Shear bond strength of fluorinated grapheme nanoparticles modified dental adhesives

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Abstract
The success of adhesion to the tooth structure plays an important role in the dental restoration and the loading of adhesive compositions with fillers and nanoparticles has a significant reinforcement effect of the adhesive, thus improving physicomechanical properties of the material, and, consequently, the bond strength between the restoratives and dental substrates. The current study was designed to investigate the effects of the addition of 2% of fluorinated graphene nanoparticles (FGN) on the shear bond strength for two types of commercially available universal adhesive using etch and rinse technique as a bonding procedure. Forty extracted premolar teeth were included and divided into four groups of ten teeth. For group I, we used Prime & Bond Universal adhesive alone. For group II, we used Prime & Bond Universal adhesive +2% FGN nanoparticles. For group III, we used All Bond Universal adhesive alone. For group IV, we used All Bond Universal adhesive+2% FGN. After 24 hours, shear bond strength was tested utilizing the Laryee universal testing machine. A higher shear bond strength was observed with adhesives containing 2% FGN in comparison to study groups (P<.01). In conclusion, according to the results the addition of 2% of fluorinated graphene nanoparticles to the etch-and-rinse adhesive systems affect the shear bond strength and seem to be a good option for reinforcing adhesive systems.

Keywords Adhesive systems, shear bond, universal adhesive, Etch and Rinse adhesive technique

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Introduction
In order to further improve the bond strength and reduce the nanoleakage, inorganic nanofillers were introduced into the dental adhesive. The nanofillers diffused into the collagen fibrils in the dentin could strengthen the hybrid layer and then increase the bond strength (1). Currently, there are some chemicals/agents or bonding protocols available that increase or stabilize bond strength between resin-based materials and dental substrates (2). The loading of adhesive compositions with fillers and nanoparticles has led to a significant reinforcement effect of the adhesive. Nanoparticles have been used to reinforce the organic matrix of resin adhesives, thus improving the physicomechanical properties of the material, and, consequently, the bond strength between the restoratives and dental substrates (3). Once the hybrid layer is strong, the water uptake is reduced, hydrolysis is diminished, and proteases activity is retarded, thereby reducing the rate of bond degradation over time (4,5).

Graphene, a two-dimensional (2D) material consisting of carbon atoms which was arranged in honeycomb lattice, has been studied extensively to enhance performances of other materials due to their unique properties, such as excellent mechanical properties (breaking strength of 42 N/m and Young’s modulus of 1.0 TPa), extreme chemical stability and the mechanical strength enhancement effect of graphene and graphene family materials could also be explained by the crack branching mechanism which has been investigated in previous study (6,7). So we think it may also be suitable for FG (8). Fluorinated graphene (FG), an up-rising member in the family of graphene derivatives, is a kind of one-molecule-thick material (9). So it could speculate that as a member of the graphene family, FG may also have a similar effect. Based on these studies, it is reasonable to assume that FG may be beneficial for reinforcing adhesives and improving its performance as dental materials (10,11).
Materials and Methods

Preparation of modified universal adhesives

Two commercial universal adhesive agents; All Bond Universal adhesive agent and Prime&Bond Universal adhesive (Dentsply, Tusla dental specialties, USA) and All-Bond Universal adhesive (BiscoInc, USA) were selected in this study. The fluorinated graphene nanoparticles (FGN) (Hyper Chem Co., chain, Let. No. Q19011701) was used as filler particles and it was submitted to the salinization process to improve the adhesion interface between the filler nanoparticles and the adhesive matrix so that the FGN was treated with 3-metacryloxypropyltrimethoxy (MPS) saline coupling agent using a procedure described by Deb et al. in 1996. A percentage of 2 w% of FGN was chosen from a pilot study out of 4 different percentages (1, 2, 3, 4 w.%) because this construction didn’t adversely affect the viscosity, color stability and flow ability of the adhesives. Then the FGN was dispersed in 6 ml of each adhesive by ultra-sonication for 1 h to obtain a homogenous mixture.

Sample preparation

The teeth were mounted in the acrylic block with dimensions of 1.5 cm depth, length and width to the level of the cement enamel junction with the entire crown of the tooth should be exposed. A flat surface for bonding procedure was obtained by cutting through the occlusal surface of the crown about (1 mm) below the dentin-enamel junction so that removing the enamel to expose the normal dentin using low speed cutting with finer metal abrasive disks., after that, each dentin surface will be polished with 600-grit silicon carbide paper under running water for 60 seconds to create a standardized smear layer. Finally, a flat and polished dentin surface that is free from enamel should be obtained.

Surface bonding procedure

As a general procedure for all groups, the dentin surface of the sample teeth was etched with 37% phosphoric acid gel for 15 seconds, and rinsed with distilled water for 15 seconds to make sure complete washing of the etchant gel. The adhesive bonding was applied and gently rubbing with micro brush, and the solvent was evaporated with a gentle stream of air from 1 cm for the dentin substrate for 10 seconds and light-cured with continuous output using a conventional intensity light, with curing distance of 5 mm and lasting for 10 s using LED wireless light cure device (SDI, Australia). For each group, a cylindrical mold (inner diameter = 4 mm, thickness = 2 mm) was fabricated specifically (custom made) for this study and was used as a composite mold and placed on the adhesive-treated dentin surface. The mold was filled with a composite (Filtek Supreme Ultra Universal, 3M ESPE,USA) which was light-cured for 60 seconds. The bonded specimens were stored in distilled water at 37°C for 24 h and then tested for bond strength.

Shear bond strength test

Debonding for restoration for the tested groups was tested utilizing Laryee universal testing machine (Tinius Olsen, Germany) at the University of Technology / Department of material engineering. A notched type rod that called the (Notched-Shear Bond Strength Test, ISO 29022) was mounted on the Universal Testing Machine Load was applied at a cross-head speed of 0.5 mm/min until the bond failed. Dentin shear bond strength was calculated using this formula:

\[
\text{Shear bond strength} = \frac{N}{\pi d^2}
\]

Where N is the load at failure, and d is the diameter of the bonded area.

Statistical Analysis

Statistical analysis was done by Statistical Package for Social Sciences (SPSS) version 24. Results were calculated as mean ± standard deviation of means (SD). A comparison between the two groups was done by using the student-t-test and the statistically significant differences were considered when P<0.05.

The Results

Shear bond strength (SBS)

The results were obtained by testing ten samples of each group studied. The descriptive statistics which represent the mean and SEM of the shear bond strength in (MPa) are summarized in table (1) and illustrated in figure (1). The maximum and minimum values of mean SBS were observed in GroupII (the incorporated Prime&Bond Universal bonding agent with 2% fluorinated graphene) and GroupIII (the non-incorporated All Bond Universal bonding agent) groups respectively (Table 1). T-test was performed to identify the presence of statistically significant differences and showed a highly significant difference in SBS mean group II (the incorporated All Bond universal + 2% FGN).

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fluorinated graphene) in comparison with group I (all bond universal bonding agent alone) (p<.01). Furthermore, it was shown that there is a highly significant difference in SBS mean group IV (the incorporated Prim& Bond universal + 2% fluorinated graphene) in comparison with group III (Prim& Bond universal bonding agent alone) (p<.01).

Table 1: Descriptive and inertial statistics of the groups

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>Shear bond strength (MPa) Mean ± SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group I: the non-incorporated (Prime &amp; Bond Universal) bonding agent (Control group).</td>
<td>10</td>
<td>189.5 ±1.372a</td>
</tr>
<tr>
<td>Group II: the incorporated (Prime &amp; Bond Universal) bonding agent with 2% fluorinated graphene</td>
<td>10</td>
<td>227.5 ±3.154 b</td>
</tr>
<tr>
<td>Group III: the non-incorporated (All Bond Universal) bonding agent (Control group).</td>
<td>10</td>
<td>186.5 ±2.812b</td>
</tr>
<tr>
<td>Group IV: the incorporated (All Bond Universal) bonding agent with 2% fluorinated graphene</td>
<td>10</td>
<td>225.5 ±2.626 b</td>
</tr>
</tbody>
</table>

Figure1. Bar chart showing the mean of shear bond strength in the four experimental adhesive groups.

Discussion
Durable and high bond strength to dentin is the goal of all the restorative materials and procedures. Clinical failure of adhesive restorations happens more often due to insufficient sealing of cavity walls, with subsequent discoloration, rather than complete loss of retention (22; 23). Bond strength was tested by the shear bond test because it is a simple and reliable method(24). The graphene is biocompatible and non-cytotoxic and this makes it an ideal material to incorporate into dental polymers to increase their strength and durability. It is well known that graphene has high mechanical strength and has been shown to enhance the mechanical, physical and chemical properties of biomaterials (25).
The previous study showed that 1 wt. % graphenenanosheets (GNS)/HAp composites exhibited a 30% increase in VHN compared to pure HAp\(^{26}\). The compressive strength and fracture toughness of 0.5 wt. % graphene/nano-58S bioactive glass composite has significant improvements by 105% and 38% \(^{27, 28}\). The modification of All Bond Universal adhesive with 2w% FGN was increasing the shear bond strength from 189.5 to 227.5 also the addition of FGN 2w% to the Prime& Bond Universal adhesive was increase significantly the shear bond strength from 186.5 to 225.5.

The enhancement of mechanical properties may be caused by the following reasons. First, the addition of FGN to the adhesive system leads to an increase in the filler percentage of the adhesive system which performs lower shrinkage stress, and it might contribute to enhancing the bond strength and this is supported with previous research \(^{29}\). Second, the two-dimensional structure and high specific surface area of FGN enabled mechanical interlocking with matrix and the load was transferred from the matrix to the FG nanosheet when a crack is initiated because of the difference in elastic modulus. Third, the mechanical strength enhancement effect of graphene and graphene family materials could also be explained by the crack branching mechanism which has been investigated in the previous study \(^{30, 32}\) and by four different aspects of the mechanism including crack bridging, pull-out, crack deflection and crack tip shielding \(^{31, 33}\).

In conclusion, the results show that the addition of 2% fluorinated graphene nanoparticles was significantly improved the shear bond strength of both adhesives to dentine. Further studies are recommended to clarify other beneficial effects of FGN that may justify their uses as adhesive filler.

Reference


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