Abstract

Aim: This study was conducted to evaluate the effect of mechanical alteration of enamel surface on shear bond strength in different bonding techniques.

Materials and Methods: Seventy samples were fabricated and randomly divided into three groups: Group A (n = 30) – prepared enamel surfaces, Group B (n = 30) – unprepared enamel surfaces, and Group C (n = 10) – prepared enamel surfaces + etch and rinse which served as a control group. Group A and Group B were further divided into three subgroups (n = 10), sub-Group A1, B1 (nanohybrid composite + self-etch), sub-Group A2, B2 (self-adhering composite), and A3, B3 (self-adhering composite + self-etch). Teflon ring molds were used to make composite resin cylinders with the specific bonding protocol of each group. Shear bond strength testing was conducted, and data were analyzed.

Results: Mean shear bond strength values were as follows: C > A1 > A3 > A2 > B1 > B3 > B2.

Conclusions: Prepared enamel surfaces resulted in higher shear bond strength values as compared to unprepared enamel surfaces. Prior application of self-etch agent resulted in higher shear bond strength values of self-adhering composite in prepared and unprepared enamel surfaces.

Keywords: Prepared enamel surfaces; self-adhering composite; shear bond strength; unprepared enamel surfaces

INTRODUCTION

Resin-based composites gain their retention most importantly through micromechanical adhesion to the tooth structure. Since its introduction, the etch and rinse approach has been the most effective and reliable technique to achieve effective bonding of resin composite to enamel. However, it involves a two-step or three-step approach with additional rinsing and drying of tooth, which makes it cumbersome and technique sensitive.

To simplify this procedure, a nonrinse approach was introduced in the form of self-etch adhesives. Self-etch systems combine all the steps of etching, priming, and bonding into one step by incorporating both hydrophilic and hydrophobic acidic functional monomers, water, and organic solvents in a single bottle solution, thus making it more user friendly and less technique sensitive.

Recently, self-adhering flowable composite (SAC) has been introduced, which is claimed to be less technique sensitive with reduced chairside time and postoperative sensitivity. The adhesion of self-adhering composite to enamel and dentin has been attributed to the presence of glycerol phosphate dimethacrylate (GPDM) monomer, which acts as an etchant as well as contributes to the chemical bond which forms between hydroxyapatite of tooth and the phosphate group of GPDM. Resin tags formation, facilitated by hydroxyethyl methacrylate (HEMA) additionally contributes to the bond strength. However, various authors have reported relatively low bond strength values of SAC to dental hard tissues, thus necessitating an alteration in the bonding approach.

Restorative demands frequently present with clinical situations...
requiring bonding to intact, altered, or prepared enamel surfaces, which might bond differently with adhesive resins. Considering the limited information on bonding of SAC to enamel available in literature, this study was undertaken to evaluate the effect of mechanical alteration of enamel surface on shear bond strength of self-adhering composites utilizing different adhesive modalities.

**MATERIALS AND METHODS**

Freshly extracted human permanent maxillary central incisors with intact labial surfaces were obtained. Teeth were decoronated, and crowns were partially embedded in self-curing acrylic resin using plastic ring molds (2 cm × 3 cm) while ensuring the horizontal orientation of the exposed enamel labial surface.

Seventy such samples were fabricated and randomly divided into three groups:
- Group A: prepared enamel surfaces (n = 30)
- Group B: unprepared enamel surfaces (n = 30)
- Group C: control group (prepared enamel surfaces, nanohybrid composite + etch and rinse) (n = 10).

Surface preparation of the samples was done with the help of a coarse abrasive finishing disk (Shofu-black) of particle size 100 µm. Ten unidirectional strokes were made to remove 0.5 mm of surface enamel.

Samples in Group A and Group B were further distributed randomly into three subgroups based on the adhesive technique and resin composite used.
- a. Sub-Group A1 (n = 10) and Sub-Group B1 (n = 10) (nanohybrid resin composite + self-etch)
- b. Sub-Group A2 (n = 10) and Sub-Group B2 (n = 10) (self-adhering resin composite)
- c. Sub-Group A3 (n = 10) and Sub Group B3 (n = 10) (self-adhering resin composite + self-etch).

**Protocol for application of bonding agent**

Samples of all groups were subjected to their specific bonding protocols (according to manufacturer’s instructions) to form resin composite cylinder which includes:
- a. Application of etch-and-rinse system (Scotchbond multipurpose, 3M ESPE)
  - The enamel surfaces of the samples in Group C were etched using Scotchbond multipurpose etchant for 15 s followed by bonding agent application (Adper Single Bond), which was cured for 20 s.
- b. Application of self-etch system (Adper Easy Bond, 3M ESPE)
  - Self-etch system was used in four Subgroups (A1, A3 and B1, B3) for bonding

resin composite cylinders. Bonding agent was applied in gentle agitation using a fully saturated applicator for 20 s.

- c. No bonding agent was used for samples of Sub Groups B2 and A2.

**Protocol for fabrication of resin composite cylinders**

Teflon ring molds of diameter 3.2 mm and height 5 mm were used for making composite resin cylinders. The ring molds were stabilized in the center of the enamel labial surface with the help of sticky wax.

Resin composite cylinder was fabricated using nanohybrid resin composite (Filtek Z 250 XT, 3M ESPE) in groups A1, B1, and C and self-adhering composite (Dyad Flow, Kerr) and in groups A2, A3, B2, and B3.

The Teflon ring molds were filled incrementally with composite resin with each single increment of 2 mm with the help of plastic filling instrument (Hu-Friedy). Curing of each increment was done for 20 s. The increments were added and cured until the ring mold was completely filled.

**RESULTS**

The highest mean shear bond strength value was observed in control group C (28.66 MPa), whereas the lowest mean shear bond strength value was observed in Sub-Group B2 (7.3 MPa) [Table 1]. Prepared enamel surfaces resulted in significantly higher shear bond strength values for all adhesive strategies as compared to unprepared enamel surfaces (P < 0.01). Mean shear bond strength values can be arranged in the following order: C > A1 > A3 > A2 > B1 > B3 > B2. The difference between all subgroups was statistically significant (P < 0.01) except A1 and A3 (P > 0.01) [Table 2].

**DISCUSSION**

While using adhesive restorations, an operator may encounter a number of clinical situations which may (cavity preparation, veneer preparations, etc.) or may not (pit and fissure, luting of orthodontic brackets, etc.) require the surface preparation of enamel.[8] Hence, this aspect was investigated in this study with respect to different adhesive strategies, and this may have a direct influence on bond strength of adhesive restorations.

In the present study, enamel surface preparation was done to homogeneously remove the prismless enamel and achieve a uniform and controlled surface preparation. Thick prismless enamel layer may compromise with resin tag formation, leading to short and poorly defined tags due to inhibition of permeation of self-etch primer and
bonding agents in the enamel layer. The presence of partial unetched areas further compounds the problem.\[9\]

In the present study, prepared enamel surfaces showed a higher shear bond strength as compared to unprepared enamel surfaces in all the groups. The mean shear bond strength value of nanohybrid composite (17.18 MPa) for prepared enamel surface was significantly higher than that of unprepared enamel surface (14.14 MPa).

Various other studies in the literature also corroborate with the findings of the current study. Yazici et al.\[10\] reported a mean shear bond strength value of 22.7 MPa for nanohybrid composite on prepared enamel surface when used with a self-etch approach as compared to unprepared enamel surface (20 MPa). Senawongse et al.\[8\] reported significantly higher microshear bond strength of ground enamel as compared to intact enamel surface in an etch-and-rinse adhesive system, a two-step and a one-step self-etching system.

A number of reasons may contribute to higher shear bond strength values in prepared enamel. Unground enamel has a thick prismless layer, which negatively influences the etching morphology. The increased inorganic content in the highly mineralized aprismatic layer makes it less conductive to bonding. Furthermore, the outermost enamel layer has high fluoride content due to it being subjected to many changes in the oral environment after tooth eruption. The ions present in saliva may saturate calcium phosphate and fluoride ions on enamel surface and may convert into fluorapatite, thus making it resistant to etching.\[11\]

In the present study, the highest bond strength among all the groups was exhibited by the control group. This may be directly attributed to the fact that phosphoric acid etching is to date the most reliable and clinical method to enhance the bond strength and ensure the clinical durability of composite resin. The mechanism involves the acid dissolution of enamel, creating microporosities on enamel surface, which exposes hydroxyapatite crystals. The resin monomers, which are subsequently applied, envelope the exposed crystals and infiltrate into microporosities, which on curing creates a strong micromechanical interaction and an intimate bond between the enamel and resin.\[9\]

In the present study, the SAC recorded relatively lower mean shear bond strength values (7.3 MPa) as compared to other adhesive protocols. Similar results were observed by Bektas et al.\[4\] and Paciﬁci et al.\[5\] In these studies, the mean shear bond strength of Dyad Flow to enamel was recorded to be \(<7\) MPa. On the contrary, Kurtzman and Afrashtehfar\[12\] reported shear bond strength of 17.8–22.4 MPa for Dyad Flow on enamel. Shear bond strength of self-adhering composite to dentin has also been evaluated by Almaz et al.\[13\] who recorded a mean of 7.27 MPa; however, Kurtzman and Afrashtehfar\[12\] reported it to be in the range of 17.8–22.4 MPa. The difference in the mean values could be attributed to the difference in methodology. Studies in literature also suggest better shear bond strength of Dyad Flow to dentin without separate etching or bonding procedure.

Although self-adhering composite contains HEMA to increase their wettability, they do not contain any solvent, which leads to decrease in their penetration capacity.

### Table 1: Mean shear bond strength values (in MPa) of all the groups

| Sample number | Control (C) | Prepared | Unprepared |
|---------------|-------------|----------|------------|
|               | Subgroup (A1) | Subgroup (A2) | Subgroup (A3) | Subgroup (B1) | Subgroup (B2) | Subgroup (B3) |
| Mean          | 28.66       | 17.18    | 15.31      | 17.003       | 14.14        | 7.3          | 13.03        |
| SD            | 1.105       | 0.790    | 1.199      | 0.634        | 1.331        | 1.276        | 0.854        |
| Maximum       | 30.3        | 18.7     | 17         | 18           | 15.7         | 9.8          | 14.4         |
| Minimum       | 26.8        | 16.1     | 13.6       | 16.2         | 12.1         | 5.4          | 12           |
| Median        | 28.75       | 17.25    | 15.3       | 16.85        | 14.25        | 6.85         | 12.9         |

**SD:** Standard deviation

### Table 2: One-way ANOVA F-test of significance among the mean shear bond strength values of different subgroups of prepared and unprepared group

| Source of variation | Source of variation | Prepared: A1,A2,A3 | Unprepared: B1,B2,B3 |
|---------------------|--------------------|--------------------|----------------------|
|                     | SS                 | df                 | MS                   | F        | P               | F critical |
| Between subgroups   | 21.315             | 2                  | 10.66                | 12.98    | 0.0001          | 5.488     |
| Within subgroups    | 22.162             | 27                 | 0.82                 | <0.01 (significant) |
| Total               | 43.477             | 29                 |                      |          |                 |           |

**SS:** Sum of square, **MS:** Mean of square

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Although self-adhering composite contains HEMA to increase their wettability, they do not contain any solvent, which leads to decrease in their penetration capacity.
into the enamel surface and consequently a decrease in their shear bond strengths.\textsuperscript{[14]}
Furthermore, higher filler content (70% by weight) and relatively higher molecular weight of GPDM molecule (356 g/mol) in Dyad Flow increase its viscosity and therefore might lead to poor penetration capacity and also affect its wettability.\textsuperscript{[15]}

In the present study, bond strength of self-adhering composite when combined with self-etch adhesive increased on both prepared and unprepared enamels. This can be attributed to the mild acidic nature of self-etch adhesive, which has a pH of 2.3. These adhesives demineralize enamel only to a superficial depth while leaving sufficient hydroxyapatite available for bonding with monomer of self-adhering composite. They also contain ethanol as solvent, which is essential to increase the wettability of the adhesive material, enhance the penetration of the functional monomers, and permit closer adhesive-substrate chemical interaction with hydroxyapatite, leading to the increase of the bond strength.\textsuperscript{[16]} The presence of free HEMA, a low-molecular weight water-soluble methacrylate monomer in combination with ethanol as solvent, enhances the wetting, penetration, and bonding of GPDM molecule present in Dyad Flow.\textsuperscript{[17]} In addition, HEMA, being common monomer in both the agents, enhances the miscibility of hydrophilic and hydrophobic components of the adhesive and prevents phase separation reaction.\textsuperscript{[15]}

CONCLUSIONS

Since prepared enamel surfaces resulted in higher shear bond strength values for all adhesive strategies as compared to unprepared enamel surfaces, hence, it is advisable to roughen the enamel surface before carrying out any adhesive protocol. Etch and rinse method resulted in the highest shear bond strength values as compared to other adhesive protocols. In situations where self-adhesive composite has to be used, prior application of self-etch adhesive would increase the shear bond strength in both prepared and unprepared enamel surfaces.

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Conflicts of interest
There are no conflicts of interest.

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