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Evaluation of Shear-Bond-Strength of Dental Self-Adhering Flowable Resin-Composite versus Total-Etch One to Enamel and Dentin Surfaces: An *In-Vitro* Study

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Abstract

AIM: This study aimed to assess the shear bond strength of a self-adhering flowable resin composite versus a total-etch one to different surfaces of permanent-molars.

MATERIAL AND METHODS: Thirty-six sound human permanent molars were used. The teeth were embedded in acrylic blocks, such that their buccal surfaces were shown. The teeth were divided into three groups: Group I: Uncut-Enamel, Group II: Cut-enamel-surfaces with minimal-grinding and Group III: dentin-surfaces. Half of the teeth in each group were used for bonding to a self-adhering flowable resin-composite (Dyad-flow, Kerr, USA). While the other half of each group was bonded to a total-etch flowable resin-composite (Filtek™Z350-XT,3M-ESPE, USA) which necessitate etching and bonding. Teflon-mold was used for constructing resin composite cylinders (3 × 3 mm) over the buccal surfaces. The Dyad-flow was applied in the central hole of the mould placed upon tooth-surface, and then light-cured for 20 seconds. The Filtek-Z350-XT was applied similarly after etching and bonding steps. The teeth were stored in 37°C distilled water for 24 hours. The strength was measured using a universal testing machine and statistically analysed. Modes of failure were studied using digital-microscope.

RESULTS: Mean values of shear bond strength for the Dyad and Filtek-Z350-XT in the uncut-enamel were 3.5 and 24.6MPa respectively, while that for cut-enamel were 4.5 and 12.7MPa respectively (Both highly statistically significant $P \leq 0.01$) and in dentin were 4.3 and 6.7MPa respectively (Statistically significant $P \leq 0.05$). The failure mode for Dyad was mainly adhesive (un-cut or cut-enamel 83.3% adhesive and 16.7% mixed, while in dentin 100% adhesive). While the modes of failure for Filtek-Z350-XT in enamel, either cut or un-cut, were 50% cohesive and 50% mixed, whereas in dentin 100% adhesive.

CONCLUSION: Bonding of self-etch ″Dyad-flow″ flowable resin-composite was lower than the total-etch one in enamel and dentin. Thus further material improvement may be required.

Introduction

Recently, flowable composite resins become very common in dental clinics [1]. The low viscosity of these materials allows their easier manipulation with more adaptation than conventional resin composites [2]. A new self-adhering flowable composite (Dyad™-flow, Kerr, USA) was recently introduced in the market [3]. Incorporation of an acidic adhesive-free composite may lead an interaction between the material and tooth structures, both chemically and micro mechanically [4].

The multi-step etch and rinse approach, contrary to the self-etch, involves a phosphoric acid-etch step which results in deep pits in enamel's hydroxyapatite. The resin tags incorporated within this acid-etched enamel lead to an optimal bond with the enamel surface effectively sealing the restorations' margins. The traditional etch and rinse adhesives are still regarded as 'gold-standard' [5], [6], [7], [8].

For a strong bond between the tooth structure and a restoration, primary chemical interaction between resin and tooth structure could be a tremendous aid. Functional monomers, in particular like 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), have been proven to interact with hydroxyapatite through primary ionic binding which results in chemical bond in addition to micro-mechanical interlocking [9], [10]. However, this chemical bonding should also be stable in an aqueous environment. Chemical bonding promoted by 10-MDP was proven to be more stable in water than that provided by other functional monomers like 4-META (4-methacryloyloxyethyl trimellitic acid) and phenyl- P (2-methacryloyloxyethyl phenyl phosphoric acid) [10]. The 10-MDP is present in some commercial bonding agents applied before resin-composite restorations.

On the other hand, the self-adhering flowable composite "Dyad-flow" contains an adhesive monomer called glycerol phosphate dimethacrylate "GPDM" having two functional groups; the first is an acidic phosphate for both tooth etching and chemical bonding with its calcium content, while the other is a methacrylate group for polymerisation [3]. Contrary, it was reported that GPDM "etches" instead of "bonds" to hydroxyapatite [10].

This study aimed to assess the shear bond strength of self-adhering flowable resin-composite "Dyad-flow" compared to the total-etch one (preceded by etching and bonding steps) to un-cut and cut enamel as well as dentin of permanent molars.

Methods

Specimens' preparation and grouping

Thirty-six permanent molars were embedded in acrylic blocks (19 mm diameter X 16 mm height), such that their buccal surfaces were shown and aligned with the acrylic surfaces (Cold cure acrylic resin, Acrostone, Egypt). The sample size was calculated using G-power with effect size 0.7, Power 80% and alpha error 5% for a final total sample size 36 molars. The teeth were randomly divided into three groups: Group I: Enamel surfaces without any intervention (Uncut Enamel), Figure 1a, Group II: Enamel surfaces with minimal grinding (Cut Enamel), Figure 1b and Group III: Dentin surfaces, Figure 1c. In groups II and III, the buccal surfaces of the teeth were ground using a grinding machine (Redwing, Handler, USA) under water coolant. Half of the teeth in each group were used for bonding to a self-adhering flowable resin-composite (Dyad™-flow, Kerr, USA). While the other half of each group was bonded to a total-etch flowable resin-composite (Filtek™Z350-XT, 3M-ESPE, USA) which necessitate etching and bonding.

Figure 1: A photograph showing the different teeth surfaces: A) Uncut Enamel); B) Cut Enamel; C) Dentin

Bonding Procedure

A specially designed holed-split Teflon mould was used for constructing composite-resin cylinders from the previous flowable composites (3 × 3 mm) over the buccal surfaces of the mounted teeth.

Self-adhering flowable resin-composite

The Dyad™-flow was applied in a central hole of the mould upon each tooth surface, and then light-cured for 20 seconds using a light-curing unit (Satelec, Acteon, France). The teeth with the bonded resin cylinders were stored in distilled water at 37°C for 24 hours.

Total-etch flowable composite

Before the application of Filtek™Z350-XT flowable composite, etching and bonding steps were performed. A phosphoric acid etching gel 37% (Eco-Etch, Ivoclar Vivadent, Liechtenstein) was applied for 15 seconds, then rinsed and air-dried. A bonding agent containing 10-MDP (Universal Single Bond, 3M ESPE, Germany) was applied for the teeth in a rubbing motion for 20 seconds. Then, a gentle air stream was applied over the bonding agent for 5 seconds, then light-cured for 10 seconds. Then, the flowable composite was applied through the mould and cured as previous. The teeth were stored in distilled water at 37°C for 24 hours.

Shear bond strength test

Each acrylic block was secured with tightening screws to the lower fixed compartment of a universal testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK), with a load cell of 5 kN. A shearing load with the compressive mode of force by mono-bevel-chisel was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The load required for de-bonding was recorded in Newton. The data were recorded automatically using computer software (Nexygen-MT Lloyd Instruments).

Shear bond strength was calculated as the load at failure divided by bonding area to express the bond strength in MPa: $\tau = P/\pi r^2$

Where; τ = bond strength (in MPa), P = load at failure (in N), π = 3.14, r = radius of cylinder (in mm). The strength was recorded blindly by a different assessor, and the data were statistically analysed. Modes of failure were studied using a digital microscope (Scope Capture Digital Microscope, Guangdong, China), and recorded as cohesive, adhesive or mixed failure by a different assessor blindly.

Statistical Analysis

Statistical analysis was performed for all data using the statistical package for social science IBM®, SPSS® statistics for windows computer software version 20 {IBM® (IBM corporation, NY, USA) and SPSS® (SPSS Inc., an IBM company, USA)}. Analysis of variance (ANOVA) was used for determining the statistical significance of the mean shear bond strength values between the groups. All graphs were made using Excel Microsoft windows in 2010. The *p*-values were considered statistically significant if less than or equal 0.05 and highly statistically significant if less than or equal 0.01, while not statistically significant if greater than 0.05.

Results

Mean values of shear bond strength for the Dyad and Filtek-Z350-XT in the uncut-enamel were 3.5 ± 1.6 and 24.6 ± 6.2MPa respectively, while that for cut-enamel were 4.5 ± 2.7 and 12.7 ± 4.5MPa respectively (Both highly statistically significant $P \leq 0.01$) and in dentin were 4.3 ± 1.6 and 6.7 ± 1.7MPa for Dyad and Filtek-Z350-XT respectively (Statistically significant $P \leq 0.05$), as shown in Table 1.

Table 1: Mean Shear Bond Strength Values for the different groups

	Uncut Enamel*Dyad	Uncut Enamel*FiltekZ350-XT	Cut Enamel*Dyad	Cut Enamel*FiltekZ350-XT	Dentin*Dyad	Dentin*FiltekZ350-XT
Mean	3.5	24.6	4.5	12.7	4.3	6.7
SD	1.6	6.2	2.7	4.5	1.6	1.7
P-value	0.000224 Highly statistically significant $P \leq 0.01$		0.006954 Highly statistically significant $P \leq 0.01$		0.0319845 Statistically significant $P \leq 0.05$	

The failure mode for Dyad was mainly adhesive either in un-cut enamel or cut-enamel (both were 83.3% adhesive and 16.7% mixed), while in dentin was 100% adhesive, Figure 2.

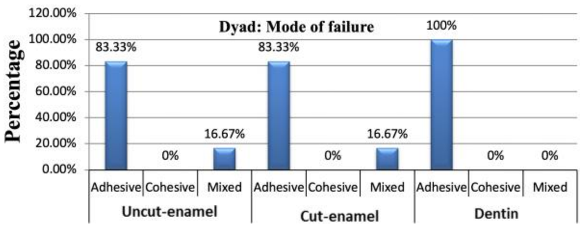

Figure 2: Histogram showing the mode of failure for Dyad in uncut, cut enamel and dentin

While the modes of failure for Filtek-Z350-XT in enamel, either cut or un-cut, were 50 % cohesive and 50 % mixed, whereas in dentin 100% adhesive, Figure 3.

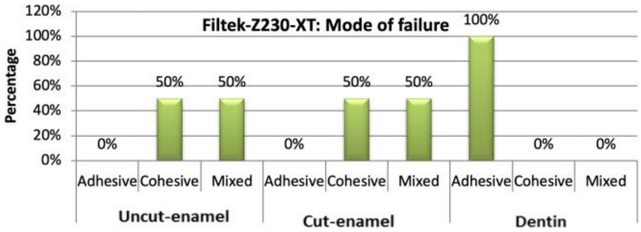

Figure 3: Histogram showing the mode of failure for Filtek-Z350-XT in uncut, cut enamel and dentin

Discussion

For this study, two types of flowable resin-composite were tested: self-adhering one (Dyad™-flow) versus flowable composite (Filtek-Z350-XT) proceeded by etching, rinse and bonding agent (Universal Single Bond). Both flowable composites were applied to uncut, cut enamel and dentin surfaces of permanent molars. In the three different surfaces of teeth, the multistep Filtek-Z350-XT showed significantly higher bond strength than the self-adhering Dyad-flow. This may be attributed to the etching and bonding steps before its application. The multi-step etch and rinse approach, contrary to the self-etch "Dyad-Flow", involved a phosphoric acid-etch step which resulted in deep pits in the hydroxyapatite-rich substrate of enamel. The mechanical interlocking of the resin tags of the bonding agent with the acid-etched enamel leads to the best bond to the enamel which effectively seals the restorations' margins on the long term [5], [6], [7], [8]. While, in the dentin the phosphoric acid demineralised the smear layer, exposing the collagen fibres of the superficially demineralised dentin. These may also increase the micromechanical interlocking of the bonding agent within the dentin surface [11], [12], [13].

Furthermore, the multi-step approach in this study utilised the single universal bond which may aid in increasing the bond strength. This may be attributed to the strong chemical bonding to the tooth structure by phosphate monomer group 10-methacryloyloxydecyl dihydrogen phosphate monomer (10-MDP) in their composition [14], [15], [16]. It was shown that an effective chemical interaction occurs between hydroxyapatite and MDP, resulting in the formation of a stable nano-layer that could form a stronger phase at the adhesive interface increasing the bond strength [17], [18]. Also, stable "MDP-Ca" salt deposition accompanied by nano-layering may clarify the high bond stability which has been proven previously in the laboratory and clinical researches [16], [19], [20]. Moreover, the ethanol solvent of single universal Bond, due to its high vapour pressure, competed with moisture, replacing it. This promoted the infiltration of monomer through the nano-spaces of the exposed collagen network. This served as a framework for the formation of a resin-demineralised dentin hybrid layer [13].

On the other hand, the Dyad-flow contains an adhesive monomer called glycerol phosphate dimethacrylate "GPDM" having an acidic phosphate functional group which could etch the teeth and claiming that it could also bond chemically with their calcium content [3]. However, it was reported previously that the chemical bonding potential of GPDM was not available [10]. This might explain the lower bond strength of self-adhering flowable resin-composite "Dyad-flow" to different teeth surfaces compared to other resin-composite (Filtek-Z350-XT) preceded by 10-MDP application in a universal single bond which chemically bond to tooth structure [14], [15], [16]. It was noticed that in both types of flowable resin-composite, dentin recorded lower shear bond strength value in comparison to uncut and cut enamel and the mode of failure was 100% adhesive in dentin regardless of the bonding technique used. This might be attributed to the higher organic content of the dentin, its heterogeneous composition and the poor wettability of its collagen fibrils by the adhesive material [4], [21], [22], [23], [24], [25]. Thus, bonding to dentin is still representing a challenge in many adhesive systems.

In conclusion, bonding of self-adhering ″Dyad-flow″ flowable resin-composite was lower than the total-etch one in enamel and dentin. Thus further material improvement may be required.

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