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Original Article

Dentistry Section

Tensile Bond Strength of Self Adhesive Resin Cement After Various Surface Treatment of Enamel

SAHIL SEKHRI[1], SANJEEV MITTAL[2], SANDEEP GARG[3]

ABSTRACT

Introduction: In self adhesive resin cements adhesion is achieved to dental surface without surface pre-treatment, and requires only single step application. This makes the luting procedure less technique-sensitive and decreases postoperative sensitivity.

Aim: The purpose of this study was to evaluate bond strength of self adhesive resin after surface treatment of enamel for bonding base metal alloy.

Materials and Methods: On the labial surface of 64 central incisor rectangular base metal block of dimension 6 mm length, 5mm width and 1 mm height was cemented with RelyX U200 and Maxcem Elite self adhesive cements with and without surface treatment of enamel. Surface treatment of enamel was application of etchant, one step bonding agent and both. Tensile bond strength of specimen was measured with universal testing machine at a cross head speed of 1mm/min.

Results: Least tensile bond strength (MPa) was in control group i.e. 1.33 (0.32) & 1.59 (0.299), Highest bond strength observed when enamel treated with both etchant and bonding agent i.e. 2.72 (0.43) & 2.97 (0.19) for Relyx U200 and Elite cement. When alone etchant and bonding agent were applied alone bond strength is 2.19 (0.18) & 2.24 (0.47) for Relyx U200, and 2.38 (0.27) 2.49 (0.16) for Max-cem elite. Mean bond strength was higher in case of Max-cem Elite as compared to RelyX U200 resin cement, although differences were non–significant ($p > 0.05$).

Conclusion: Surface treatment of enamel increases the bond strength of self adhesive resin cement.

Keywords: Acid etching, Bonding agent, Enamel, Self etch

INTRODUCTION

In current dental practice, minimal invasive procedures are widely advocated as they preserve as much sound tooth structure as possible. An enamel adhesive technique is used in dental procedures such as veneers and resin-bonded fixed partial dentures [1]. In addition to preservation of dental hard tissues in these prosthodontic restorations other advantage is no need of analgesia.

Buonocore first used acid to etch enamel for sealing pits and fissures in 1955 [2]. Adhesion to enamel is achieved through acid etching of this highly mineralized substrate; which increases the surface area for bonding. Etching creates micro porosities in enamel, resin flows into them and form tag like extensions [3]. Enamel bonding is the main reason for the clinical success of several restorative procedures [4].

Resin cements are the material of choice for cementation of indirect adhesively cemented restorations. These have improved physical characteristics, lower solubility & better wear resistance and marginal closure. The longevity of indirect restorations is directly related to the adhesive effectiveness between dental tissues and resin cements. Therefore, a durable bond at the tooth restoration interface is fundamental for long-term success of an adhesive restoration.

Depending on the treatment of dental tissues, resin cements can be classified as total-etch, self-etch, and self-adhesive resin cements. Total etch resin cement requires the use of phosphoric acid followed by multi or 2-step total-etch adhesive before the application of the resin cement. Self-etch resin cements use an acidic or self-etching primer, which is not rinsed away, to modify the dental tissue surfaces before bonding. Self-adhesive resin cements were introduced in 2002. They are also called as universal, all-purpose or multipurpose resin cements. These cements are able to bond to dental tissues without previous application of a bonding adhesive [5]. Resin matrix of these cements consists of multifunctional acid methacrylate (carboxylic or phosphoric) [6] that demineralize/infiltrate into the tooth substrate, resulting in micro-mechanical retention [7,8]. It has been reported that chemical retention may also occur by monomer bonding to Ca^{2+} ions of hydroxyapatite [9].

As multi-step application technique is complex and rather technique sensitive, there is a growing interest in the use of self-adhesive resin cements as adhesion is possibly achieved to dental surface without surface pre-treatment, and requires only single step application [10]. This makes the luting procedure less technique-sensitive and decreases postoperative sensitivity. Many claim superiority in retentive strength, working and handling characteristics [11,12].

Conventional resin cements have established a reputation for acceptable bonding effectiveness. However, there are conflicting reports regarding the enamel bond strengths of indirect restorations using self-adhesive resin cements [7,8,13,14]. The acidity of nonrinsed acidic primers may negatively affect the bond strength to enamel [15-17]. Some found improved bonding after phosphoric acid pre-treatment of dental surface before the application of self-adhesive resin cements [18-20]. However, the effectiveness of acid pre-treatment of enamel has been questioned [21,22]. Thus, the purpose of this study was to compare and evaluate tensile bond strength of self-adhesive resin cements to dental enamel with and without surface treatments.

MATERIALS AND METHODS

The present invitro study was conducted in the Department of Prosthodontics including crown and bridge, MM College of Dental Sciences and Research, Mullana, Ambala in 2014. The study was designed for comparative evaluation of tensile bond strength of self-adhesive resin cements bonded to enamel with and without surface treatment.

Specimen Preparation

Non-carious, unrestored, human incisors of comparable crown sizes from patients of age 40-50 were stored in 0.5% chloramine in distilled water. Teeth were cleaned with ultrasonic scaler to remove any soft tissue and calculus. All teeth were examined carefully to rule out pre-existing crown fracture, hypoplasia, caries and restoration. Teeth were embedded in chemically cured acrylic resin blocks. Labial surfaces of teeth were flattened with diamond point to obtain a flat enamel surface.

Impressions of 64 prepared teeth were made with monophase polyvinyl siloxane impression material. Rectangular wax pattern of 6 mm length, 5mm width and 1 mm height was fabricated. A loop was attached to the centre of wax block, which will serve as attachment to be connected to the universal testing machine. All (64) rectangular wax patterns were invested and casted using Ni-Cr alloy. The castings thus obtained were then sandblasted using 50 µm aluminium oxide particles at 6.3MPa pressure for 10 seconds at a distance of 10mm.

Sixty four tooth specimens with Ni-Cr rectangular blocks were randomly assigned to four groups according to type of surface treatment of enamel. Each group contains 16 specimens.

GROUP 1 – Control group. No surface treatments of enamel.

GROUP 2 – 35 % phosphoric acid {3M ESPE, St. Paul, MN, USA} was applied for 15 seconds, and then rinsed with air-water spray from dental three-way syringe and air dried.

GROUP 3 – One step adhesive {3M ESPE, St. Paul, MN, USA} was applied for 15 seconds and gently air-blown, and light-curing done for 10 seconds.

GROUP 4 – Both phosphoric acid and one step adhesive were applied.

Specimens from each group were further divided into two subgroups, according to the different resin cement investigated in this study. Each subgroup contains eight specimens. In subgroup A cement used for bonding was RelyX U200 {3M ESPE, Neuss, Germany} and in subgroup B it was Maxcem Elite {Kerr, CA, USA} cement [Table/Fig-1]. Specimens were then immersed in distilled water for one week.

Relyx U200	Base paste – Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, Silanated Fillers, Initiator component. Catalyst paste – Methacrylate monomers, Alkaline Fillers, Silanated Fillers, Initiator component.
Maxcem-Elite	Base – Urethane dimethacrylate, fluroaluminosilicate glass. Catalyst paste – Bisphenol-A-diglycidyl methacrylate, Triethyleneglycoldimethacrylate, Glycerol dimethacrylatedihydrogen phosphate, Bariumaluminosilicate glass

[Table/Fig-1]: Composition of self-adhesive resin cements.

Tensile Bond Strength Test

Tensile bond strength was determined using universal testing machine at a cross head speed of 1mm/min. A tensile load was applied till rectangular metal block debonded. The load at break was noted and tensile bond strength in MPa was calculated by dividing the imposed force (N) at the time of fracture by the bonding area (30mm^2) using the following formula [23].

$$\text{Tensile bond strength (MPa)} = \frac{\text{Failure load (N)}}{\text{Bonded surface area (mm}^2\text{)}}$$

Results thus obtained were tabulated, compared and subjected to statistical analysis.

RESULTS

Statistical analysis was done with t-test, using one-way ANOVA & Post-Hoc test. Mean and standard deviation of the tensile bond strength are shown in [Table/Fig-2]. The maximum tensile bond strength occured in Group 4 (etchant and bonding agent), followed by Group 3 (bonding agent only) & Group 2 (etchant only) in both the subgroups. The least bond strength was shown by Control Group (Group 1) (that was statistically significant when compared with treatment group).

Mean bond strength was higher in case of Max-cem Elite resin cement as compared to RelyX U200 resin cement regardless of surface treatment. When comparisons were made between the corresponding groups of both cements by t-test the results were non–significant (p > 0.05). [Table/Fig-3].

	Group 1 (Conventional)	Group 2 (Etching)	Group 3 (Bonding agent)	Group 4 (Etch + Bonding agent)
Subgroup A (Relyx U200)	1.33(0.32)	2.19(0.18)	2.24(0.47)	2.72(0.43)
Subgroup B (Max-cem elite)	1.59(0.299)	2.38(0.27)	2.49(0.16)	2.97(0.19)

[Table/Fig-2]: Tensile bond strength in MPa (standard deviation) of tested adhesive cements with different surface treatment of enamel

Group	Subgroup	N	Mean	Std. Deviation	Std. Error Mean
1	Subgroup A	8	1.335027	.3226287	.1140665
	Subgroup B	8	1.598569	.2997434	.1059753
2	Subgroup A	8	2.196094	.1866701	.0659978
	Subgroup B	8	2.383204	.2728289	.0964596
4	Subgroup A	8	2.726172	.4329330	.1530649
	Subgroup B	8	2.971485	.1915651	.0677285
3	Subgroup A	8	2.248829	.4711528	.1665777
	Subgroup B	8	2.491147	.1622796	.0573745

	t-test for Equality of Means						
						95% Confidence Interval of the Difference	
Group	T	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	Lower	Upper
1	-1.693	14	.113	-.2635421	.1556982	-.5974815	.0703972
2	-1.601	14	.132	-.1871094	.1168767	-.4377850	.0635663
4	-1.466	14	.165	-.2453134	.1673799	-.6043075	.1136808
3	-1.375	14	.191	-.2423175	.1761816	-.6201895	.1355545

[Table/Fig-3]: Tests-Multiple comparisons for tensile bond strength (MPa) for corresponding sub groups of different group (T-Test)

Surface treatment of enamel (Group 2,3,4) significantly improved the tensile bond strength compared with control (Group1) ($p<0.001$). Combined use of etchant & bonding agent (Group 4) significantly ($p<0.05$) improved bond strength than using etchant (group 2) and bonding agent alone (Group 3) except in group 3 of subgroup A. However, when the Group 2 (etchant) was compared with Group 3 (bonding agent) the results were insignificant ($p>0.05$) [Table/Fig-4,5].

Sub-group	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Sub-group A	1	2	-.8610676(*)	.1851268	.000	-1.366521	-.355614
		3	-.9138026(*)	.1851268	.000	-1.419256	-.408349
		4	-1.3911454(*)	.1851268	.000	-1.896599	-.885692
	2	1	.8610676(*)	.1851268	.000	.355614	1.366521
		3	-.0527350	.1851268	.992	-.558189	.452719
		4	-.5300778(*)	.1851268	.037	-1.035532	-.024624
	4	1	1.3911454(*)	.1851268	.000	.885692	1.896599
		2	.5300778(*)	.1851268	.037	.024624	1.035532
		3	.4773428	.1851268	.069	-.028111	.982797
	3	1	.9138026(*)	.1851268	.000	.408349	1.419256
		2	.0527350	.1851268	.992	-.452719	.558189
		4	-.4773428	.1851268	.069	-.982797	.028111

[Table/Fig-4]: Post-Hoc Tests- Multiple comparisons for tensile bond strength (MPa) with and without surface treatments of dental enamel for sub Group A (RelyX U-200).
*The mean difference is significant at the .05 level.

DISCUSSION

Smear layer is defined as "any debris, calcific in nature, produced by reduction of dentin, enamel or cementum" [24,25]. This

Sub-group	Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
Sub-group B	1	2	-.7846349(*)	.1191935	.000	-1.110070	-.459199
		3	-.8925780(*)	.1191935	.000	-1.218013	-.567143
		4	-1.3729166(*)	.1191935	.000	-1.698352	-1.047481
	2	1	.7846349(*)	.1191935	.000	.459199	1.110070
		3	-.1079431	.1191935	.802	-.433379	.217492
		4	-.5882818(*)	.1191935	.000	-.913717	-.262846
	4	1	1.3729166(*)	.1191935	.000	1.047481	1.698352
		2	.5882818(*)	.1191935	.000	.262846	.913717
		3	.4803386(*)	.1191935	.002	.154903	.805774
	3	1	.8925780(*)	.1191935	.000	.567143	1.218013
		2	.1079431	.1191935	.802	-.217492	.433379
		4	-.4803386(*)	.1191935	.002	-.805774	-.154903

[Table/Fig- 5]: Post-Hoc Tests- Multiple comparisons for tensile bond strength (MPa) with and without surface treatments of dental enamel for sub group B (RelyX U-200) (Maxcem Elite).
*The mean difference is significant at the .05 level.

iatrogenically produced layer of debris interferes with bond formed between a tooth and the restorative material [26,27]. Bond strength is increased either by removal of the smear layer prior to bonding (etch-and-rinse, phosphoric acid etching), or by use of bonding agents that can penetrate beyond the smear layer and incorporate it into the bonding layer (self-etch, self-adhesive resin cements) [28]. With self-adhesive resin cements, the rationale is to superficially demineralize the enamel and simultaneously infiltrate the etched enamel with resin to create a resin reinforced hybrid layer [29].

When comparing the adhesive properties of adhesive luting cements it is very essential that all the factors which influence the retention of adhesive luting cement to tooth and restorative surface are standardized. The bond strength of adhesive luting cements to enamel and dentin varies [30]. The differences observed between bond strengths in enamel and dentin can be attributed to the differences in the composition between enamel and dentin substrates. Enamel is almost a completely mineralized tissue, whereas dentin is less mineralized, with organic matrix and dentinal tubules. Thus, adhesion to enamel is typically stronger and more stable than adhesion to dentin [31,32]. So, enamel surface of all the teeth were flattened in such a way to ensure that enamel was continuous and dentin was not exposed.

The differences in the bonding performances of adhesive luting agent's invivo and invitro can be contributed to the dehydration of in vitro specimens and presence of partially humid environment in vivo. Thus, the prepared tooth samples were stored in distilled water to prevent dehydration of tooth structure and to prevent contamination of tooth surface.

In the present study Nickel-Chromium casted rectangular blocks before cementation were sandblasted with 50 µm aluminium oxide to improve adhesion of cements to metal. O'Connor et al., found that the retention of cast restorations micro blasted with 50 µm Aluminium oxide and luted with different cements exhibited improved retention than non micro blasted cast restorations [33].

Acid etching before the application of self-adhesive resin cements resulted in significantly higher bond strength when compared with the control group (no surface treatment).

Phosphoric acid remove smear layer and demineralise or etch the enamel. Resultant enamel surface has high surface energy and surface area. The resin penetrated into these micro porosities, intimately adheres to form retentive resin tag, generating higher micromechanical retention [23,34]. The extent and depth of the etching patterns influence the bonding performance of an adhesive [35-37]. In contrast, in control group intact smear layer prevented the cement from reaching into deeper unaffected enamel.

The results of this study were in agreement with the studies by Duarte et al., and Lin et al., in which tensile bond strength was greater of etched enamel as compared with the control group [23,38]. Similar results were also noted by Turp et al., although they did their studies on dentin [39]. However, pH, pka, hydroxyapatite buffer potential, etching time, and orientation of enamel prisms may affect the demineralization and, consequently, the bond strengths [40-43]. Variation in the structure of surface enamel also can affect bond properties. Studies have shown that aprismatic enamel can negatively affect the bond strengths [17]. Bonding to the cervical enamel can generate significant lower bond strengths than bonding to mid coronal enamel, because of the aprismatic enamel layer [44]. Aprismatic enamel appears to be more acid-resistant than prismatic enamel [45].

Combined use of phosphoric acid and one step bonding agent resulted in significantly greater bond strength to enamel than control group, use of etchant and one step bonding agent alone. Phosphoric acid removed the mineral deposits (smear layer) and the bonding agent infiltrated the exposed enamel surface. In contrast, use of one step bonding agent alone resulted in only a moderate etching pattern, the adhesive did not infiltrate completely into the smear layer, and hence a thick adhesive layer was created [23].

When etchant was used alone it increases the enamel roughness. Resin cement may not infiltrate deeply into the micro porosity due to the viscosity of the resin cements when compared with bonding agent. This is supported by the study by Lin et al., in which the tensile bond strength was greater when phosphoric acid was used in combination with bonding agent [23].

The tensile bond strength was greater when one step bonding agent was applied to the enamel surface as compared with the control group (no surface treatment) for both the cements. Bonding agent cause demineralization of the enamel and adhesive flow into them and polymerised, so, bonding mechanism is micromechanical. 10-MDP (10-methylacryloyloxydecyl dihydrogen phosphate) chemically bond to the calcium in the hydroxyapatite, this could increase the overall bond strength [9].

Recently, Pashley et al., reported that contemporary self-etching systems could produce different etching patterns on unground enamel depending upon their respective aggressiveness [17]. Accordingly, self-etching systems could promote a mild, moderate or aggressive etching pattern on intact enamel, depending on the acidic dissociation constants (pka values) of their acidic monomers and the concentration of these monomers in the adhesives.

This was supported by Vaz et al., in which application of bonding agent increases the bond strength as compared with untreated enamel [46]. Similar results were reported by Lin et al., in which application of G-bond results in higher bond strength as compared with untreated enamel surface [23].

No significant difference in the tensile bond strength was observed among RelyX U-200 and Max-cem Elite resin cements. These findings showed that the different composition and solvent of self-adhesive resin cements did not affect the bond strength. Similar results were reported by André et al., and Lin et al., in which no significant differences were observed among the different self-adhesive resin cements [23,47]. However, André et al did their studies on dentin [47].

LIMITATION

This invitro study provide important information, however, they do not replace clinical trials. Scanning Electron Microscopy (SEM) evaluation of fractured surfaces was also not done. Additional invitro studies will be required to evaluate the durability of the formed bond after simulated aging. Nowdays microtensile bond strength, shear bond strength and SEM examination are frequently used. Therefore, further studies evaluating longevity of resin bonds created in dental treatment, effect of various surface treatments for tooth and restoration surface, bond strength to dentin and ultra-

morphological features of bonding interfaces created by such newly available products are required.

Clinical Implications

The greatest advantage of self-adhesive cements is the easy and fast application technique. But this time saving technique is not as effective as self-etch/total etch resin luting agents. Surface treatment of tooth surface with phosphoric acid and/or boding agent enhances the bonding of cement to tooth restorative surface. So, the benefit of time saving with self-adhesive cements occurs at the expense of bond strength.

In clinic total etch technique should be used with self-adhesive resin cements, even if it defeats the principle of self-adhesiveness and the user-friendliness of such materials.

CONCLUSION

The pre-treatment of enamel with etchant &/or single step bonding agent before the application of self-adhesive resin cements improves the tensile bond strength. Tensile bond strength was insignificantly higher in case of Max-cem Elite resin cement as compared to RelyX U200 regardless of surface treatment.

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PARTICULARS OF CONTRIBUTORS:
1. PG Student, Department of Prosthodontics, Maharishi Markandey College of Dental Sciences and Research, Ambala, Haryana, India.
2. Professor, Department of Prosthodontics, Maharishi Markandey College of dental sciences and Research, Ambala, Haryana, India.
3. Professor and Head, Department of Prosthodontics, Maharishi Markandey College of dental sciences and Research, Ambala, Haryana, India.

NAME, ADDRESS, E-MAIL ID OF THE CORRESPONDING AUTHOR:
Dr. Sanjeev Mittal,
MMCDSR, Mullana, Ambala, Haryana, India.
E-mail: ambaladental@yahoo.com

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