EGYPTIAN
DENTAL JOURNAL

Vol. 63, 3611:3616, October, 2017

I.S.S.N 0070-9484

Fixed Prosthodontics, Dental materials, Conservative Dentistry and Endodontics

www.eda-egypt.org • Codex : 90/1710

# SHEAR BOND STRENGTH AND INTERFACIAL MICROSCOPIC EXAMINATION OF TWO TYPES OF FLOWABLE RESIN-COMPOSITE TO ENAMEL SURFACE OF DECIDUOUS MOLARS

Rasha M. Abdelraouf*, Manar Mohammed** *and* Fatma Abdel Gawad***

## ABSTRACT

**Aim:** Evaluation of shear bond strength of a self-adhering flowable resin-composite versus total-etch one to enamel surface of deciduous molars. Interfaces between these restorations and primary enamel were also microscopically investigated.

**Materials and Methods:** Twenty freshly extracted human deciduous molars were used. For the shear bond strength, twelve molars were embedded in acrylic blocks, such that their buccal surfaces were aligned with the acrylic. The enamel surfaces were subjected to minimal grinding. The teeth were randomly divided into two groups, Group I: Self-adhering flowable resin-composite (Dyad™-flow, Kerr, USA); Group II: Total-etch flowable resin-composite necessitate etching and bonding (Filtek™Z350-XT, 3M-ESPE, USA). A specially designed holed-split Teflon mold was used for constructing resin-composite cylinders (3x3mm) over the buccal surfaces of the mounted teeth. For group I, resin-composite was applied directly on teeth surfaces using the mold and light-cured for 20seconds. For group II, using the mold the following steps were performed: acid etching (15 seconds), 2) bonding agent (light-curing 20 seconds) and 3) resin-composite (light-curing 20 seconds). The teeth were stored in 37°C distillate water for 24 hours. The shear bond strength was recorded and statistically analyzed. Modes of failure were studied using digital microscope. For interfacial examination, cavities (class V) were prepared in buccal surface of eight teeth, filled by the two flowable composites as previous (n=4/group) and scanned using scanning electron microscope. For each group, two teeth were examined from buccal aspect, while the other two were sectioned and inspected.

**Results:** Mean bond strength values for groups I and II were 5 and 21.6 MPa respectively with highly significant difference P=0.005 (P value ≤ 0.01). Modes of failure for groups I and II were [100%adhesive] and [16.7%cohesive within tooth + 83.33%mixed] respectively. SEM micrographs of group I revealed a gap at enamel-restoration interface, while group II showed cohesive failure within enamel at the margins.

**Conclusions:** The bonding performance of the self-adhering resin-composite "Dyad™-flow" still needs further enhancement. Modifications may be required to prevent marginal enamel cracks with the use of Total-etch "Filtek™ Z350-XT" flowable resin-composite.

* Department of Biomaterials, Faculty of Oral and Dental Medicine, Cairo University, Egypt
** Department of Pediatric and Community Dentistry, Faculty of Oral and Dental Medicine, October 6 University, Egypt
*** Department of Pediatric Dentistry and Dental Public Health, Faculty of Oral and Dental Medicine, Cairo University, Egypt

## INTRODUCTION

Demineralization and caries of enamel of primary teeth is a common clinical problem[1,2]. The primary enamel had higher dissolution tendency than permanent one. In addition, children's desire to eat sweets, may lead to the increased incidence of caries in deciduous teeth which need to be restored[3]. Due to the increased demand of tooth colored restorations, resin-composites had become restorations of choice in several cases[4]. Flowable resin-composite, being in an injection form, offered easier manipulation and better adaptation to cavity walls than conventional one[5]. By virtue of its easily handling, the application time of flowable resin-composites was reduced. Moreover, self-adhering flowable composites were introduced to further decrease the working time by eliminating acid-etching and bonding steps. "Dyad™-flow" (Kerr, USA) was introduced in the dental market as a self-adhering flowable composites applied directly to the prepared cavity without prior teeth treatment[6]. This would decrease the chair-side time which may valuable especially in pediatric dentistry. However, its bonding is still questionable. Therefore this study was conducted to assess the bond strength of a self-etch flowable composite "Dyad™-flow" to enamel of deciduous teeth and inspect the enamel-composite interface and compare these with a total-etch flowable one preceded by etching and bonding.

## MATERIALS AND METHODS

Twenty freshly extracted human deciduous molars were used; twelve for shear bond strength test and eight for interfacial examination.

### Shear bond strength test:

#### *a) Specimens Preparation:*

For each tooth, cold cure acrylic resin (Acrostone, Egypt) was mixed and packed in pre-polymerized dough stage in a rubber cylindrical mold (19mm diameter x 16mm height). Then each molar was embedded in the acrylic so that its buccal surface was aligned with the acrylic blocks. After polymerization of the acrylics, the enamel surfaces were subjected to minimal grinding using a grinding machine (Red wing, Handler, USA) under water coolant. The enamel was ground such that the superficial enamel layer was removed, *figure 1*.

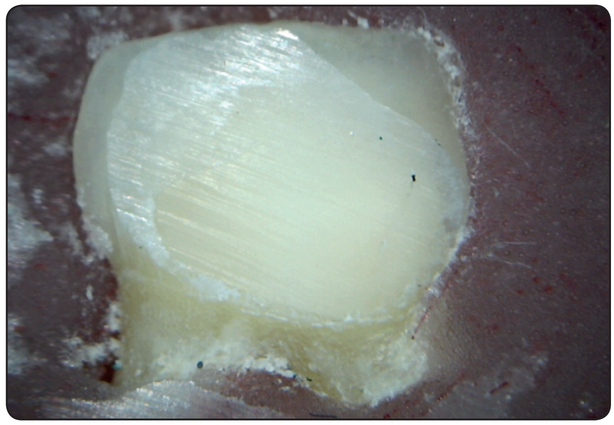

Fig. (1): Enamel surface after grinding

#### *b) Bonding Steps:*

The teeth were divided randomly into two groups according to the type of flowable composite-resin used in bonding, table1; Group I: Self-adhering flowable composite (Dyad™-flow, Kerr, USA); Group II: Total-etch flowable resin-composite need etching and bonding (Filtek™Z350-XT, 3M-ESPE, USA). A split Teflon mold with a central hole (3x3mm) was used for constructing resin-composite cylinders over the buccal surfaces of the mounted teeth. For group I, the self-adhering flowable composite "Dyad™-flow" was applied on teeth surfaces using the mold and light-cured for 20 seconds using a light emitting diode (L.E.D.) light curing unit (Satelec, Acteon, France). For group II, acid-etching was applied for 15 seconds (37% phosphoric acid, Eco-Etch, Ivoclar Vivadent, Liechtenstein), then rinsed, and air dried. This was followed by bonding-agent application (Universal-Single-Bond, 3M-ESPE, Germany), light-curing for 20 seconds, and finally applying the flowable composite"Filtek™Z350-XT" which was light cured for 20 seconds. The teeth were stored for 24 hours in 37°C distillate water.

TABLE (I) Materials used, their manufacture and composition

| Product | Manufacturer | Composition |
|---|---|---|
| Dyad™-flow | Kerr, USA | GPDM, prepolymerized filler, 1- µm barium glass filler, nanosized colloidal silica, nanosized Ytterbium fluoride |
| Filtek™ Z350-XT | 3M-ESPE, USA | Bis-GMA, Bis-EMA, TEGDMA, zirconia, silica |
| Universal-Single-Bond | 3M-ESPE, USA | Bis-GMA, MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, silane, ethanol, water |
| Eco-Etch | Ivoclar Vivadent, Liechtenstein | Phosphoric acid (37 wt.% in water), thickening agent and color pigments. |

***Where GPDM: Glycerol-phosphate-dimethacrylate, Bis-GMA; Bisphenol-A diglycidyl ether dimethacrylate, Bis-EMA: Bisphenol-A polyethylene glycol diether dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate***

### *c) Shear bond strength test:*

A universal testing machine was used (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with a crosshead speed of 0.5 mm/min. A shear force was applied via a mono-bevel-chisel. The load required for de-bonding was recorded in Newton. 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; $\tau$ =bond strength (in MPa), P =load at failure (in N), $\pi$ =3.14, r =radius of cylinder (in mm)

The strength was blindly recorded by a different assessor and the data were statistically analyzed. Modes of failure were studied using digital microscope (Scope Capture Digital Microscope, Guangdong, China), and recorded as cohesive, adhesive or mixed failure.

### *d) Statistical Analysis:*

Statistical analysis was performed 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)}. Independent-t-test was used for determining the statistical significance for the mean shear bond strength between two groups. 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.

### Interfacial examination

Class V cavities were prepared in buccal surfaces of primary molars [7], filled by the two flowable composites as previous (n=4/group). Two teeth from each group were examined from buccal surface to examine enamel-restoration interface at margins. While the other two were sectioned bucco-lingually to inspected internally enamel-restoration interface. Scanning electron microscope (SEM) (Supra40, Carl-Zeiss-NTS-GmbH, Germany) was used to assess the interface with an accelerating voltage of 20-30 kV.

## RESULTS

Mean bond strength values for groups I and II were 5 and 21.6 MPa respectively with highly significant difference P=0.005 (P value ≤ 0.01). Modes of failure for groups I and II were [100%adhesive] and [16.7%cohesive within tooth + 83.33%mixed] respectively.

SEM micrographs of group I revealed a gap (7.6µ ±0.3) at tooth-restoration interface both at margins, *figure 2* and internal interface, *figure 3*. Group II showed cohesive failure within enamel at the margins, *figures 4*, 5. A crack within enamel was observed with width 7.9µ ±0.8 and depth 128.8µ±1.

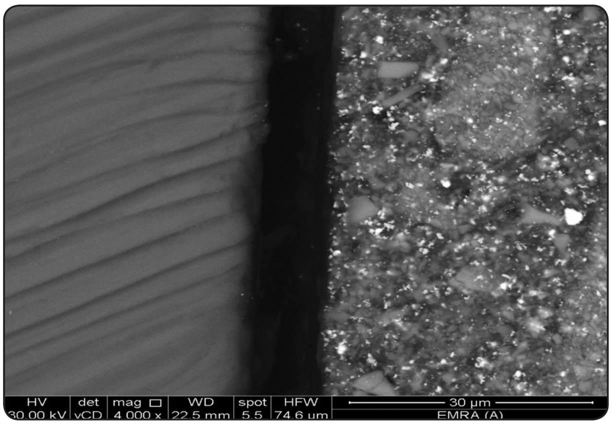


Fig. (2): Interface between self-adhering flowable composite "Dyad-flow" and enamel of intact tooth at restoration margin from buccal aspect.

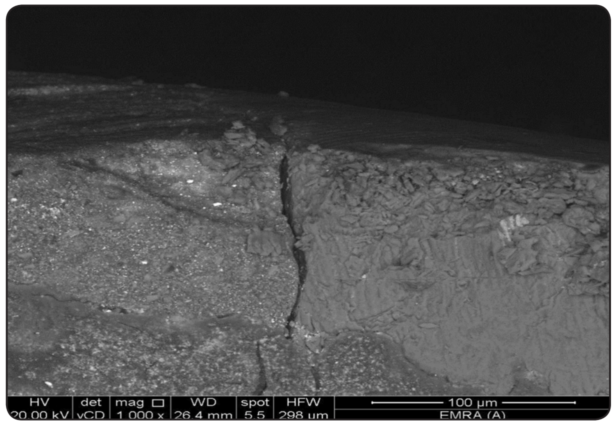


Fig. (3): Internal interface between self-adhering flowable composite "Dyad-flow" and enamel of sectioned tooth

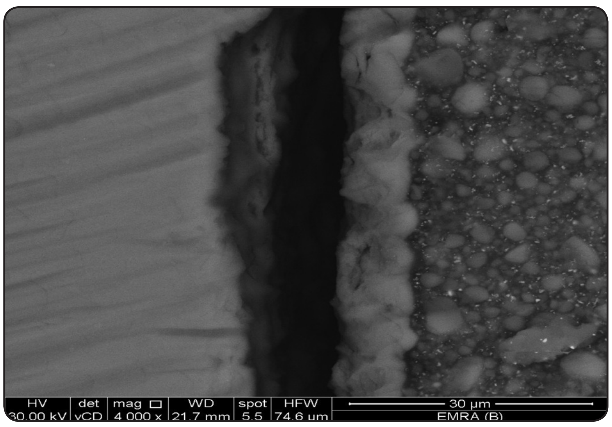


Fig. (4): Interface at margin between total-etch flowable composite "Filtek Z350-XT" and enamel of intact tooth at restoration margin from buccal aspect.

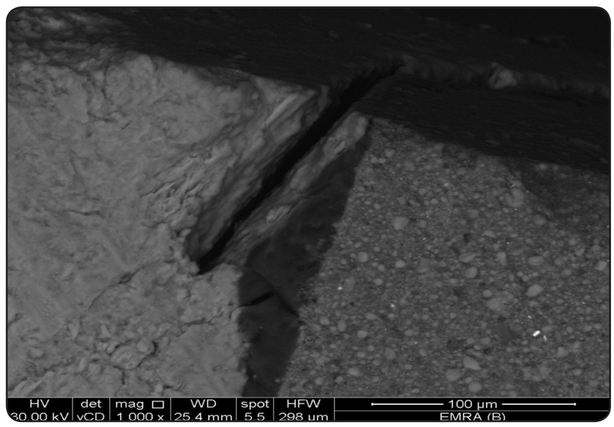


Fig. (5): Internal interface between total-etch flowable composite "Filtek Z350-XT" and enamel of sectioned tooth

## DISCUSSION

The demand for esthetic restorations increased in last years[6]. Moreover, reducing dental operation steps is another demand especially with uncooperative children[8]. Self-adhering flowable fulfilled these demands; however its bonding is questionable[9]. The gold standard in resin-composite bonding to tooth structure was the total-etch one[10]. Therefore, in this study, a self- adhering resin-composite "Dyad-flow" was compared versus total-etch "Filtek Z350-XT". Restoring early carious lesions within enamel with early diagnosis are bases for a successful minimal invasive treatment[11]. This may be helpful in pediatric dentistry. Thus, in this study primary enamel was ground simulating minimal cavity preparation[12].

The bond strength of the self-adhering flowable composite "Dyad-flow" (5 Mpa) was significantly lower than that for the total-etch "Filtek Z350-XT" (21.6 MPa) in bonding to enamel of the deciduous teeth. This may be attributed to the etching step in the latter which resulted in micro-irregularities in enamel which interlocked with the resin tags[10]. Moreover, the bonding agent used in this study (universal single bond) may increase the bond strength as it contained phosphate monomer group; 10-methacryloyloxydecyl-dihydrogen-phosphate monomer (10-MDP). This functional monomer interacted with the enamel hydroxyapatite by strong chemical bond[13].

In contrast, the self-adhering flowable composite "Dyad-flow" contained an adhesive monomer termed glycerol-phosphate-dimethacrylate "GPDM". This monomer had two functional groups; an acidic phosphate for both tooth etching and chemical bonding with its calcium content, whereas the other was methacrylate group for polymerization[6]. However, it was revealed that "GPDM" "etches" rather than "bonds" to hydroxyapatite[13]. This may lead to inferior bonding than MDP in universal single bond which chemically bond to tooth structure.

The low bond strength of the self-adhering "Dyad-flow" was confirmed by SEM micrographs, *figures 2, 3,* where a gap was present between enamel and these restorations at the margins as well as internally along cavity walls. This might be attributed to polymerization shrinkage and resultant contraction stress of resin-composite due to curing, which were not resisted by bonding to tooth structure as being weak [14]. On the other hand, cohesive failure within enamel was observed in total-etch "Filtek Z350-XT" at the margins of the cavity, *figures 4, 5*. The strong bonding to the enamel, accompanied by polymerization shrinkage may lead this cracking.

It should be noted that the polymerization shrinkage of flowable low viscosity composites were higher than high viscosity non flowable composites as the later had more filler content[13,14]. Therefore, the flowable composites suffered from a relatively higher polymerization shrinkage which may affect the interfacial bonding with tooth structure[15,16]. Competing against the bond strength, adhesive failure may occur as in self-adhering "Dyad-flow" as this bond was weak[17]. Contrary, strong bond within enamel due to etching and bonding prior to "Filtek Z350-XT" application may exceed the polymerization shrinkage led to cohesive failure in enamel rather than de-bonding. This was in agreement with Fusayama who observed fractured enamel margins around resin-composite restorations and attributed this to polymerization shrinkage stress [18]. Incidence of enamel cracks at cavosurface margins increased with phosphoric acid etching specially in cervical cavities [19]. As the cervical hydroxyapatite crystals were randomly oriented with atypical enamel prisms[20]. This may lead to enamel cracks in the class V cervical cavities in this study associated with total-etch composite.

When bonding to enamel and polymerization shrinkage of resin composite were high, stresses generated may separate enamel rods at margins from the adjacent rods. This occurred when bond strength exceeded the fracture toughness of enamel rods[21]. On the other hand, when bonding to enamel were inferior to the polymerization shrinkage of composite, de-bonding may occur[17]. Thus, such balance may be mandatory to reserve the tooth-restoration integrality at the interface which may be a critical challenge.

## CONCLUSIONS

Self-adhering "Dyad-flow" showed inferior bonding to primary enamel with gap formation at interface. Although the bond strength of total-etch "Filtek Z350-XT" was high, yet cohesive failure within enamel at margins were detected. Further materials modifications may be required.

## ACKNOWLEDGEMENT

We would like to dedicate this work to the soul of our lovely Prof. Dr. Mervat Rashed (Professor of Pediatric Dentistry, Department of Pediatric Dentistry and Dental Public Health, Faculty of Oral and Dental Medicine, Cairo University, Egypt). We wished to thank her for her cooperation and help in this research.

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