ANALYSIS OF SEM PHOTOMICROGRAPHS FOR DEBONDING PATTERN WITH NANO-COMPOSITES AS AN ORTHODONTIC BONDING ADHESIVE

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Abstract

Objective: To evaluate nanocomposite for use as an orthodontic bonding agent; using shear bond strength measurement and scanning electron microscopy to study debonding pattern. **Materials and Methods**: Study was performed in-vitro on fifty non-carious extracted healthy premolar teeth. Nano-composite (Filtek[™] Z350 XT, 3M, ESPE, St. Paul, MN) was used, Enlight Light Cure Adhesive (SDS, Ormco, CA, USA) was used in the conventional adhesive group. Debonding and shear bond testing was performed using a digital universal testing machine (UTM-G-410B, Shanta Engineering). After debonding, all teeth were examined under a Scanning Electron Microscope. Area of remaining composite on the enamel was measured using the area measurement tool of the free PDF editor Foxit® Reader version 6.1.1.1031. **Results:** The mean SBS of conventional adhesive was found to be significantly higher in the conventional adhesive group. Percent remnant adhesive was also higher in the conventional adhesive group. **Conclusion:** Nanocomposite can be potentially used as an orthodontic bonding agent.

INTRODUCTION

Buonocore (1955) published his revolutionary article on etching and adhesion of acrylic filling materials to enamel1; which led to introduction of direct bonding of orthodontic brackets to the enamel surface by Newman in 19652. Newman used epoxy adhesives for bonding orthodontic attachments to the enamel surface. Conventional bonding agents being used these days are composed of an organic diacrylate (Bisphenol A-glycidyl methacrylate: BIS-GMA), a silane coupling agent, and a high percentage of quartz, silica in the form of filler. Recent advances in adhesive technology is targeted towards achieving an optimal bond strength, which provides clinically reliable biomechanics. The bond achieved should be strong enough to control tooth movement in all three dimensions and at the same time it should be weak enough to fail safely during debonding without any damage to enamel surface. Decreasing bond failure ensures continuous control of tooth movement which results in efficient treatment. The advent of nanotechnology in dentistry has opened up new horizons for development of dental materials. Recently,

nano-fillers (aluminofluorosilicate) with mean particle size of 80 ran have been introduced which has led to increased hardness and superior flexural strength of composite. The present study was conducted on newly introduced restorative material, nano-composite (Filtek[™] Z350 XT) for use as an orthodontic adhesive in comparison with conventional light-cure bonding adhesive. Evaluation and comparison of SBS values and adhesive remnant index (ARI) scores was done.

MATERIAL AND METHOD

Fifty non-carious extracted healthy premolar teeth, extracted for orthodontic purposes, were collected over a period of a year. Root canal treated tooth or tooth with any restoration, teeth with hypoplastic areas, cracks, or irregularities of the enamel structure, pre-treatment with chemical agents such as alcohol, formalin, or hydrogen peroxide, teeth that had enamel damage as result of the extraction procedure were excluded. The teeth were stored in normal saline³ at room temperature until bonding was done. The normal saline was replaced frequently to limit bacterial proliferation. Tooth were mounted vertically in 2 cm x 1 cm x 2 cm PVC pipe filled with auto-polymerizing acrylic resin as shown in Figure 1.

All the tooth specimens were gently polished (for 10 seconds) with an oil free pumice



Figure 1: A sample tooth mounted on acrylic filled PVC tube

solution to clean the enamel surface^{4, 5}. Orthodontic brackets Sapphire Series 022' MBT UL Bicuspid bracket with hooks (Modern Orthodontics, Ludhiana) were bonded on all teeth to have a uniform bracket base area for all specimens. ART-L3 curing light (Bionart medical tech. inc., Unicorn mediden pvt. Ltd., Taiwan) with wavelength 430-470 nm, flux intensity: 1000 mW/cm2 was used for curing. The brackets were bonded in such a way that the loading blade was directed parallel to the bracket base.

Study Groups

The teeth were randomly divided into 2 groups: Group 1: Conventional Bonding Adhesive group, Group 2: Nano-composite group.

Group 1:

Enlight Light Cure Adhesive (SDS, Ormco, CA, USA) was used in the conventional adhesive group. Enamel surface was etched for 30 seconds, with Enlight etchant (37% phosphoric acid). The etched surface was then dried with clean oil and water free air for

15 seconds. Then Ortho Solo sealant supplied with the Enlight kit was applied over the etched area with a brush. Enlight adhesive paste was applied directly to the bracket base. Brackets were positioned on the facial surface and a seating pressure of 10 ounces for 10 seconds was applied in the middle of the bracket by a Dontrix gauge (E.T.M Corporation, Monrovia, California, USA)⁶.

Group 2:

After polishing of the enamel surface, bonding was done according to the manufacturer's instructions. Enamel surface was etched using Scotchbond[™] Multi-purpose Etchant (3M, ESPE, St. Paul, MN) for 15 seconds. Then the etchant was rinsed off by water jet for 10 seconds. The excess water was blotted using a cotton pellet, the surface was left glistening. Immediately after blotting 2-3 coats of adhesive (Adper[™] Single Bond 2, 3M, ESPE, St. Paul, MN) was applied to the etched enamel for 15 seconds with gentle agitation using a fully saturated applicator. Then it was gently air thinned for five seconds to evaporate the solvents, followed by light curing for 10 seconds. Nano-composite (Filtek[™] Z350 XT, 3M, ESPE, St. Paul, MN) was then coated on the bracket base mesh directly from the syringe. The bracket was then positioned under a seating pressure of 10 ounces for 10 seconds. The adhesive was then cured for 20 seconds (10 seconds from mesial and 10 seconds from a distal direction).

Debonding procedure: Bond strength testing

Debonding and shear bond testing was performed after 24 hours from bonding⁷ using a digital universal testing machine (UTM-G-410B, Shanta Engineering). The specimen was clamped in the attachment and a tangential load was applied by the loading plunger at a crosshead speed of 1mm/minute. The force was directed at the ligature groove, between the wings and base, for consistency, stability and accuracy⁸.

This force application was more representative of in-vivo loading and ensured a more consistent application of debonding force due to less chances of slipping of the metal blade⁹. The debonding force was parallel to the bracket/adhesive interface. The load was measured in Newton. The values obtained were divided by the bracket base area which was 9.152 mm² (measured by Optical Profile Projector); to obtain SBS in Megapascal (MPa).

After debonding, all teeth were examined under a Scanning Electron Microscope (JEOL, JSM-6510 Series). The labial surface were coated with gold-palladium plating and the specimens were then examined under the Scanning Electron Microscope at 27x magnification, 10 kV. The photomicrograph of the enamel surface obtained from scanning electron microscopy were evaluated for the area of remaining composite on the enamel using the area measurement tool of the free PDF editor Foxit® Reader version 6.1.1.1031 as shown in figure 2.

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Figure 2: Area Calculation using the free PDF reader Foxit[®]. First the Scale provided by the SEM machine at the bottom (white arrow) was measured to provide a working scale. Region marked A denotes the bond failure at the bracket enamel interface. Subtracting Area 1 from total adhesive area (Area 3) gives area of residual adhesive on the tooth surface. Region B denotes the area of cohesive failure and its area can be calculated by subtracting Area 1 from Area 2

ARI was evaluated along with percentage adhesive remnant on tooth surface. ARI coding was done using the criteria proposed by Årtun and Bergland in 1984¹⁰. Cohesive failure was also identified on the SEM photomicrograph as area of remnant adhesive without bracket base impression.

VALUE	CRITERION	INTERPRETATION
0	No adhesive left on the enamel.	Fracture at enamel- adhesive interface
1	Less than half of the adhesive left on enamel.	
2	More than half of the adhesive left on enamel.	Fracture at cement-
3	The entire adhesive left on the enamel with distinct impression of bracket mesh.	bracket interface

 Table 1: Adhesive Remnant Index (ARI) Given by Årtun and Bergland

Statistical Analysis

SBS, ARI, percentage remnant adhesive and cohesive failure are were expressed as mean \pm SD and were compared using independent samples t test, with calculation of 95% confidence interval

(CI) using bootstrapping. A two-sided P-Value <0.05 was considered to be statistically significant. All analyses were performed on the statistical software SPSS v21.0.0 64-bit edition for Windows.

RESULTS

The mean SBS of conventional adhesive was found to be significantly higher in the conventional adhesive group (Table 2). The mean difference in SBS between the two groups was 2.76 MPa (95% CI - 1.73 to 3.77). The ARI as well as percent remnant adhesive was also higher in the conventional adhesive group. However there was no significant difference in cohesive failure between both groups.

		SBS (MPa)	ARI	Remnant Adhesive (%)	Cohesive Failure (%)
Conventional		10.59 ± 2.03	1.96 ± 0.79	67.16 ± 32.96	4.04 ± 5.18
Nanocomposite		7.84 ± 1.51	1.12 ± 1.01	35.80 ± 37.99	2.40 ± 3.92
Mean Difference		2.76	0.840	31.36	1.64
95% CI of mean	Lower	1.73	0.32	11.14	-0.97
difference	Upper	3.77	1.36	51.59	4.25
P- Value		<0.001	0.002	0.003	0.213

Table 2: Comparison of Study Groups

DISCUSSION

Newer materials are continually introduced in operative dentistry, orthodontists adopt some of these innovations like the use of self-etching primers, RMGIC, chlorhexidine, varnishes etc¹¹. Nano-composite has been recently introduced with promising finish and flexural strength. This study was conducted to compare SBS of nano-composite and conventional light cure composite as orthodontic bonding agent.

In vitro shear bond strength test does not simulate the clinical situation; in the oral cavity the potential loading is complex with multidirectional forces acting on the enameladhesive and adhesive-bracket interface as well as stresses introduced by application of orthodontic forces. However, in-vitro SBS testing gives an indication of the anticipated bond strengths in vivo.

Commercially available adhesive systems have different particle sizes, variable viscosities and concentrations of fillers. According to the manufacturers the Filtek

Supreme Plus universal restorative nano-composite that was used in this study contains a unique combination of two types of nanofillers (5-75 nm) and nanoclusters¹². Nanoparticles are discrete non-agglomerated and non-aggregated particles of 20-75 nm in size while nano-cluster fillers are loosely bound agglomerates of nano-sized particles.

The agglomerates act as a single unit enabling high filler loading and high strength. Ostertag et al.¹³ found an increase in shear and torsional bond strengths with increasing concentrations of adhesive filler. Thus it was expected to achieve higher bond strength for nano-composites, however in our study the SBS values for nano-composites was significantly lower than the conventional light-cure bonding adhesives.

The SBS results of the present study are similar to those of Uysal et al (2010)¹⁴ and Hosseinzadeh-Nik et al (2013)¹⁵ who found that the conventional orthodontic adhesives showed higher SBS values than the nano-composite and the difference was statistically significant. The consistency of the nano-composite adhesive paste is fairly thick and it does not flow readily which makes its manipulation difficult.

Lower SBS achieved in our study may be attributed to the compact consistency of Filtek[™] Supreme plus Universal Restorative System. According to Proffit and Fields¹⁶ a successful orthodontic adhesive should be fluid enough to penetrate etched enamel.

After SBS test, ARI was evaluated for all the specimens in the two groups to identify the weakest point in the bracket-adhesive-enamel system using SEM. Enamel damage and remnant index can be easily assessed on SEM; however SEM can provide only subjective information. No enamel damage was observed in any of the specimen used in this study.

To quantify remnant adhesive the open source free PDF editor Foxit, Adobe Acrobat was used. Cohesive failure was quantified in a similar way; by measuring area covered by adhesive without bracket base impression.

This was be easily done on SEM photomicrographs, as adhesive remnant with/without bracket base impression are clearly defined. For the assessment of the failure site of debonded interfaces, most studies have used the ARI^{17-18} . ARI depicts bond failure which can either be adhesive failure occurring between enamel and adhesive, or between adhesive and bracket or cohesive failures occurring within the adhesive, within the tooth, or within the bracket itself. Residual adhesive in percentage has been reported in this study as it is more accurate than ARI, where each score represents a wide percentage of remaining adhesive, e.g., ARI: 1= 0-50%.

Also various modified ARI have been given after the original iteration of ARI which was given by Årtun and Bergland^{10, 17, 19}. Residual adhesive in percentage will allow a more uniform way of reporting data for comparing similar studies. Residual adhesive in conventional composite group was higher than nano-composite group. When ARI scores were compared, it was found that ARI score for conventional composites was higher than that of nano-composite.

Lower ARI in the range observed for nano-composite can be attributed to the thick consistency of the adhesive. Advantages of lower ARI included; lesser chair-side time taken to mechanically remove remnant adhesive after removal of the bracket. Enamel damage during mechanical adhesive removal and polishing is also reduced²⁰.

Usually adhesive and cohesive failure co-exist (Powers & Messersmith, 2001)²¹, this mixture of failure patterns has been demonstrated clinically as well²². A comparison of cohesive failure revealed mean percent cohesive failure was higher for conventional composite, Enlight. Cohesive failures reflect high adhesion strengths, as the adhesion between adhesive-enamel and adhesive-bracket interface would be so strong that failure within the material occurs.

CONCLUSION

Following conclusions were drawn from the present study

- 1. SBS achieved for nano-composite and conventional orthodontic bonding adhesive were within clinically acceptable range, although it was significantly higher for conventional orthodontic bonding adhesive as compared to nano-composite.
- 2. Open source free PDF editor Foxit, Adobe Acrobat served as an effective tool for comprehensive analysis of SEM photomicrographs.
- 3. Adhesive remaining on tooth was found to be significantly higher for conventional orthodontic bonding adhesive as compared to nano-composite.
- 4. Mean percent cohesive failure was higher for conventional composite reflecting high adhesion strength.

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