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Original Research

Comparative Evaluation of Bond Strength of Dual-Cured Resin Cements: An *In-Vitro* Study

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Abstract:
Background: To compare the microtensile bond strength of resin cements to enamel and dentin and to determine the type of bond failure using stereomicroscope.

Materials and Methods:
In this *in-vitro* study 40 teeth were embedded in acrylic resin and divided into two main groups i.e., Group A for enamel and Group B for dentin. Each group is again subdivided into four subgroups, which are as follows; Subgroup 1 for Calibra resin cement, Subgroup 2 for Paracem, Subgroup 3 for Variolink II and Subgroup 4 for Rely X ARC. These resin cements were applied on enamel and dentin according to manufacturer's instructions followed by incremental build-up of composite resin on the top of resin cements. Each tooth was sectioned perpendicular to the resin-substrate interface with a slow speed diamond saw under water cooling yielding sections of approximately 1 mm^2. On an average, three sections from each tooth were used for testing. The beams obtained after sectioning were stressed to failure under tension in a custom made stainless steel forceps held in a universal testing machine (Lloyd) at a crosshead speed of 1.0 mm/min. Results were analyzed using two-way analysis of variance, independent *t*-test, and Tukey's HSD *post-hoc* test.

Results: Cements bonded to enamel substrates showed higher mean bond strength compared to dentin, which is statistically significant. Rely X ARC showed highest mean bond strength to both the substrates.

Conclusion: There was a significant difference between the bond strength to enamel and dentin and, Rely X ARC resin cement showed higher bond strength compared with the other groups.

Key Words: Dentin, enamel, resin cements

Introduction

Adhesive dentistry emphasizes on the development of materials to understand the interaction with the tooth tissues.[1] The rapid advancement in adhesive technology has extensively influenced modern restorative dentistry. Despite numerous advances made in adhesive technology during the last 50 years, the bonded interface itself remains the Achilles heel of an adhesive filling. Especially water sorption is thought to destabilize the adhesive-tooth bond, which include the heterogeneity of tooth structure and composition, the features of the dental surface exposed after cavity preparation, and the characteristics of the adhesive itself, such as its strategy of interaction with both substrates and its basic physicochemical properties.[2]

Cementation is a vital step in the process of retention, marginal seal and durability of indirect restorations. From the past two decades, resin cements continue to evolve with advancements in adhesive dentistry.[3]

Dual-polymerizing resin luting agents are widely used to bond esthetic indirect restorations because of their low solubility, low viscosity, clinically acceptable film thickness, better mechanical properties than conventional cements, ability to bond tooth to restorative material when used with bonding agents, and lower microleakage compared to other luting materials.[4]

Effective adhesion to enamel has been achieved with relative ease and has reportedly proven to be a durable and clinical procedure for routine applications in modern adhesive dentistry.[5]

Bonding to dentin has been considered more difficult and less predictable. The main obstacle is the heterogenous nature of dentin, with hydroxyapatite deposited on a mesh of collagen fibers with hydrophilicity definitely presenting one of the major challenges for the interaction of modern adhesives with dentin.[6,7]

Different mechanical tests can be found to evaluate bond strength to tooth structures. The microtensile bond strength (μ-TBS) is more versatile, as there is better economic use of teeth, as multiple specimens obtained from a single tooth enable more inventive set-ups and better-controlled substrate variables. However, several micro-specimen preparation protocols are being used worldwide, one is being more technique-sensitive than the other. Today, so-called "trimmed" and "non-trimmed." Micro-specimens are prepared, both having advantages and disadvantages. Non-trimmed micro-specimens are definitely most easy and fast to prepare. Trimming the micro specimens at the interface to so-called hourglass-shaped specimens better concentrates stresses at the interface, but involves a more invasive specimen procedure.[2]

Materials and Methods

The present *in-vitro* study was conducted in the Department of Conservative Dentistry and Endodontics, M.R. Ambedkar Dental College and Hospital, Bengaluru. 40 recently extracted intact human molars were used in this study. Attached soft tissue and calculi are removed with a hand scaler. Teeth were cleaned with slurry of pumice and water and examined under a ×30 stereomicroscope (Lynx, Shimadzu, Tokyo, Japan) to ensure that they were free of surface cracks, decalcification or any sign of previous grinding. The teeth were then placed in 0.9% physiologic saline prior to preparation. Teeth selected were randomly divided into two groups of 20 each. Each group was later sub-divided into four subgroups of five each.

Forty teeth were embedded in acrylic resin after sectioning of roots in a custom made alignment to enable standardization of the specimen during curing. The buccal enamel surface was then demarcated to outline the flat-test area for bonding. The occlusal third of the buccal and lingual surfaces was usually outside the bonding area due to its inclination. The enamel surfaces were manually ground with a 60 grit -silicon carbide paper under water cooling for 60 s. For dentinal exposure, the crowns of the teeth were sectioned using a low-speed diamond disc with water lubrication up to 1 mm below the dentino-enamel junction. Any remaining enamel was removed by polishing with silicon carbide paper. An artificial smear layer was obtained by wet grinding with 600 grit silicon carbide paper.

The commercial name, composition, and the manufacturer or the materials used in the study are listed in Table 1.

Forty teeth selected were randomly divided into two groups of 20 each. Each group was later sub-divided into four subgroups of five each.

Subgroup A1 and B1

Calibra resin cement was used with dual-cured Prime and Bond NT (two step etch-and-rinse bonding system). The substrates were etched for 15 s with 37% phosphoric acid, then rinsed for 30 s and dried. Mix equal drops of Prime and Bond NT and self-cure activator into the same mixing well for 1-2 s with a clean, unused brush tip. Using the disposable brush supplied, immediately apply mixed adhesive/activator to thoroughly wet all the tooth surfaces. Cure mixed adhesive/activator for 10 s using a curing light.

Equal parts of base and catalyst were mixed for 20 s, and then applied to the surface of the tooth. To standardize the amount of luting material placed on each specimen, templates was prepared, which was 3 ± 0.2 mm for all specimens. The composite resin of dimensions 4 mm × 4 mm was built incrementally over the cured resin cement. The cement was pre-cured for 20 s, all excess was removed. The cement was cured for 40 s in two different areas for a total of 80 s.

Subgroup A2 and B2

Paracem dual-cured resin cement was used with Parabond adhesive system (two-step self-etch), which is chemical curing adhesive system. The tooth substrates were conditioned for 30 s with a non-rinse conditioner and gently air-dried for 2 s. After conditioning, equal amounts of adhesive A and adhesive B were mixed and applied to the dentin surface in two coats, the bonding agent was left in place undisturbed for 30 s before the excess was gently air dried.

Procedure is same as mentioned above.

Subgroup A3 and B3

Variolink cement was used with dual-cured ExciTE F DSC (two step etch-and-rinse). The substrates were etched for 15 s with 37% phosphoric acid, then rinsed for 30 s. Apply it to the enamel and dentin and agitate the adhesive on the prepared surfaces for 10 s. Light cure for 10 s.

The procedure is same as mentioned above.

Subgroup A4 and B4

This cement was used with Adper Single Bond 2 (two step etch-and-rinse). Substrates were etched for 15 s with 37% phosphoric acid, then rinsed for 30 s and dried just enough to remove all the water from tooth surface, but without desiccating the tooth. Immediately after blotting, apply 2-3 consecutive coats of Adper Single Bond 2 adhesive to etched enamel and dentin for 15 s with gentle agitation using a fully saturated applicator. Gently air thin for 5 s to evaporate solvents. Avoid excess adhesive on all prepared surfaces. Light-cure for 10 s per bonding surface.

Procedure is same as mentioned above.

Specimens were stored at 37°C in 100% relative humidity for 24 h to ensure complete polymerization. Each tooth was sectioned perpendicular to the resin-substrate interface with a slow speed diamond saw under water cooling yielding sections of approximately 1 mm^2 (Figure 1). On an average, three sections from each tooth were used for testing. The beams obtained after sectioning were stressed to failure under tension in a custom made stainless steel forceps (Figure 2) held in a universal testing machine (Lloyd) at a crosshead speed of 1.0 mm/min (Figure 3).

Subsequent to μ-TBS, fracture modes were observed by Stereomicroscopic observations of both sides of the failed bonds. The actual mode of failure was recorded according to the following criteria given by Morais *et al.*[4]

Type I	Adhesive along the cement and substrate interface
Type II	Cohesive within the substrate
Type III	Cohesive within the LA
Type IV	Mixed when simultaneously exhibiting the remnants of both the hybrid layer and the LA

LA: Luting agent

Statistical analysis
The statistical data derived from the bond strength of four dual-cured resin cements to enamel and dentin was analyzed by two-way analysis of variance (ANOVA), independent *t*-test and Turkey's HSD ($P < 0.05$) to determine the effect of the substrate on each material.

Results
When mean bond strengths of four resin cements (Subgroups 1, 2, 3, 4) were compared, Subgroup 4 (Rely X ARC) showed highest mean bond strength to both the substrates, which is significant ($P < 0.001$) compared with other groups, whereas Subgroup 2 (Paracem) showed the lower bond strength to both the substrates when compared to other groups (Table 1 and 2).

When comparison of mean TBS to enamel and dentinal substrates was done using two-way ANOVA, cements bonded to enamel substrates showed higher mean bond strength compared to dentin, which is statistically significant with $P < 0.001$ (Tables 3 and 4).

Discussion
The present study was conducted with μ-TBS test with non-trimming version. It apparently places stress on the adhesive interface during specimen preparation and handling.[8]

Very few studies were done comparing the μ-TBS of dual-cured resin cements to enamel and dentin.[9-11]

The present study compared the μ-TBS of dual-cured resin cements to enamel and dentin on recently extracted human molar teeth. Four resin cements were compared i.e., Calibra (two-step etch-and-rinse adhesive system), Paracem (two-step self-etch adhesive system), Variolink II (two-step etch-and-rinse adhesive) and Rely X ARC (two-step etch-and-rinse adhesive system). These cements were bonded to enamel (buccal surface of the tooth) and to the dentin (1 mm below the DEJ on the occlusal surface) of the tooth according to the manufacturer's instructions.

The results obtained were system and substrate specific. Mota *et al.*[12] and Eick *et al.*[13] claimed that bond strengths to enamel and dentin should be higher than 20 MPa to compensate adequately for the stresses caused by polymerization shrinkage.

When substrates were compared, the study showed significant bond strength to enamel compared to dentin. This is in agreement with studies done by La Fuente *et al.*[9] and Ritter *et al.*[11]

This can be explained on the basis that on enamel, acid-etching selectively dissolves the enamel rods, creating micro porosities, which are readily penetrated, even by ordinary hydrophobic bonding agents, creating micromechanical interlocking of resin tags with capillary attraction, whereas on dentine the main obstacle is the heterogeneous nature, with hydroxyapatite deposited on a mesh of collagen fibers. In addition, dentin is closely connected with pulp tissue through numerous fluid-filled tubules, which traverse through dentine from the pulp to the dentino-enamel junction. The hydrophilic property of dentin represents one of the major challenges for the interaction of current adhesives with dentin. The presence of smear layer and smear plugs that obstruct the dentinal tubules is also an another co-factor that may not be underestimated.[6]

On enamel, among the resin cements tested, significant differences in the mean bond strength was found. Rely X ARC showed an average mean bond strength value of 29.36 MPa, which is statistically significant compared to other dual-cured resin cements.

Acid-etching creates micro porosities on the inter-prismatic enamel through which the hydrophobic monomers of the bonding agent may penetrate creating high micromechanical retention.[14] Rely X ARC used with Adper Single bond 2 has shown low viscosity, which could contribute for its best penetration into the surface to provide reliable bond strength preventing micro leakage.[13-15] The low viscosity monomer

Table 1: Mean bond strength of the four subgroups in enamel substrates.

Group A (enamel)			
Subgroups	**Mean**	**SD**	***P*-value**
Subgroup A1	21.23	1.72	<0.001
Subgroup A2	17.63	1.67	
Subgroup A3	24.25	1.44	
Subgroup A4	29.36	2.30	

SD: Standard deviation. *P*<0.05 indicates statistically significant difference between the groups, one-way ANOVA done among subgroups in Group A [Table 3] showed that subgroup A4 showed highest mean bond strength whereas subgroup A2 showed lowest bond strength

Table 2: Mean bond strength of the four subgroups in dentinal substrates.

Group B (dentin)			
Subgroups	**Mean**	**SD**	***P*-value**
Subgroup B1	18.76	1.79	<0.001
Subgroup B2	18.66	1.10	
Subgroup B3	21.35	1.50	
Subgroup B4	26.48	1.24	

P<0.05 indicates statistically significant difference between the groups. One-way ANOVA done among Sub groups in Group B [Table 2] showed that Sub group B4 showed highest mean bond strength whereas Sub group B2 showed lowest bond strength

Table 3: Inter comparison of TBS between subgroups in Group A done using Tukey's HSD *post-hoc* multiple comparison test.

(I) Cement	(J) Cement	Mean difference (I-J)	*P*-value
Subgroup A1	Subgroup A2	3.606	<0.001
	Subgroup A3	3.017	<0.001
	Subgroup A4	8.129	<0.001
Subgroup A2	Subgroup A3	6.623	<0.001
	Subgroup A4	11.735	<0.001
Subgroup A3	Subgroup A4	5.112	<0.001

TBS: Tensile bond strength, The mean difference is significant at the 0.05 level, inter-group comparison done between the three subgroups using Tukey HSD *post-hoc* test, higher mean difference is found between subgroup A2 and subgroup A4, lower mean difference is noticed between subgroup A1 and subgroup A3

Table 4: Inter comparison of TBS between subgroups in Group B done using Tukey's HSD *post-hoc* multiple comparison test.

(I) Cement	(J) Cement	Mean difference (I-J)	*P*-value
Subgroup B1	Subgroup B2	0.101	0.997
	Subgroup B3	2.588	<0.001
	Subgroup B4	7.719	<0.001
Subgroup B2	Subgroup B3	2.689	<0.001
	Subgroup B4	7.820	<0.001
Subgroup B3	Subgroup B4	5.131	<0.001

TBS: Tensile bond strength. The mean difference is significant at the 0.05 level, inter-group comparison done between the three subgroups using Tukey'S HSD *post-hoc* test, when sub group B1 is compared with subgroup B2, mean difference between the groups is 0.101 which is not statistically significant, higher mean difference of 7.820 is noticed among subgroup B2 and subgroup B4

allows better mobility and distribution of free radicals inside the resin material, which can increase the polymerization reaction and the monomer conversion. This cement also contains larger amount of chemical and physical initiators, which in turn resulted in higher degree of conversion under the three polymerization conditions.[13]

On dentine, among the resin cements tested, significant differences in the mean bond strength was found. Rely X ARC showed an average mean bond strength value of 26.48 MPa, which is statistically significant compared with other dual-cured resin cements.

This could be attributed to that on application of 37% phosphoric acid, the exposed collagen of superficial demineralized dentin may provide reactive groups that can chemically interact with bonding primers. The solvent present in Single bond, due to its high vapor pressure, helps in forming a framework for the creation of a resin-demineralized dentin hybrid layer, resulting in a strong micromechanical interlocking between resin and the superficially demineralized dentin. This could probably account for the higher bond strength values showed by Subgroup 4 when compared to all the other groups.[8]

It should be considered that adhesive systems containing acetone (Prime and Bond NT) used with (Calibra) or ethanol (ExciTE F DSC) used with Variolink II are unable to rehydrate a surface that was dried after applying acid to form an effective hybrid layer, whereas Adper Single bond 2 used with Rely X ARC is an ethanol based adhesive, which invariably contains small amount of water. This extrinsic water together with an increase in intrinsic moisture caused by the removal of the smear layer could have resulted in rehydration of the partially collapsed collagen matrix during the adhesive application enhancing the bonding.

In this study, Paracem cement showed the least mean bond strength to enamel and dentine (17.63 MPa and 18.66 MPa) when compared to other cements whereas in contrast to other cements, it showed higher bond strength to dentine compared to enamel though which is not statistically significant. This is in agreement with studies of Perdigão and Geraldeli[16] and Ritter *et al.* [11] whereas conflicting results were reported by Hannig *et al.*[16] Shimada *et al.*[17] and Hikita *et al.*[10] who demonstrated that enamel bonding with self-etch adhesives is of the same magnitude as enamel bonding after phosphoric acid etching.

The results of this study could be attributed to that Paracem is used with two-step self-etch adhesive system, which showed minimal etching pattern with enamel resulting in shallow inter-crystallite infiltration of the resin and lack of inter-prismatic resin tag formation, which in turn results in poor penetration of adhesive monomer,[1,15] whereas on dentine, the lower bond strength could be attributed to that, self-etching priming systems combine the etching and priming steps. This can be attributed to the calcium and phosphate ions being solubilized from the apatite crystals, which are suspended in alcohol and water solvents in the primer, which limits the ability of adhesives to penetrate the primed surface, due to the buffering capacity of dentin and due to the high ion concentrations of calcium and phosphate.[8] In addition to it, chemical incompatibility between chemical/dual-cured resin cements and simplified adhesive systems can negatively influence the adhesive cementation of indirect restorations, which in turn result in incomplete polymerization of resin-based materials.[19]

A review by Kramer *et al.* showed that the newer luting materials exhibit excellent flow characteristics with mean film thickness ranging from 8 to 21 µm. The thickness of the luting materials used in the study was not equal to the thickness usually obtained clinically beneath the indirect restorations.[20]

In this study, in order to produce specimens viable for testing with a standardized thickness, a 3 mm thick layer is used. Therefore, these materials may produce different results when compared to that applied in a thin layer.

The loss of specimens during microtensile specimen preparation is not uncommon. In the present study also pretesting failures were observed. Several approaches have been applied to deal with the pretesting failures: (a) Exclude all pretesting failures from further statistical analysis, which obviously overestimates the actual bond strength; (b) assign a bond strength value of for instance 0 MPa to each pretesting failure. This actually penalizes the product too severely, as there was a certain bond strength; and (c) a modification of the former approach by assigning a pre-determined value to each pretesting failure, as for example the lowest µ-TBS value measured within the respective Group 2 however, in the present study third approach is followed i.e., the pretested samples were assigned a low µ-TBS value measured within the respective group.

In the present study, fracture modes were observed by stereomicroscopic at ×30 magnification. Observations of both sides of the failed bonds were evaluated and it was observed that bond strength values may be accountable for the modes of failures at the bonded interface as the cements with higher bond strength values have increased cohesive failures.[21] Failure modes of resin cements to enamel and dentin were given

For Rely X ARC and Variolink II predominantly failures were cohesive in resin cement. This could be attributed to the presence of the suitable bond between the resin adhesive and tooth substrate as found in accordance with the studies done by La Fuente *et al*.,[9] Thaine *et al*.[18] whereas for Paracem resin cement, more failures were adhesive between the resin cement and substrate interface. This could be attributed to the higher permeability of the simplified self-etch systems, which promote faster hydrolytic degradation. In addition to hydrolytic degradation, chemical incompatibility between dual-cured resin cements and simplified adhesive systems can negatively influence the adhesive cementation of indirect restorations resulting in incomplete polymerization of resin-based materials.[18]

Therefore, the results of the present *in-vitro* study provide an insight for the clinicians about the interaction of the dual polymerizing materials with etch-and-rinse and self-etch adhesives systems to enamel and superficial dentin. Further *in-vivo* studies are required to explore various aspects of these systems.

Conclusion

It can be concluded that the μ-TBS value obtained for Group A was significantly higher than Group B. among the cements tested in Group A and Group B, Rely X ARC (Subgroup 4) showed higher bond strength, followed by Variolink (Subgroup 3), Calibra (Subgroup 1) and Paracem (Subgroup 2).

When failures modes were assessed most of the specimens in Rely X ARC showed cohesive failures in the resin cement whereas for Paracem resin cement most of the specimens debonded in adhesive mode.

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