Shear Bond Strength of Composite to Dentine with Various Adhesive Systems

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ABSTRACT

Background
Different dental adhesive systems are used in modern restorative dentistry. Older adhesive systems such as etch and rinse systems of generation 4 have been extensively tested and proven reliable, newer adhesive systems such as self-etch systems of generation 7 and 8 have been developed with the aim of improving bonding performance, durability and to be less technique sensitive.

Aim
This study aimed to compare the shear bond strength of universal adhesive systems from generation 7 (Futurabond DC) and Generation 8 (Scotchbond Universal, Optibond Universal) with an etch-and-rinse system from generation 4 (Optibond FL).

Methods
Four groups consisting of six specimens were prepared. The dentine specimens were then treated with four adhesive systems 1. Optibond FL, 2. Optibond Universal, 3. Futurabond DC and 4. Scotchbond Universal. Composite pillars were placed on the specimens and a shear bond strength test was performed. The results were analyzed with SPSS using One-way Kruskal-Wallis test (p<0.05).

Results
The OptiBond FL adhesive system exhibited the highest bond strength values, while the lowest values were observed in the Futurabond DC group. The results indicated significant differences in bond strength when comparing OptiBond FL to Futurabond DC and Scotchbond Universal (p<0.05). However, no significant difference was found between OptiBond FL and OptiBond Universal (p>0.05).

Conclusions
The three component etch-and-rinse system showed the highest median value of the systems tested. OptiBond FL and OptiBond Universal showed similar bond strength values, with the latter requiring fewer steps in clinical practice. More studies are needed for long-term durability and reliability.
BACKGROUND

The development of dental bonding agents has revolutionized the way of restoring teeth. Previously, practitioners had to remove large quantity of tooth substance to get an adequate mechanical adhesion through techniques such as undercuts, grooves and sharp internal angles it is now instead possible to bind filling material with micro mechanical retention (Vaidyanathan and Vaidyanathan, 2008).

Already in 1949, the chemist Oskar Hagger also known as the “Father of Modern dental adhesives” developed Sevriton Cavity seal, a dental adhesive product that bond to the surface of the tooth on a molecular level (Söderholm, 2007). Later, in 1954, Michael Buonocore, a researcher at the Eastman Dental center in Rochester, New York proved in experiments that by etching the enamel with a strong inorganic acid the bond strength of resin-enamel increased. Buonocore even then tested phosphoric acid which now is the accepted acid to use when etching the tooth surface (Buonocore, 1955).

Two decades after Buonocore findings, Takao Fusayama 1979 came up with the Total-etch method where the dentine and enamel are etched simultaneously with acid which resulted in stronger retention than when just etching to enamel (Fusayama, 1997). In the early 1990s the etching of the dentine were globally accepted as the Total etch technique or the “Etch-and-rinse technique” since the acid usually gets rinsed away after etching (Shen et al., 2021). The adhesives that are used today follow an “Etch and rinse” or a “self-etch” approach (Van Meerbeek et al., 2011).

Adhesive systems are commonly divided into “generations” which describe the systems characteristics, when and in what order the different types of adhesives systems were developed (Sofan et al., 2017). The systems are further divided into categories known as: Etch-and-rinse and self-etch systems (Shen et al., 2021).

The fourth generation bonding system is considered as the “golden standard” in dentine bonding and the use of the “total-etch” technique is one of the main characteristics of the generation. The total-etch technique allows the enamel and dentine to be etched simultaneously (Kugel et al., 2000). The fourth generation bonding systems most often consist of a 3-step system with separate containers (etchant, primer and bonding) and are applied sequentially and has been proven to be very effective when used correctly (Sofan et al., 2017).
Due to the many containers and application steps the demand for simpler and less technique-sensitive bonding agents urging manufacturers into keep developing new more user friendly adhesives (Van Meerbeek et al., 2011).

Fifth-generation adhesives is an etch-and-rinse strategy where the primer and bonding agent are combined together in a 2-step method (Shen et al., 2021).

Sixth-generation adhesives include 2-step self-etch adhesives, these systems does not involve a separate etching step. Instead an acidic self-etch primer is used, which is not rinsed.

Seventh-generation adhesives combine etch, priming and bonding in one single step without water rinse.

Eight-generation adhesives are so called universal adhesives and can be applied with both Etch-and-rinse or Self-etch. This enables the dentist to decide technique according to personal choice and decide which adhesive protocol that is most suited for the cavity being prepared (Van Meerbeek et al., 2020).

The aim of this study was to test the bonding strength of new adhesive systems (generation 7 and 8) to dentine using the self-etch technique and compare them with the etch-and-rinse technique of a fourth generation bonding system.
MATERIALS & METHODS

Twelve anonymized human caries free teeth were gathered from dental clinics in Umeå. The root and the crown were separated from each other and then the crown of the teeth were split in a palatinal and buccal part using a low-speed diamond saw (Minitom, Struers Aps, Ballerud, Denmark). To obtain stability in the molds, both the palatinal and buccal sides of the prepared teeth were slightly polished using a grinding machine (Labopol-30, Struers LLC, Westlake, United States) with a rough grit (P=120). To prevent the teeth specimen from floating up when the epoxy resin was poured into the molds, the teeth were placed on double sided tape in the bottom of the molds with the palatinal or the buccal side facing down. The Specimens were embedded in molds with mixed EpoFix Resin and Epofix Hardener (Struers Aps, Ballerup, Denmark. Lot 2178-01) following the manufacturer’s instruction. Two parts of hardener and 15 parts of resin by volume were mixed for 2 minutes and left to hardened for 12 hours in room temperature. Using a grinding machine (Labopol-30, Struers LLC, Westlake, United States) the specimen were then polished with a rough grit to obtain parallelity of the specimen (P=120). Using a grinding machine with a horizontal rotating arm the specimen were fixed with an axial force of 10 N (Appendix 1). The specimens were polished with (P= 120) until approximately 4 mm of dentine were exposed to ensure sufficient bonding area for placing a composite button. The specimens were stored in 1% Chloramine-T (Sigma aldrich co, St Louis, United States) solution until ready for testing. Preparing the specimen for testing, the testing area was polished with (P= 500) for a fine surface, no more than four hours prior to the bonding procedure according to the ISO (International Organization for Standardization) 29022:2013.

Bonding procedure

The specimens were randomly selected into four groups consisting of 6 specimens for each adhesive system. The specimens were preconditioned according to the manufacturer’s instructions. The four adhesive systems 1. Optibond FL, Kerr corporation, Orange, California, USA, 2. Optibond Universal, Kerr corporation, Orange, California, USA, 3. Futurabond DC, VOCO, Cuxhaven, Germany, 4. Scotchbond Universal DCA, 3M, Neuss, Germany. The composition of the adhesive systems, see Table 1. The bonding procedure of the specimens was prepared according to manufacturer’s instructions (Table 2) and cured using a LED curing light with a wavelength of 1000 mW/cm² (VALO, Ultradent Products, Inc. South Jordan, United States).
After the adhesive has been light cured, the specimen was inserted into a bonding clamp (Appendix 2) containing a white plastic button mold with a hole diameter of (2.38 ± 0.03) mm that were centered over suitable dentine bonding area and mounted according to ISO 29022:2013.

Composite (Ceram.X Spectra st. Lot 2204000637) pillars were applied carefully into the mold and ensuring good contact with the bonding surface using a small flat end packing instrument (LM-Dental, Pargas, Finland). The composite was cured according to the manufacturer’s instructions using a LED curing light with a wavelength of 1000 mW/cm² (VALO, Ultradent Products, Inc. South Jordan, United States).

**Notched-edge shear bond strength test**
The specimens were placed in a test base clamp and underwent a shear bond strength using a Ultratester Bond Strength Testing Machine (Ultradent Products, Inc., South Jordan, UT, USA) according to the ISO standard 29022:2013. The composite pillars underwent a crosshead load speed of 1.0± 0.1 mm/min until failure. The values were noted in MPa.

**Statistical analysis**
Using SPSS, a statistical analysis was made to compare if there was a significant difference in shear bond strength between our reference material (Optibond FL) compared to the other materials used in the study. First a descriptive statistical data analyze was performed to see if the values were normally distributed, the test showed that two of the materials had a skewness less than -1 or greater than +1 indicating that the results were not normally distributed. Therefore, one-way Kruskal-Wallis test was performed. The statistical significance was set to p<0.05.

**Ethical reflection**
The aim of the study was to test the bonding strength of new adhesive systems (Generation 7 and 8) to dentine and compare them with more studied and generally accepted adhesive systems (Generation 4). To examine if the new systems with fewer steps and with less technical sensitivity can perform as well or better than the three component etch-and-rinse systems regarded as the “Golden Standard”. To reduce the risk of incorrect data, the test will follow ISO Standard 29022:2013 carefully.
**Literature**

The referred literature in the study was found by searching in the electronic database PubMed and from scientific literature. The terms that were used on PubMed was: “Dental bonding systems”, “Adhesive systems dental bonding”, “Dental bonding generation”, “shear bond strength of dental adhesives”, “Dentin bonding agents”, “OptiBond FL”, “Universal adhesives”, “Scotchbond Universal”, “Futurabond DC”, “Optibond Universal”. In addition, the tutor provided articles, and the reference lists of articles found on PubMed were examined to obtain further literature. The literature search was conducted from September 2022 to February 2023.

**RESULTS**

The highest value of bond strength was observed in OptiBond FL with a median of 22.85 MPa. The group with the lowest value was observed in Futurabond DC with a median of 15.85 MPa (Fig.1). The result of the one-way Kruskal-Wallis test showed that there was a significant difference between Optibond FL compared to both Futurabond DC and Scotchbond Universal ($p<0.05$). No significant difference was found between Optibond FL and Optibond Universal ($p>0.05$) (Fig.2).

**DISCUSSION**

It has been suggested that a minimum bond strength of 17-20 Mpa is necessary to withstand the shrinkage forces of resin composite materials on enamel and dentine (Hedge et al., 2008). In the present study, all adhesive systems used met the necessary bond strength requirements for dentine, except for FuturaBond DC, which exhibited a slightly lower value. However, the results showed that the total etch system, OptiBond FL, had better bond strength compared to the self-etching adhesives. This result aligns with the research of Bouillaguet et al. (2001) who reported that OptiBond FL a conventional three-step adhesive system (generation 4) showed higher values of bond strength to dentine compared to self-etching one-step adhesive systems (generation 7). On the other hand, Kiremitci et al. (2004) reported that self-etching adhesive systems exhibited greater bond strength compared to traditional total-etch systems, with the all-in-one system yielding the highest bond strength among them. A more recent study demonstrates similar or even greater bonding strength of an universal bonding system compared to OptiBond FL, both when using self-etch mode and etch-and-rinse mode (Jang et al., 2016).

It has been suggested that removal of debris found on the surface of the dentine also known as “smear layer”, should be removed before the application of the bonding materials (Saikaew
et al., 2022). Self-etch adhesives is not as efficient in this process of removing smear layer and opening of dentinal tubules compared to three-step total etch bonding agents that results in complete removal of smear layer (Vashisth et al., 2012; Saikaew et al., 2022). Using 37% phosphoric acid has shown to remove smear layer effectively and result in deep intertubuli demineralization (Perdigão et al., 1996).

However, the total etch three step is more technique sensitive. The operator needs to take into consideration that the dentine must not be over etched due to the risk of the collapse of dentine collagen fibres which hinders the resin from fully penetrating the dentine tubules weakening the bond strength (Nör et al., 1996).

A study from 2011 that assess the impact of clinical expertise on dentine bonding strength when using self-etch and total-etch showed that the dentine bond strength of self-etch adhesive was comparable between a student group and the expert group, while the bond strength that was achieved with the etch-and-rinse adhesive was lower in the student group than in the expert group (Giachetti et al., 2011).

Another advantage with self-etch systems is that they do not include the “rinse step” and therefore prevents the risk of the dentine being over or under dried that leads to impaired bonding strength (Sarr et al., 2010).

In our present study, OptiBond FL showed higher median values than all of the other materials tested, there was only a significant difference between shear bond strength of OptiBond FL compared to Scotchbond Universal and Futurabond DC. The results did not show any significant difference between OptiBond FL and Optibond Universal.

Futurabond DC obtained the lowest mean result among all tested bonding systems. However, there are other studies that have shown higher results than what our study demonstrated, mean values of 22.524 MPa (El Sayed et al., 2015). It is not evident why Futurabond DC exhibits such low results even though the manufacturer’s instructions were followed.

The specimens underwent a preparation process consisting of several steps in accordance with ISO 29022:2013, as detailed in the "Methods" section. The process included pretreatment, conditioning, and composite material application. To ensure consistency, the same investigator carried out each step for every specimen group, in order to minimize any differences in technique that could potentially affect the results. One specimen was used as a test for each group to allow the investigators to adjust to working with the new adhesive.
Nevertheless, factors such as uneven adhesive application or inconsistent pressure during composite material condensation are examples of human-related variables that are difficult to control. These factors could have influenced the shear bond strength measurements.

Due to the limited availability of teeth, only a few specimens were prepared for each tested material. With a larger sample size, the result may have been different and could have altered the outcome.

CONCLUSION

The three component etch-and-rinse system showed the highest median value of the systems that were tested in the study. Previous studies and our test results show a significant difference between our reference material and two of the other materials in this study. Based on the current studies findings, both OptiBond FL and OptiBond Universal demonstrated comparable bond strength values. Furthermore, the advantage of OptiBond Universal requiring fewer steps makes it a potentially favorable option in clinical practice. However, additional studies are necessary to confirm these findings and ensure the long-term durability and reliability of using OptiBond Universal over OptiBond FL. Overall, the choice of adhesive system should be made on a case-by-case basis, considering the specific clinical situation and preferences of the dental practitioner.

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REFERENCES


**Table 1. A table showing the composition of the Adhesive systems used in the study.**

<table>
<thead>
<tr>
<th>Adhesive system</th>
<th>Composition</th>
</tr>
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</table>
| Optibond FL, Kerr corporation, Orange, California, USA | Primer: hydroxyl-ethyl methacrylate (HEMA), glycerol phosphate dimethacrylate (GPDM), mono-2-methacryloyloxyethyl phthalate, water, ethanol, photoinitiator  
Adhesive: bisphenol A diglycidyl methacrylate (Bis-GMA), HEMA,GPDM, barium-aluminum borosilicate glass, disodium hexafluorosilicate, silica |
| Optibond Universal, Kerr corporation, Orange, California, USA | GPDM, GDM, HEMA, dimethacrylate, acetone, ethanol |
| Futurabond DC, VOCO, Cuxhaven, Germany | Organic acids, BIS-GMA, HEMA, TMPTMA, BHT, ethanol, fluorides, CQ, amine, catalysts |
| Scotchbond Universal DCA, 3M, Neuss, Germany | MDP phosphate monomer, dimethacrylate resins, Bis-GMA, HEMA, methacrylate-modified polyalkenoic acid copolymer, camphorquinone, filler, ethanol, water, initiators, silane |
**Table 2.** A table showing the application steps of each adhesive system.

<table>
<thead>
<tr>
<th>Adhesive (Batch number)</th>
<th>Application steps</th>
</tr>
</thead>
</table>
| Optibond FL, Kerr corporation, Orange, California, USA (Primer: 8450239) (Bonding: 8494940) | 1. Apply Kerr Gel Etchant with 37.5% phosphoric acid for 15 sec.  
2. Rinse with water 15 sec.  
3. Gentle air dry for a few seconds.  
5. Using the same microbrush apply OptiBond FL Adhesive for 15 sec with a light scrubbing motion. Air thin the adhesive for 3 sec.  
6. Light cure for 20 sec at 1000 mW/cm².  
7. Apply composite according to manufacturer’s instructions. |
| Optibond Universal, Kerr corporation, Orange, California, USA (8706849) | 1. Shake bottle 10 sec.  
2. Apply adhesive using a disposable microbrush, rub in for 20 sec.  
3. Dry adhesive with gentle air first and then medium air for at least 5 seconds.  
4. Light cure for 10 sec at 1000 mW/cm²  
5. Apply composite according to manufacturer’s instructions. |
| Futurabond DC, VOCO, Cuxhaven, Germany (2203223) | 1. Apply adhesive in a layer of medium thickness using a disposable microbrush, rub in for 20 sec.  
2. Dry adhesive with an air syringe.  
3. Light cure for 10 sec at 1000 mW/cm²  
4. Apply composite according to manufacturer’s instructions. |
| Scotchbond Universal DCA, 3M, Neuss, Germany (8886482) | 1. Apply adhesive using a disposable microbrush, rub it in for 20 sec.  
2. Dry adhesive with a gentle stream of air for 5 sec.  
3. Light cure for 10 sec at 1000 mW/cm²  
4. Apply composite according to manufacturer’s instructions. |
**Fig 1.** A Box-plot presenting the data. The y-axis showing the bond strength in MPa. The x-axis showing the different adhesive systems used in the study.

<table>
<thead>
<tr>
<th>Reference adhesive</th>
<th>Compared to</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>OptiBond FL</td>
<td>OptiBond Universal</td>
<td>0.525</td>
</tr>
<tr>
<td></td>
<td>Futurabond DC</td>
<td>&lt;.001</td>
</tr>
<tr>
<td></td>
<td>Scotchbond Universal</td>
<td>0.047</td>
</tr>
</tbody>
</table>

**Fig 2.** A table showing the p-value comparing OptiBond FL to all the other adhesives tested in the study. The result showed that there was a significant difference between Optibond FL compared to both Futurabond DC and Scotchbond Universal (p<0.05). No significant difference was found between Optibond FL and Optibond Universal (p>0.05).
APPENDIX

1. The grinding process preparing parallelity of the specimens with the Labopol-30 grinding machine.

2. The specimen placed in the bonding clamp with the white plastic button mold.
3. The specimen positioned in a test base clamp. The clamp is fixed in the Ultratester bond strength testing machine.