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Research Article

Comparative Evaluation of the Shear Bond Strength and Debonding Properties of a New Self Etch Adhesive Composite Cement and Self Etching Primer Composite System Used for Orthodontic Bracket bonding - An *In Vitro* Study

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Abstract

Background of the Study: Over the last 50 years the bonding of various adhesives to enamel and dentin has developed a niche in nearly all areas of dentistry, including orthodontics. Combining conditioning and priming into a single step improves bonding time and cost-effectiveness to the clinician and Orthodontists use the acid-etch bonding technique to attach brackets to the enamel surface. Maintaining a sound, unblemished enamel surface after debonding the brackets is a primary concern to the clinician. Self-adhesive cements were introduced in 2002 as a new subgroup of resin cements. Self-adhesive cements do not require any pre treatment of the tooth surface. Once the cement is mixed, its application is a single clinical step. Sufficient research is not available with regard to whether self etching adhesives can be satisfactorily used in the field of orthodontics to bond the brackets. Maxcem Elite Chroma (Kerr Dental) is a self-etch/self-adhesive resin cement with a Colour Cleanup Indicator. The use of Maxcem Elite Chroma in the field of orthodontics is rather unexplored. Its basic properties such as SBS, the debonding properties and its effect on enamel demineralization need to be evaluated to commend its use for bonding in orthodontics.

Aims and Objective: Comparative evaluation of the shear bond strength and debonding properties of self etch adhesive composite cement (Maxcem Elite Croma) versus self etching primer composite system ((Transbond Plus SEP, 3M Unitek, Monrovia, Calif) used for orthodontic bracket bonding.

Materials and Methods: This was an *in vitro* study, conducted in controlled laboratory settings. Forty extracted human premolars extracted for purpose of orthodontic treatment. 20 premolars bonded with orthodontic brackets using the self-etch primer Transbond Plus (3M Unitek) containing both the acid and the primer by following manufacturer's recommendation and 20 premolars bonded with orthodontic brackets using the self-etching composite adhesive (Maxcem Elite™ Chroma Self-etch/Self-adhesive Resin Cement) cement by following manufacturer's recommendation). Results of continuous measurements were presented as mean ± SD. Significance was assessed at 5% level of significance at 95% confident interval. Mann Whitney U test was used to compare the shear bond strength of self-etching composite adhesive to self etch primer). Chi square test was used to evaluate the differences in ARI scores among both the groups. Chi square test showed that there were statistically significant differences among the groups ($p < 0.001$).

Results: Maxcem elite chroma exhibited the highest mean shear bond strength of 25.7 MPa and Transbond Plus (3M Unitek, Monrovia, CA, USA) exhibited the mean shear bond strength of 18.7 MPA. Maxcem elite chroma exhibited the highest ARI index thereby demonstrating its better debonding properties.

Conclusion: Within the limitations of the current study we concluded that: 1. New self-etch, self-adhesive bonding system surprisingly showed high SBS as compared to self etch primer, 2. It also demonstrated a high ARI scores on the bracket surface which signifies that the debonding takes place mostly at tooth resin interface thereby reduces the chances of deleterious effects like crazing of enamel surface from debonding forces.

Keywords: Bonding; Shear Bond Strength; Adhesive Remanent Index; Self Etch Bonding Agent; Self Etch Adhesive Resin Cements

Introduction

Over the last 50 years the bonding of various adhesives to enamel and dentin has developed a niche in nearly all areas of dentistry, including orthodontics.

Adhesion currently encompasses a combination of mechanical, adsorption, diffusion, and electrostatic phenomena [1]. Conventional adhesive systems use three different agents-an enamel conditioner, a primer solution, and an adhesive resin to bond orthodontic brackets to enamel.

A unique characteristic of some bonding systems in operative dentistry is that they combine the conditioning and priming agents into a single acidic primer solution for simultaneous use on both enamel and dentin [2,3]. Combining conditioning and priming into a single step improves bonding time and cost-effectiveness to the clinician and indirectly to the patient. These systems were used originally on dentin [3,4]. Essentially, the acidic part of the primer dissolves the smear layer and incorporates it into the mixture. Acidic primer solutions also demineralise the dentin and encapsulate the collagen fibres and hydroxyapatite crystals [5]. This simultaneous conditioning and priming allows penetration of the monomer into the dentin. The adhesive resin component will then diffuse into the primed dentin, producing a "hybrid layer [5]. While these conditioners were initially developed for use on dentin, studies have determined that adhesive systems combining conditioning and priming were also found to be effective when bonding to enamel [6].

Orthodontists use the acid-etch bonding technique to attach brackets to the enamel surface. Maintaining a sound, unblemished enamel surface after debonding the brackets is a primary concern to the clinician. As a result, bond failure at the bracket-adhesive interface or within the adhesive is more desirable (safer) than at the adhesive-enamel interface, because enamel fracture and crazing have been reported at bracket debonding especially with ceramic brackets [7]. It is possible that the depth of the etched enamel surface created by phosphoric acid may be a factor contributing to the incidence of enamel fracture [8-10]. Therefore, alternative enamel conditioners, such as maleic acid, and the newly introduced acidic primers that contain phenyl P may be beneficial, if they can maintain clinically useful orthodontic bracket bond strength while decreasing the depth of enamel dissolution.

Until recently, resin cements were divided into these two subgroups according to the adhesive system used to prepare the tooth prior to cementation. One group utilizes etch-and rinse adhesive in the other group, enamel and dentin are prepared using self-etching primers.

Self-adhesive cements were introduced in 2002 as a new subgroup of resin cements. These materials were designed with the intent to overcome some of the shortcomings of both conventional (zinc phosphate, polycarboxylate, and glass-ionomer cements) and resin cements, as well as to bring the favourable characteristics of different cement classes into a single product.

Self-adhesive cements do not require any pre treatment of the tooth surface. Once the cement is mixed, its application is a single clinical step, similar to the application procedures of zinc-phosphate and polycarboxylate cements. These are still relatively new and detailed information on their composition and adhesive properties is limited. Although the basic adhesion mechanism appears to be the same for all self-adhesive cements, its multifunctional monomers with phosphoric acid groups simultaneously demineralise and infiltrate enamel and dentin. The dominant setting reaction is the radical polymerization that can be initiated by light exposure or through the self-curing mechanism. As the smear layer is not removed, no postoperative sensitivity is expected. Self-adhesive cements are claimed to be moisture tolerant.

It has been demonstrated that shear bond strengths (SBSs) of brackets bonded using different "self-etch" primers (SEPs) were not significantly different from those associated with brackets bonded with the conventional acid-etch technique [9,11]. It has also been concluded that SEPs that produce a minimal etch pattern can still provide adequate bracket SBS [12]. Additionally, it was observed that when using a self-etching adhesive to bond brackets, no significant difference was seen when the SEP was cured either in separate steps or simultaneously with the bonding adhesive.

Sufficient research is not available with regard to whether self etching adhesives can be satisfactorily used in the field of orthodontics to bond the brackets. The SBS, the debonding properties and its effect on enamel demineralization have been documented rather elusively.

Maxcem Elite Chroma (Kerr Dental) is a self-etch/self-adhesive resin cement with a Colour Cleanup Indicator, to tell the optimal window of time to clean up excess cement. The use of Maxcem Elite Chroma in the field of orthodontics is rather unexplored. Its basic

properties such as SBS, the debonding properties and its effect on enamel demineralization need to be evaluated to commend its use for bonding in orthodontics.

Objectives of the Study

The objectives of the current study are:

1. To evaluate the shear bond strengths (SBSs) of self-adhesive resin materials and compare them with self etch primer composite system.
2. To evaluate the ARI index of self-adhesive resin materials and compare them with self etch primer composite system.

Materials and Methods

This was an *in vitro* study, conducted in controlled laboratory settings. The materials used were:

1. Forty extracted human premolars extracted for purpose of orthodontic treatment
2. Thymol 0.1%
3. Pumice
4. Polishing cups
5. Orthodontic premolar metal bracket (Ortho Organizers, MBT 0.022, Carlsbad, USA)
6. Self etch primer Transbond Plus (3M Unitek, Monrovia, CA, USA)
7. Self etching adhesive (Maxcem Elite™ Chroma Self-etch/Self-adhesive Resin Cement)
8. LED curing unit (Coltene S.P.E.C.® **3**)
9. Plastic (PVC) rings for mounting premolars
10. Mounting jig
11. MecMesin (Intelligent Load Unit) Universal testing machine
12. Optical microscope (Olympus BX51).

Method of collection of data/methodology

In this study, the experimental model consisted of 1 control group and 1 experimental groups:

- **Group 1: Control group:** 20 premolars bonded with orthodontic brackets (Ortho Organizers, MBT 0.022, Carlsbad, USA) using the self-etch primer Transbond Plus (3M Unitek) containing both the acid and the primer by following manufacturer's recommendation.
- **Group 2: Experimental group:** 20 premolars bonded with orthodontic brackets (Ortho Organizers, MBT 0.022, Carlsbad, USA) using the self-etching composite adhesive (Maxcem Elite™ Chroma Self-etch/Self-adhesive Resin Cement) cement by following manufacturer's recommendation.

Sample size estimation

Analysis: A priori: Compute required sample size
Input: Tail(s) = One
Effect size d = 0.82
α err prob = 0.05
Power (1-β err prob) = 0.80
Allocation ratio N2/N1 = 1.

Output: Noncentrality parameter δ = 2.5930677
Critical t = 1.6859545
Df = 38
Sample size group 1 = 20
Sample size group 2 = 20
Total sample size = 40
Actual power = 0.8163646.

Methodology

Forty freshly extracted human premolars extracted for the purpose of orthodontic treatment were collected and stored in a solution of 0.1% (wt/vol) thymol.

Inclusion criteria

1. Extracted human premolar teeth extracted for the purpose of orthodontic treatment will be selected only if they have intact buccal enamel.

Exclusion criteria

2. Teeth having surface cracks from extraction forceps,
3. Teeth treated with chemical agents, and
4. Teeth having caries or restoration
5. Teeth undergone attrition.
6. Teeth previously not tested or bonded for any purposes.

Groups tested

Group 1 (control group)

Twenty premolar were bonded using the manufacturer's recommended protocol then is applied by continuously rubbing the self etch primer Transbond Plus (3M Unitek) on the enamel surface

for 3 - 5 seconds. The SEP is then lightly dried using compressed air for 1 - 2 seconds. Each pre coated bracket was placed on the tooth and held for 10 seconds. The force gauge can be used to help assure a uniform adhesive thickness between the bracket and enamel. The bracket was then light cured using a LED curing light for 20 seconds (10 seconds for each proximal side).

Group 2 (experimental group)

Twenty premolars were bonded, following the *m*anufacturer's recommendations, using the self-etchant adhesive (Maxcem Elite™ Chroma Self-etch/Self-adhesive Resin Cement). Maxcem Elite Chroma was applied on enamel surfaces, and bracket bases were bonded in place without separate enamel etching step before bonding. This self-etch, self adhesive resin cement was polymerized with LED curing light for 20 s each on both mesial and distal sides (i.e. 40s total).

Compositions of adhesive materials used in this study

Adhesive material	Manufacturer	Filler weight (%)	Composition
Transbond XT Light-cure adhesive with self etch primer	3M Unitek, Monrovia, CA, USA	80	35% phosphoric acid, silane-treated quartz, bisphenol A diglycidyl ether dimethacrylate, bisphenol A bis-(2-hydroxyethyl)-ether dimethacrylate, silane-treated silica
Maxcem Elite Chroma Self-etch, self-adhesive	Kerr Italia, 84018 Scafati, Italy	69	GPDM, methacrylate ester monomers, HEMA, 4 methoxyphenol, cumene hydroperoxide, titanium dioxide and pigments

GPDM: Glycerol Dimethacrylate Dihydrogen Phosphate; HEMA: 2-Hydroxyethyl Methacrylate; MEHQ: Monomethyl Ether Hydroquinone.

Determination of Bond Strength and De bonding procedure

Bond strength was determined by a universal testing machine (MecMesin Intelligent Load Unit) with 1 kN load cell at crosshead speed of 1 mm/minute. Each tooth was oriented with the testing device as a guide such that its labial surface was parallel to the force during the shear strength test. An occlusal-gingival load was applied to the bracket with a blade-end steel rod attached to the crosshead of the universal testing machine. This produced a shear force at the bracket-tooth interface. The load at bracket failure was recorded electronically in Newtons by a computer connected to the MecMesin machine. The SBS values were calculated in megapascal by dividing the force by the area of the bracket base (MPa = N/mm^2). The maximum load necessary to debond or initiate bracket fracture was recorded in Newton and then converted into megapascal as a ratio of Newtons to the bracket surface area. The bracket surface area of Ortho Organizer maxillary firstpremolar bracket is 10.27 mm^2.

Adhesive remnant index

After bond failure, the teeth and brackets were examined under 10x magnification. Any adhesive remaining after bracket removal was assessed using a modified Adhesive Remnant Index (ARI) and scored with respect to the amount of resin material adhering to the enamel surface. The ARI scale has a range between 5 and 1:

Score 0 = no adhesive left on bracket
Score 1 = less than 25% of adhesive left on bracket
Score 2 = 25% of adhesive left on bracket
Score 3 = 50% of adhesive left on bracket
Score 4 = 75% of adhesive left on bracket
Score 5 = 100% of adhesive left on bracket.

Results

Shear bond strength

Mean SBS values (in MPa) of the two test groups and their descriptive statistics are shown in table 1 and figure 1 respectively. Maxcem elite chroma exhibited the highest mean shear bond strength of 25.7 MPa and Transbond Plus (3M Unitek, Monrovia, CA, USA) exhibited the mean shear bond strength of 18.7 MPA. The difference found between the two groups was seen to be statistically significant ($p < 0.005$). Results of continuous measurements were presented as mean ± SD. Significance was assessed at 5% level of significance at 95% confident interval. Mann Whitney U test was used to compare the shear bond strength of self-etching composite adhesive to self etch primer.

ARI Scores

Table 2 shows ccomparison of ARI scores among the groups. Figure 2 shows the distributions of ARI scores, by percentage, for

	Shear bond strength [Mean ± SD]	Z	P
Self-etching composite adhesive (Experimental)	25.74 ± 1.96	-5.458	0.00*
Self-etch primer (Control)	18.77 ± 1.64		
Mann-Whitney U, *Statistically significant, p < 0.05			

Table 1: Comparison of shear bond strength of Self-etch primer and Self-etching composite adhesive.

Figure 1: Comparison of shear bond strength of Self-etch primer and Self-etching composite adhesive.

all the test groups. Chi square test showed that there were statistically significant differences among the groups (p < 0.001). Chi square test was used to evaluate the differences in ARI scores among both the groups.

This table shows that the mean shear bond strength of self-etch primer (18.77 ± 1.64) was less than self-etching composite adhesive (25.74 ± 1.96) and this difference was statistically significant (p = 0.00).

This table shows that majority (30%) of samples had score 3 followed by score 2 (22.5%) and score 4 (20%). In the experimental group, majority (35%) of samples had score 3 followed by score 4 (30%) and score 5 (20%). In the control group, majority (35%) of participants had score 2 followed by score 3 (25%) and score 1 (20%). However there was no significant difference between them (p = 0.08).

Discussion

The direct bonding of orthodontic brackets has revolutionized and advanced the clinical practice of orthodontics. However, there is a need to improve the bonding procedure by saving time and minimizing enamel loss without jeopardizing a clinically useful bond strength. Although recent bonding systems have been proven

Groups	ARI scores (%)								
	0	1	2	3	4	5	Total	Chi-square	P
Self-etching composite adhesive (Experimental)	0.0	2.5	5.0	17.5	15.0	10.0	50.0	9.711	0.08
Self-etch primer (Control)	2.5	10.0	17.5	12.5	5.0	2.5	50.0		
Total	2.5	12.5	22.5	30.0	20.0	12.5	100		
*Statistically significant, p < 0.05									

Table 2: Comparison of ARI scores among the groups.

Figure 2: Comparison of API scores among the groups.

reliable, improvements are still necessary to minimize technique sensitivity and reduce the chair time by decreasing the number of steps during the bonding procedure. Traditionally, using acid etchants followed by a primer was an essential part of the bonding procedure of composite adhesives, to allow good wetting and penetration of the sealant into the enamel surface [10,11]. The use of the new self adhesive resin cements for orthodontic purposes has not yet been fully evaluated. In general, they are thought to simplify the clinical handling of the adhesive systems by combining the etchant, primer and composite resin in 1 application. The present study evaluated 2 adhesive systems-Transbond Plus with a 2-component self-etch primer and Maxcem Elite Chroma self etching

Group	Score 0 (%)	Score 1 (%)	Score 2 (%)	Score 3 (%)	Score 4 (%)	Score 5 (%)
TXT (Control)	1 (5)	4 (20)	7 (35)	5 (25)	2 (10)	1 (5)
MEc (Experimental)	0 (0)	1 (5)	2 (10)	7 (35)	6 (30)	4 (20)

Table B

adhesive resin cement. The findings indicated that both bonding systems provided adequate shear bond strengths but Maxcem elite chroma exhibited the highest mean shear bond strength of 25.7 MPa and Transbond Plus exhibited the mean shear bond strength of 18.7 MPA. The difference found between the two groups was seen to be statistically significant ($p < 0.005$). This was an *in vitro* study, and care should be taken in comparing the results with those that might be obtained in the oral environment. In addition, more research is needed to determine the shear bond strength of these new self-etch adhesive resin cements over a longer time period-e.g. 24 hours and 1 week after bonding and after thermocycling.

Transbond™ plus self etching primer

Minimum bond strength for clinically acceptable bracket bonding was reportedly 6 - 8 MPa [12]. In the present study, Transbond XT self-etch resin materials showed clinically acceptable bond strengths for bracket bonding. It has been proved in many studies that the SBS of Transbond XT SEP is comparable to conventional Transbond XT. Transbond XT is a traditional composite resin filled with filler particles of diverse sizes [13] and which reportedly exhibited clinically acceptable bond strength values. Transbond XT was used as the gold standard in many SBS studies [12-42]. However, its SBS values varied widely between 8.9 ± 3.9 and 18.1 ± 5.5 MPa in literature. Mineral structures of the teeth used in these studies might have an effect on the SBS of different bonding agents, thus accounting for this large disparity in the SBS data of Transbond XT. Secondly, pressure exerted by the handpiece instrument during polishing might have an effect on tooth surface features, which would then affect the SBS of adhesive materials and cause differences in the SBS of Transbond XT among different studies.

Phosphoric acid etchant versus self-etch adhesive systems

Pre-treatment with 37% phosphoric acid increases bond strength [43,44] because thick outer enamel layer may prevent the permeation of self-etch primers and bonding agents, thus leaving some areas partially unetched. This then results in formation of shorter and poorly defined resin tags. By removing the outer enamel with phosphoric acid etching, longer resin tags are formed and thus bond strength is increased [45]. On the other hand, self-etch primers and bonding agents have some advantages over the phosphoric acid etchant. Self-etch primers simplify the clinical handling of adhesive systems by combining the conditioning and priming agents into a single product[4,5]. Self-etch bonding agents prevent aggressive decalcification and bulk enamel loss which are characteristics of phosphoric acid etching [46]. This means that they reduce the risk of enamel damage due to their reduced ability to sufficiently etch and penetrate the enamel surface [47]. Most self-etch adhesives did not etch enamel as deeply as the phosphoric acid etchants did, but the shallow etching pattern compromised bonding to enamel [48]. Demineralization effects of self-etch primers and bonding agents are proportional to their acidity [49]. The lower the pH, the higher the acidity, the deeper the etch; and the pH values of self-etch adhesive systems are higher than that of phosphoric acid etchant. Self-etch adhesive systems produce high bond strengths to human coronal dentin and ground enamel surfaces [50]. With enamel, phosphoric acid pre treatment improved the bond strength; with dentin, prior acid etching was detrimental to dentin bonding effectiveness and should be avoided [53,54]. Self-etch adhesives have high viscosity, which causes an incomplete resin decrease in its bracket retentive ability.

Maxcem elite chroma

Self-adhesive resin cements were developed to reduce the number of application steps and technique sensitivity associated with conventional resin cements. Maxcem Elite is a two-paste, dual-cure resin cement which combines the conditioning, priming, and adhesive agents into a single application. Maxcem Elite contains a proprietary redox initiator system: an efficient dual-cure mechanism that allows the resin to set quickly in the absence of light curing. Moreover, this proprietary redox initiator system eliminates the inherent discoloration of BPO/tertiary amine initiator systems for more esthetic restorations. However, when compared to conventional resin cements, self-adhesive resins reportedly provided inferior bonding to both enamel [52]. Maxcem Elite chroma has a colour indicator added to its predecessor Maxcem Elite. The colour indicator is pink when two solutions are mixed and as the material goes into initial set or gel phase, the pink colour fades away and turns colourless indicating optimal time to remove any excess cement. This property is particularly beneficial for bracket bonding in orthodontics, as it tells us the time to remove flash without disturbing the set bracket position significantly. After removing the flash the composite can be command cured by light cure devise.

Bracket bond failure

Bond failure at bracket-adhesive interface, rather than at enamel-adhesive interface, is caused by the low flexural strength of composite resins. Brackets bonded to enamel surfaces are temporary: they shall be removed after active treatment. Therefore, the primary orthodontic goal is to maintain sound, unblemished enamel surfaces after debonding. Enamel damage can be caused by phosphoric acid etching, bracket removal or cleaning of the teeth after debonding. Phosphoric acid etching complicates the removal of residual adhesive on the enamel after debonding, subsequently leading to surface scratches, cracking, and loss of sound enamel [48].

ARI score "5" means that bond failure site is at adhesive-enamel interface, while score "0" means that detachment occurs at adhesive-bracket base interface. In the present study, statistically significant differences were found in the ARI scores among the groups. ARI scores of 3 and 4 were predominantly seen in Maxcem Elite. This meant that when phosphoric acid etching was omitted, interfacial bonding between enamel and adhesive became the weakest link. However, when these adhesive materials were applied to etched enamel with an SEP, the ARI decreased. From the perspective of cleaning up the teeth after debonding, it is advantageous and preferred for residual adhesive to remain at bracket bases. This makes cleaning of the enamel surfaces easier and faster, with less risk of causing damage to the enamel [51]. In the present study, Maxcem Elite yielded the highest SBS and higher incidences of ARI scores of 3 and 4 that is, a lesser amount of residual adhesive remained on the teeth. If adequate shear bond strength is attained, benefits of low ARI scores outweigh those of high ARI scores: detachment at enamel-adhesive interface lowers the risk of enamel damage and hence the probability of enamel cracks formation. But if both are achieved, that is, high SBS and high ARI like it was seen in maxcem elite group, it is highly desirable for an orthodontic adhesive. Hence it was demonstrated that Maxcem Elite chroma not only has an adequate SBS but also has a favourable debonding characteristics and that it can be considered as an alternative to total etch technique or self etch technique for bonding of brackets in orthodontics.

Conclusion

The purpose of this *in vitro* study was to evaluate of the shear bond strength and debonding properties of self etch adhesive composite cement (Maxcem Elite Croma) and self etching primer composite system ((Transbond Plus SEP, 3M Unitek, Monrovia, Calif) used for orthodontic bracket bonding. The self etch adhesive resin cements eliminates the need of separate etching and priming thereby reducing the operating time by a significant amount. Sufficient research is not available with regard to whether self etching adhesives can be satisfactorily used in the field of orthodontics to bond the brackets. The SBS, the debonding properties and its effect on enamel demineralization have been documented rather elusively.

Hence the current study was conducted to evaluate the same.

The conclusions from this study are as follows:

1. New self-etch, self-adhesive bonding system surprisingly showed high SBS as compared to self etch primer.
2. It also demonstrated a high ARI scores on the bracket surface which signifies that the debonding takes place mostly at tooth resin interface thereby reduces the chances of deleterious effects like crazing of enamel surface from debonding forces.
3. Both of the above properties are desirable for orthodontic bracket bonding and hence it can be considered as an alternative to traditional bonding systems.

Within the limitations of this *in vitro* study, we drew the above mentioned conclusion. However, further *in vitro and in vivo* studies of these self-etch, self adhesive bonding systems are needed to obtain data and findings useful and important for clinical practice which can be focussed to demonstrate following:

1. The effects of mineral structure of teeth and handpiece pressure on the SBS of different adhesive materials.
2. The effect of aging on the SBS of this new adhesive which can be checked for both *in vivo* and *in vitro* environments.
3. The surface characteristics of enamel before and after debonding when the self-etch, self adhesive bonding systems were used.

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