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EFFECT OF POLYACRYLIC ACID PRETREATMENT ON THE MICROTENSILE BOND STRENGTH OF SELF-ADHESIVE RESIN CEMENT AND RESIN MODIFIED GLASS IONOMER CEMENT TO DENTIN

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ABSTRACT

Aim of the study: to evaluate in vitro the effect of dentin surface pretreatment with polyacrylic acid on the microtensile bond strength (μTBS) of self-adhesive resin cements and resin modified glass ionomer luting cements (RMGIC).

Materials and Methods: The occlusal enamel of 16 teeth was removed perpendicular to the long axis of teeth to expose flat dentin surface at a standardized depth which is 1mm apical to DEJ. The teeth were randomly divided into 4 groups according to cementation protocol of indirect resin composite blocks namely; RelyX-Unicem with no pretreatment, RelyX-Unicem with polyacrylic acid pretreatment, FujiCEM (RMGIC) with no pretreatment and FujiCEM (RMGIC) with polyacrylic acid pretreatment. The restored teeth were mounted on the cutting machine, sectioned into a series of 1 mm^2 thick. The sticks were stressed to failure under tension using Universal Testing Machine to record the microtensile bond strength. The collected data were submitted to ANOVA and Tukey's test.

Results: The highest mean value was detected in RelyX-Unicem with no pretreatment, followed by Fujicem with polyacrylic acid pretreatment; whereas the lowest value was recorded in Fujicem with no pretreatment. Using two ways ANOVA revealed that material type had a statistically significant effect ($p=0.003$), with a higher mean value in RelyX-Unicem. However, pretreatment had a non-significant effect ($p=0.510$). The interaction of the two variables had a statistically significant effect ($p<0.0001$).

Conclusion: Polyacrylic acid adversely affects the microtensile bond strength of self-adhesive resin cement**;** however it improves bond strength of RMGIC.

KEY WORDS: dentin, microtensile bond strength, self-adhesive resin cements, RMGIC, polyacrylic acid

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INTRODUCTION

Adequate adhesion between restorations and teeth is one of the major factors of successful indirect restoration [1]. An ideal luting cement must provide sustainable bonds with different materials, sufficient compression and tensile strengths, wettability, and resistance to dissolution in the oral cavity [2]. There are various types of cement: zinc phosphate cement (ZPC), poly carboxylate cement, glass ionomer cement, resin-modified glass ionomer cement (RMGIC), and resin cement [3]. Resin cement has better compressive and tensile strengths, toughness, resilience, and extremely lower solubility than other luting agents. Moreover, it is aesthetically excellent and provides several color option [4].

Currently, most resin cements used for cementation of indirect esthetic restoration were either etch-and rinse or self-etch adhesive in combination with a low-viscosity dual polymerizing resin cement [5]. However, this multi-step bonding procedure is complex, technique sensitive, and it involves significant chair time. A newer generation of self-adhesive resin cements has been developed that eliminates the need for etching, priming, and bonding as separate steps. However, in spite of being easier to apply, it is important for these self-adhesive materials be capable of bonding adequately to both the dental structures and restorative material. Some studies have shown that the self-adhesive resin cements interact superficially with the enamel and dentin, and that these materials have lower bond strength to dental substrates when compared with the conventional adhesive luting technique due to that these materials basically bond to the smear layer [6, 7].

Glass ionomer cements (GICs) were introduced into dentistry in 1972 [8]. They offer many advantages, including the ability to chemical adhere to enamel and dentine, resistance to microleakage, good marginal integrity, dimensional stability at high humidity, similar coefficient of thermal expansion as tooth structures, biocompatibility, fluoride release, less shrinkage than resins upon setting and no release of free monomers [9]. However, the disadvantages of GICs, including moisture sensitivity and low tensile strength, render them less favorable for use as luting cements in indirect esthetic restorations where high dislodging stresses are encountered during function. Resin-modified glass ionomer cements (RMGICs) overcome these limitations by having more favorable mechanical properties, decreased moisture sensitivity and extended working time. Moreover, RMGIC adhesives can bond to tooth structure via two mechanisms: chemically through ionic bonding of the carboxyl group to the calcium ions of the tooth substrate and the resinous component can interlock with the conditioned tooth surface via a 'micro-mechanical adhesive mechanism'[10].

In an attempt to increase the bond strength between resin cement and tooth surfaces, surface treatments with different conditioning agents have been suggested such as Chlorhexidine digluconate (CHX), tetracyclines or several desensitizing agents. Polyacrylic acid is a mild conditioning agent employed for cavity cleansing and surface conditioning in glass ionomer restorations [11]. In these restorations, polyacrylic acid promotes the formation of irregularities on the surface of the substrate, forming an intermediate layer that facilitates ion exchange between the glass ionomer matrix and the calcium and phosphate in the partially demineralized smear layer [12]. Furthermore, the carboxyl ions in the acid increase the cleaning power and wettability of the surface [13].

The glass-ionomer adhesive Fuji Bond LC (GC) performed equally as well as the two step self-etch adhesives. However, during bond strength testing, the glass-ionomer adhesive tended to fail in the glass-ionomer material itself rather than at the interface [14].The latter two systems did not perform significantly differently from each other. Again, the significantly least favorable μTBS results were recorded for one-step self-etch adhesives, and the μTBS of these adhesives was not significantly different from that of the resin-modified glass-ionomer adhesive.

The aim of this study was to evaluate the effect of dentin surface pretreatment (no pretreatment vs. polyacrylic acid pretreatment) on the microtensile bond strength of self-adhesive resin cements and resin modified glass ionomer luting cements. The null hypothesis was that the dentin surface pretreatment with polyacrylic acid would increase μTBS of both self-adhesive resin cements to dentin

MATERIALS AND METHODS

Materials which are used in the present study have been illustrated in table (1)

Sample preparation

Sixteen molar teeth were selected for this study. All collected teeth were extracted for therapeutic reasons from patients of age group (35-45 years). The selected teeth were free of caries, cracks and hypoplastic defects. The selected teeth were thoroughly cleaned from calculus, tissue deposits, polished with pumice and rotating brush at conventional speed. The teeth were stored in saline solution at 4^{o}c for not more than one month.

Acrylic resin blocks were fabricated using a specially designed cylindrical, split Teflon mould for holding teeth. Each tooth was vertically embedded into self-curing acrylic resin *(Acrostone Dental Factor, England*) up to the level of the cervical line at CEJ.

TABLE (1) Materials used in the study.

Material	**Composition**	**Manufacturer**
Polyacrylic acid	11.5% Polyacrylic acid	Vitro Condicionador; Nova DFL, Rio de Janeiro, RJ, Brazil
RelyX Unicem (Shade A$_3$) A dual-cure self-adhesive universal resin cement	**Powder**: glass powder, initiator, silica substituted pyrimidine, Calcium hydroxide, peroxy compound and pigments. **Liquid:** Methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer and initiators. Inorganic fillers is 72 wt% and the grain particle size is <9.5μm.	**3M ESPE, St. Paul, MN, USA** **391896**
Fuji Cem 2 Self-cure, Resin modified glass ionomer luting cement	Paste A: fluoroalumino silicate glass, hydroxyethyl methacrylate, dimethacrylate, pigment, initiator. Paste B: polyacrylic acid, distilled water, silica powder, initiator	GC Corporation, Tokyo, Japan
Filtek Z350 Universal Restorative Material (Shade A3) Visible light cured nano-filled resin composite	**Organic part:** Bis-GMA, UDMA, TEGDMA and Bis-EMA resins. **Inorganic part:** Aggregated Zirconia/silica cluster with an average cluster size 0.6-1.4 micron with primary particle size of 5-20nm and a non-agglomerated/non-aggregated 20nm silica filler. The inorganic filler loading is78.5% by wt (59.5% by volume).	**3M ESPE, St. Paul, MN, USA** **152033**

Preparation of the indirect resin composite blocks

A specially constructed flat two halves split Teflon ring mould was used for the fabrication of indirect resin composite blocks. An external metal ring was used surrounding the two halves of the Teflon to keep the mould assembly. Cubical composite blocks (6x6mm in diameter and 4mm in thickness) were prepared in the space occupying the centre of Teflon mould using a visible light activated nano-filled restorative resin composite (**Filtek Z350 universal restorative).** The Teflon ring was placed on a clean microscopic glass slide, then the resin composite were incrementally packed using Teflon tipped instrument and light cured each increment of 2mm in thickness for 20 seconds using (Elipar LED Curing Light; 3MESPE), with irradiance of 1200 mW cm^2.

Before curing the final increment, a celluloid matrix strip and glass slide was pressed over the composite in order to have non porous and highly finished composite blocks. Resin composite blocks were further light cured for 20 seconds at each side after removal from split Teflon mould according to the manufacturer instruction **(3M ESPE)**.

Specimens grouping:

The teeth were divided into two main groups of 8 teeth each; according to the self-adhesive resin cement used namely; RelyX-Unicem self-adhesive and FujiCem adhesive system. Furthermore, each group was divided into two subgroups of 4 teeth each according to protocol of pretreatment either; no pretreatment or polyacrylic acid pretreatment.

Teeth preparation

The occlusal enamel of teeth was removed perpendicular to the long axis of teeth, to expose flat dentin surface at a standardized depth 1mm apical to DEJ. The occlusal tables were ground with a rotary grinding milling machine using #180-grit silicon carbide papers under continuous water coolant to create a uniform thickness of smear layer [15].

Dentin surfaces pretreatment

The teeth in the second group with polyacrylic acid surface pretreatments were manipulated according to manufacturer instruction.

The polyacrylic acid group was treated with a solution of 11.5% polyacrylic acid (Vitro Condicionador; Nova DFL, Rio de Janeiro, RJ, Brazil) for 20 sec with a moistened micro-brush in a scrubbing motion. Subsequently, the specimens were rinsed thoroughly for 20 sec and the dried with oil-free compressed air.

Cementation of the indirect resin blocks

Resin composite blocks were cemented to the dentin of all specimens using two different types of resin cements according to the manufacturer's instructions

For self-adhesive resin cement (RelyX Unicem), RelyX Unicem capsules were inserted in the Aplicap activator **(3M ESPE)** and activated for 2 seconds. The capsules were then mixed in a high-frequency rotary mixer **(Dentomax Compact, Degussa-Hüls AG, Germany)** for 10 seconds. The capsule was then inserted into gun, open nozzle and dispense cement directly onto the bonding surface of resin blocks which were seated in their place under 100 gm. standardized occlusal load. The excess cement was removed with scaler after initial light curing for 2 seconds. Each tooth surface was light cured for 40 second using Elipar LED Curing Light (3MESPE) according to the manufacturer's instructions to allow complete setting of cement.

For resin modified glass ionomer cement (FujiCEM RMGIC), Mix paste A and paste B for 10 s. The mixed cement was applied directly onto the bonding surface of resin blocks which were seated in their place under 100 gm. standardized occlusal load. The excess cement was removed with scaler and left for self-curing.

Beam preparation

After 24 hours from specimens' preparation and storage in distilled water at 37°c, mounting the tooth/composite block in the gripping attachment was done. The tooth/composite specimen was serially sectioned, using a 0.3-mm thick diamond coated disc (Buehler, IL, USA), at 2050 rpm; 8.8 mm/min feeding rate under copious coolant, mounted in an automated diamond saw (*Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA*). Serial sectioning was done in a bucco-lingual direction then rotated 90° clockwise and sectioned in a mesio-distal direction. Resultant beam were 0.9±0.1 mm in thickness and 3.5±1 mm in length. The four central sticks from each specimen were selected and their thickness was checked using a caliper

Microtensile bond strength measurement

For each tested subgroup, 16 beams were tested. Geraldeli's jig was used to mount beams onto the universal testing machine. Each beam was aligned in the central groove of the jig and glued in place by its ends using cyanoacrylate based glue (Zapit, DVA Inc, USA); The jig was in turn mounted into the universal testing machine *(Instron, MA, USA)* with a load cell of 500 N **.**Tensile load was applied, at a cross-head speed of 0.5 mm/min, until bonding failure of the specimen occurred. Micro Bond strength values were calculated in Mega Pascal *(Bluehill Lite software, Instron, MA, USA)***.**

Statistical analysis

One way analysis of variance (ANOVA) and Tukey's post hoc test were used to compare between groups. The significance level was set at $p<0.05$. Statistical analysis was performed with SPSS 16.0 (Statistical Package for Scientific Studies, SPSS, Inc., Chicago, IL, USA) for Windows.

RESULTS

The data in table (2) and figure (1) showed that the highest microtensile bond strength values were detected in Rely X Unicem with no pretreatment, followed by Fujicem with polyacrylic acid pretreatment; whereas the lowest value was recorded in Fujicem with no pretreatment. ANOVA test and Tukey's post hoc test revealed that mean values of Fujicem with no pretreatment and Rely X Unicem with Polyacrylic acid pretreatment were significantly different than the other two groups ($p<0.0001$).

The data presented in table (3) and figure (2) Using two ways ANOVA revealed that material type had a statistically significant effect ($p=0.003$), with a higher mean value in RelyX Unicem. However, pretreatment had a non-significant effect ($p=0.510$). The interaction of the two variables had a statistically significant effect ($p<0.0001$),

TABLE (2) Comparison of mean values in different subgroups (one way ANOVA test)

Groups	Mean	Std. Dev	Std. Error	Min	Max
Fujicem, no prett	6.5268^{d}	2.05417	.91865	4.57	8.89
Fujicem, polyacrylic acid	19.6243^{b}	4.06720	1.81891	14.25	25.21
Rely X Unicem, no prett	26.2629^{a}	4.68773	2.09642	19.23	31.39
Rely X Unicem, Polyacrylic acid	10.9962^{c}	3.02964	1.35490	6.93	13.93
F	29.94				
P value	< 0.0001*				

Significance level p<0.05, * significant
Tukey's post hoc test: means sharing the same superscript letter are not significantly different

TABLE (3) Effect of tested materials and treatment on the MTB strength of resin composite to dentin

Material	Mean	Std. Error	F	P value
Fujicem	13.076	1.139	11.884	.003*
Rely X Unicem	18.630	1.139		
No pre ttt	16.395	1.139	.453	.510ns
polyacrylic acid	15.310	1.139		
Interaction of both variables			77.484	.000*

Significance level p<0.05, * significant, ns=non-significant

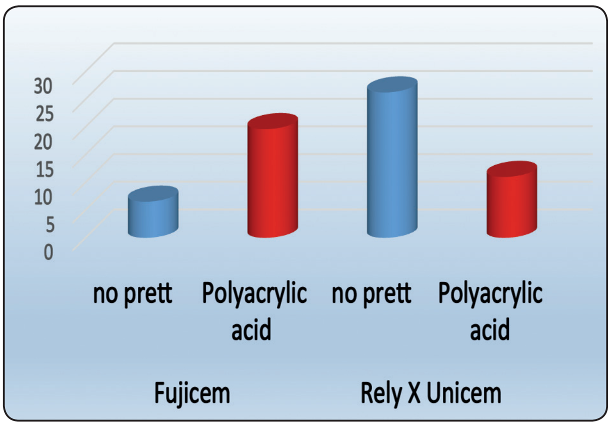

Fig (1) Column chart showing the mean Micro- tensile bond strength (MPa) values for different subgroups

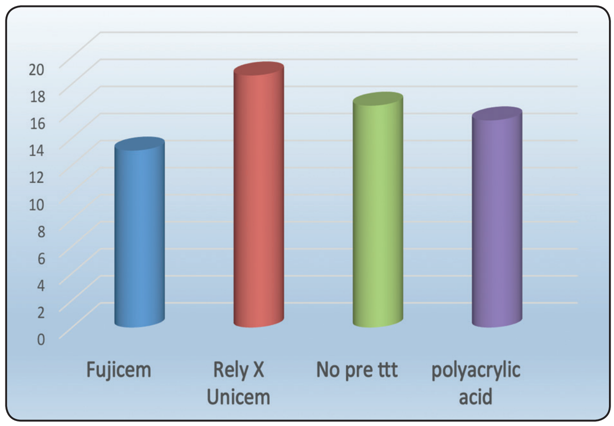

Fig (2) Column chart showing the mean Micro- tensile bond strength (MPa) values in different groups

DISCUSSION

This study compared the microtensile bond strengths of the self-adhesive resin cements to that of resin modified glass ionomer luting cements with and without dentin surface pretreatment with polyacrylic acid.

The self-adhesive resin cements are based on new monomer, filler, and initiator formulations. The acidic monomer replaces the previous three steps by combining the use of adhesive and cement into a single application. These multi-functional phosphate-based acidic methacrylates can react with the basic fillers in the luting cement and the hydroxyapatite of the hard tooth tissue [16]. Self-adhesive resin cements combine the high-strength and low-solubility advantages of resin cements with the characteristic ease of use of self-adhesive systems, making them highly attractive to the clinician.

Self-adhesive resin cements are composed of an acidic monomer (methacrylate phosphoric acid) that can partially remove and/or modify the smear layer, which enables penetration of the cement and produces micromechanical retention to the substrate [17]. This suggests that a certain chemical bond occurs through a chelating reaction between the acid monomers and the hydroxyapatite present in dental substrates [18].

RelyX Unicem is self-adhesive resin cement that consists of alkaline fillers and multifunctional phosphoric acid methacrylates, which are responsible for its self-etching. Goracci et al, in 2006[19] found that this material was unable to demineralize or dissolve the smear layer completely, no decalcification and infiltration of dentin occurred and no hybrid layer or resin tags were observe. Some reasons may be proposed for the limited capacity of the self-adhesive resin cements to diffuse and decalcify the underlying dentin effectively: (1) high viscosity, which may rapidly increase as an acid-base reaction; (2) a neutralization effect may occur during setting, since these chemical reactions involve water release and alkaline filler that may raise the pH level [20].

In 1995, a new category of adhesives was introduced named as 'RMGIC adhesive' [21]. RMGIC adhesives bond to the tooth structure via the traditional conditioning for RMGIC restorative materials, namely conditioning the dentine with polyacrylic acid (PAA) then washing and drying. The conditioning of the dentine removes the smear layer structure, except for smear plugs, partially demineralizes the dentine surface, and promotes the chemical reaction between GIC components and hydroxyapatite crystals in the tooth substrate [22]. Therefore, RMGIC adhesives can bond to tooth structure via two mechanisms: chemically through ionic bonding of the carboxyl group to the calcium ions of the tooth substrate5 and the resinous component can interlock with the conditioned tooth surface via a'micro-mechanical adhesive mechanism'[10]. In this study, FujiCEM (FJC) (GC Corporation, Tokyo, Japan), which is a RMGIC that does not require pretreatment, was used.

The null hypothesis that the dentin surface pretreatment with polyacrylic acid would increase μTBS of both self-adhesive resin cements to dentin was rejected for self-adhesive resin cement. While it was accepted for RMGIC

The bonding efficacy of RelyX Unicem deteriorated when the dentine surface was pretreated with polyacrylic acid application. The removal of the smear layer by polyacrylic acid promotes demineralization that could damage the interaction between the self-adhesive resin cement and the collagen network on the dentin surface. Although chemical bonding could have occurred between the cement and the smear layer due to the cement contain glass particles making them behave like glass ionomer, the interface between the unbonded smear layer and the underlying intact mineralized dentine remained the weakest link of the resin-dentine bond. This weakness might further deteriorate with the air drying of polyacrylic acid. These results were in accordance with El-sayed et al, 2013[23] and Mushashe etal, 2016 [24]. They stated that the self-adhesive cement requires more water in its interaction on the dental surface in order to achieve higher bond strength values. This finding may be explained by the fact that self-adhesive materials need an ionizing medium for the chemical reaction to get started. Thus, the presence of water on the enamel or dentin surface could create a better ionizing medium for the tested material, which would then increase the adhesive's ability to interact with enamel or dentin. These results in accordance with, where they found that microtensile bond strength of self-adhesive resin cement were negatively affected by surface pretreatment.

These results were in disagreement with Stona et al, 2013 [25]. This may be to the difference in application techniques where they remove excess acid with absorbent paper leaving a moist dentin surface.

This study verified the effect of 11.5% polyacrylic acid on dentin bonding to the resin modified glass ionomer cements application. This acid has been used in association with the glass ionomer cements with the aim of obtaining greater interaction of these cements with the dental substrate [10]. When polyacrylic acid is applied for 10 seconds, it removes the smear layer, keeping the smear plugs in the dentinal tubules. However, this type of etching is not so strong compared to dentin bonding to self-adhesive resin cements. These results were in accordance with Tonial et al, 2010 [26] and Hamama et al, 2014 [27]

The ANOVA results showed that RelyX Unicem have higher bond Strengths compared to FujiCEM when bonded to dentin. This may be due to slow initial cross-linking of RMGIC when they were self-polymerized. This result was in agreement with Sabatini et al, 2013 [28] where they observed lower bond strength of FujiCEM than the other self-adhesive resin cements tested due to the self-polymerizing setting reaction of RMGIC.

CONCLUSION

Under the circumstances of this study, it may be concluded that:

Polyacrylic acid adversely affects the microtensile bond strength of self-adhesive resin cement**;** however it improves bond strength of RMGIC.

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