium glass filler (0.7–1-μm), nano-sized colloidal silica (10–40 nm), nano-sized Ytterbium fluoroide (40 nm), Zinc Oxide pH:1.9, 70 wt% filler content | • Air-drying of the cavity; • Brush a thin layer (<0.5 mm) of Vertise Flow for 15–20 s; Light cure for 20 s | Kerr, Orange, CA, USA Lot: 3612354 |
| Ultimate Flow (UF)         | Bis-GMA, Bis-EMA, TEGDMA, Procrylat resins, ytterbium trifluoride silica filler 65 wt% filler content | • Place and light cure restorative in increments                                                  | 3M ESPE, St. Paul, MN, USA Lot: N332287 |
| Clearfil SE Bond (Self-etch adhesive) | Primer: MDP, HEMA, hydrophilic dimethacrylate, dl-Camphorquinone, water Bond: MDP, HEMA, Bis-GMA, dl-Camphorquinone, hydrophilic dimethacrylate, Colloidal silica, PH: 2.0 | • Apply primer 20 s; • Wait 20 s; • Use air spray to evaporate the volatile ingredients; • Light cure the bond for 10 s | Kuraray Noritake Dental, Tokyo, Japan Lot: 01093A (Primer) Lot: 01636A (Bond) |
| Scotch Bond Multi-Purpose Adhesive System (Etch-and-rinse adhesive) | Primer: HEMA, poly alkenoic copolymer, water Resin: Bis-GMA, HEMA | • Apply phosphoric acid, wait 15 s, rinse for 15 s; • Apply primer and dry gently for 5 s; • Apply adhesive; • Light cure for 10 s | 3M ESPE, St. Paul, MN USA Lot: N221115 (Primer) Lot: N223916 (Bond) |
with light curing was provided to ensure the entire packed composite was light-cured. BA/FC specimens were stored for 24-h (n=72 each) at 37°C and 100% humidity.

Shear bond strength measurement
To measure shear bond strength, each specimen was mounted in a universal testing machine (Lloyd Instruments, Fareham, UK). A steel knife blade was placed as closely as possible to the bonded surface (BA/FC). The shear bond strength test was performed at a crosshead speed of 0.5 mm/min, and the results were recorded in Newtons and converted to megapascals (MPa).

Fracture surface analysis
Fractured surfaces of specimens were observed under a stereomicroscope (Stemi 2000-C; Carl Zeiss, Göttingen, Germany) to determine the type and location of failure. Failure type was recorded as adhesive (two flat surfaces, indicating failure of the BA/resin bond), cohesive (represented by any deficiency in the BA surface), or mixed (dentin adhesive remnants and deficiency in the BA surface) (Fig. 1).

Table 2  Failure type of fracture surfaces

| Groups                  | Fracture Type    | VF 24-h | VF 72-h | UF 24-h | UF 72-h |
|-------------------------|------------------|---------|---------|---------|---------|
|                         | Adhesive         | 12      | 12      | 12      | 12      |
|                         | Cohesive         | —       | —       | —       | —       |
|                         | Mix Type         | —       | —       | —       | —       |
| 1- BA/FC                | Adhesive         | —       | —       | —       | —       |
|                         | Cohesive         | 7       | 9       | 10      | 11      |
|                         | Mix Type         | 5       | 3       | 2       | 1       |
| 2- BA/self-etch adhesive/FC | Adhesive     | —       | —       | —       | —       |
|                         | Cohesive         | 10      | 12      | 11      | 12      |
|                         | Mix Type         | 2       | —       | 1       | —       |
| 3- BA/etch-and-rinse adhesive/FC | Adhesive   | —       | —       | —       | —       |
|                         | Cohesive         | —       | —       | —       | —       |
|                         | Mix Type         | —       | —       | —       | —       |

Statistical analysis
The means and standard deviations of values were calculated. The normality test of Shapiro-Wilks was applied to the data, and all data were not found normally distributed. Thus, the comparisons between the groups were analyzed with non-parametric tests. The Mann-Whitney U-test was used to compare shear bond strength results among groups, and Wilcoxon rank sum tests were used to evaluate the significance of differences between specimens stored for 24- and 72-h. The level of significance was set to $p<0.05$.

RESULTS
Figure 2 shows mean shear bond strengths for all groups. The use of self-etch adhesive significantly reduced the bond strength of VF to BA, but the application of etch-and-rinse adhesive were significantly increased bond strength after 24- and 72-h ($p<0.05$). The use of both self-etch and etch and rinse adhesive

![Fig. 1](Representative image for failure types.
a. Adhesive failure, b. Cohesive failure, c. Mixed failure.)

![Fig. 2](Mean shear bond strength values of two FC to BA (n=12). The vertical bar indicates standard deviation.)
significantly increased the bond strength of UF to BA, but the application of etch-and-rinse adhesive was more effective than that of self-etch adhesive ($p<0.05$). Bond strength values were significantly higher in the VF-1 group than in the UF-1 (control group) after 24- and 72-h ($p<0.05$). Among specimens bonded after 24-h, shear bond strength was lower in the VF-1 group than in the UF-2 and UF-3 groups ($p<0.05$). Among specimens bonded after 72-h, bond strength was similar in the VF-1 and UF-2 groups ($p>0.05$), but significantly lower than that of the UF-3 group ($p=0.001$; Fig. 2). In all groups, mean shear bond strength values were higher after 72-h than after 24-h ($p<0.01$). Most failures in specimens in which adhesive were applied to BA (VF-2, VF-3, UF-2, UF-3) were cohesive, whereas all failures in groups with no adhesive application (VF-1, UF-1) were adhesive (Table 2).

**DISCUSSION**

Calcium silicate-based materials have been used successfully as pulp capping agents in vital pulp therapy. Following such pulp capping, direct resin composites can be used for final restoration. However, phosphoric acid etching of an MTA surface has been reported to reduce microhardness, with the selective loss of matrix from around the crystalline structures on the MTA surface. Investigators have recommended that the application of etch-and-rinse adhesive over MTA be postponed for 96-h and that low condensation forces be used for coronal restoration over MTA because of the material’s low initial compressive strength. The FCs can be used without compression forces, and the new FC (VF) does not require surface pretreatment before bonding because it acts as both an adhesive and a composite (Kerr, Technical Bulletin). In this study, the bonding effectiveness of VF to BA was tested and the effect of adhesive application on the bond strength of VF to BA was investigated. The shear bond strength of VF was increased when used with etch-and-rinse adhesive, whereas there was a decrease in bond strength while a self-etch adhesive was used prior to VF. On the other hand, the shear bond strength of VF (without an adhesive) to BA after 24-h was weaker than shear bond strength of UF/self-etch adhesive or UF/etch-and-rinse adhesive groups. However, the shear bond strength of VF (without an adhesive) was similar to UF/self-etch adhesive but weaker than UF/etch-and-rinse adhesive group. Therefore both null hypotheses were partly rejected, because bond strength data were changed in relation to the used adhesive techniques and time.

Historically, the material used during pulp capping procedures has been calcium hydroxide. Calcium hydroxide has been considered the gold standard for pulp capping; however, previous research has shown that it is not ideally suited for this procedure. The opponents of calcium hydroxide for direct pulp capping procedures cite 3 major causes of failure: 1. The porosity of the dentinal bridge that is produced, 2. Calcium hydroxide adhering poorly to dentin, 3. Inability to provide a long-term seal against microleakage. Another pulp capping material recently developed is MTA. Recent research has shown that, when MTA placed in direct contact with the human dental pulp cells, differentiated them into odontoblast-like cells. When compared with calcium hydroxide, MTA has demonstrated significantly higher frequency of dentin bridge formation, thicker and less porous dentin, and less pulp inflammation. The current data available on the use of MTA in vital pulp therapy indicate that it is the optimum material and better than the traditionally used material calcium hydroxide. It has a greater long-term sealing ability and stimulates a high quality and a great amount of reparative dentin. In clinical outcomes evaluation, it has demonstrated a high success rate. MTA is, thus, a good substitute for calcium hydroxide in vital pulp procedures. BA was the calcium silicate-based material used in this study. It is composed primarily of calcium silicate oxide and calcium silicate, and also contains hydroxyapatite, calcium phosphate silicate, calcite, and tantalum oxide (a radiopacifier). Because BA is more resistant than MTA to acid attack, the use of self-etch adhesives or etching procedures may have different effects in these two materials.

According to the manufacturers, the complete setting time of BA is between 4 to 72-h. More or less is depending on the ratio of water and powder. On the other hand, Grech et al., reported that setting time of BA is 1,260 min (21-h). In a pilot study, we placed the VF over the BA after 4, 24- and 72-h. Bonding failure occurred at the BA/FC interface before performing the shear test for 4 h setting time. Therefore, first group was not included in this study.

Self-etch adhesives have been classified based on their ability to penetrate smear layers and depth of demineralization as ultra-mild (pH>2.5), mild (pH ~2), moderately strong (pH 1–2), and strong (pH<1). The manufacturer of VF has declared that it has a pH of 1.9, and Clearfil SE Bond, a mild self-etch adhesive, has a reported pH of 2.0. Thus, VF can be expected to interact with the dental substrate in a manner similar to that of a mild self-etch adhesive. The results of this study showed that bond strength of VF-1 group (BA/VF; 3.86 MPa) was similar with UF-2 (BA/Clearfil SE Bond/UF; 3.90 MPa) group after 72-h. However, bond strength of the composite to BA was significantly lower in the VF-1 group (2.54 MPa) than in the UF-2 group (3.31 MPa) after 24-h. For all groups, bond strength values were higher after 72-h than after 24-h. The liquid of BA was composed of pure water. Hydration of BA resulted in the formation of calcium hydroxide. As stated above, this formation process lasts to 72-h. So, the initial moisture content is higher than final moisture content while setting of BA is proceeding. Thus, the internal moisture level of setting BA may lead to lower bond strengths. However, bond strength values were significantly higher for the VF-1 group than the control group (UF-1) for both time intervals.
Among the possible reasons for such a result, the GPDM monomer should be considered. The phosphate group of GPDM is responsible for acid etching. In VF and UF groups the basic determinant for shear bond strength of BA/FC can be explained with macro mechanical adhesion of both high viscosity FCs to the rough surface of BA. Moreover higher shear bond strength of VF group may be explained with contribution of GPDM monomer in VF. According to the manufacturer’s claim, the phosphate functional group creates a chemical bond with the calcium ions of the tooth (Kerr Technical Bulletin). Similarly, the phosphate functional monomer (GPDM) in VF may create chemical bond with the calcium ions of the settled BA. The chemical mechanism of probable bond between GPDM monomer and BA should be analyzed with further experiments.

Previous studies have evaluated the bond strengths of adhesives to MTA using various bonding systems. Although acid etching reduces the surface microhardness of MTA and weakens the structure of the material, the results of these studies have shown that superior MTA/composite bond strength can be achieved with etch-and-rinse adhesives in comparison with one-step self-etch systems. Phosphoric acid etching significantly enhances the surface energy of the substrate, thereby provides significantly more microretention and potentially increases the bonding effectiveness of resinous materials. In this study, BA/FC bond strength was lower in VF-1 specimens than in those treated with an etch-and-rinse adhesive and UF after 24- and 72-h. The mild nature of VF prevented it from etching the BA as aggressively as an etch-and-rinse phosphoric acid adhesive with a lower pH. This difference may explain the lower bond strength of VF to BA. Furthermore, the application of self-etch primers results in calcium deposition on the etched BA surface, and dissolved free calcium may interfere with the bonding of resin to this surface. On the other hand, the compressive strength of calcium silicate-based materials increases over time. In a study comparing short-term (24-h) and long-term (21-days) immersion of set MTA specimens in distilled water, Torabinejad et al. reported an increase in MTA/composite bond strength over time. These results may be explained by the low initial compressive strength of BA.

In previous studies, self-adhering composite showed significantly lower bond strength to enamel when compared with a conventional FC. Wajdowicz et al. evaluated the shear bond strength of two new self-adhering composites to enamel with and without the use of a etch-and-rinse adhesive. They reported that the use of an etch-and-rinse adhesive significantly increased bond strength. Ozel Bektas et al. also claimed that adhesive application improved the bond strength of VF to dentin. The results of the present study showed that the application of a multi-step etch-and-rinse adhesive ‘gold-standard’ over BA before the application of VF improved the bonding effectiveness of VF. Probably, the phosphoric acid step significantly enhances the surface energy of BA and thus provides significantly more micro-retention. However, the use of a self-etch adhesive over BA negatively affected the bond strength of VF to BA. The majority of mixed-type bonding failures were observed in the VF-2 group (BA/self-etch adhesive/VF). VF contains GPDM as a functional monomer and 2-hydroxyethyl methacrylate (HEMA) as a hydrophilic resin. The phosphate functional group creates a chemical bond with the calcium ions of the tooth (Kerr Technical Bulletin). In contrast, Clearfil SE Bond contains 10-methacryloxydecyl dihydrogen phosphate (10-MDP) as an active acidic monomer, HEMA as a hydrophilic resin, a dimethacrylate resin, a catalyst, and water to permit ionization of the acidic monomers. Particularly, the interaction of functional monomers in VF with Clearfil SE Bond may have negatively affected the bond strength of VF to BA. However, there is no available chemical analytic data to fully clear up the interaction potential between GPDM and 10-MDP.

After the shear bond strength test, the surface integrity of BA and whether occurrence of any adhesive remnant was evaluated under a stereomicroscope for determination of fracture types. Since there wasn’t a disruption in surface integrity of BA/VF group, the failure was identified as entirely adhesive. However, majority of cohesive failures in the BA/VF group may have been caused more superficial interaction with BA. Whereas an actively applied more viscous adhesive may have caused aggressive interactions on BA surface. Therefore, there may be more distinct material loss on BA surface after test.

VF exhibited significant hygroscopic expansion and shrinkage during water absorption and desorption. This may possibly affect their long-term performance. In this study, before testing, BA/FC specimens were stored for 24-h and 100% humidity. Long term storage of bonded specimens could have different effects on the results of this study. On the other hand, because of the heterogeneity in chemical composition of the adhesive solutions, these results may change with other adhesives. Thus, while interpreting the results of the study, the composition of the adhesives, particularly the monomers, solvents, and initiators should be considered.

**CONCLUSIONS**

Within the limitations of this in vitro study, Vertise flow may be used successfully with no other adhesive as an alternative to the use of a FC in combination with a self-etch adhesive after BA has set for 72-h. However, application of an etch-and-rinse adhesive may improve the adhesion of VF to BA. Further studies are needed to support these conclusions for clinical applications.
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