In-vitro evaluation of bond strength of four self-etching cements

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ABSTRACT

Indirect restorations need to be attached with adhesive luting agents to prevent them from becoming dislodged and provide adequate marginal sealing. The aim of this study was to evaluate bond strength to dentin of self-etching resin cements. Materials and methods: 75 flat dentin surfaces were randomly distributed among 5 groups, according to cements used; Group 1: RelyxU100 (3M/ESPE), Group 2: Bis Cem (Bisco), Group 3: Max Cem (Kerr), Group 4: SeT PP (SDI) and Group 5: Relyx ARC (3M/ESPE), control. Ceramic test cylinders (IPS Empress 2 / Ivoclar-Vivadent) 4.1 mm across were prepared and attached to the dentin surfaces using the different cements. A constant 25N load was applied for 1 minute and they were light-polymerized. Following storage for 24 hours at 100% humidity and 37 °C, the specimens were tested for bond strength under shear strain in an Instron testing machine at a crosshead speed of 1 mm/minute; at 7, 14 and 21 days. Data were analyzed by ANOVA and Tukey's test. There were significant differences between materials (p < 0.0001), but not between times or material/time interaction. RelyX ARC (Control Group) had the highest bond strength (15.52 MPa). Among the self-etching cements, the best behavior was found for Relyx U100 (10.80MPa), followed by BisCem (6.36 MPa), MaxCem (5.45 MPa) and SeTPP (3.17 MPa). The bond strength of the self-adhesive cements evaluated was lower than that for resin cements which require previous treatment of the dental substrate (control group). This should be taken into account during clinical selection, in particular for tooth preparations with poor retention.

Key words: *Dental cements. Resin cements. Dental bonding.*

Evaluación in vitro de la resistencia adhesiva de cuatro cementos de autograbado

RESUMEN

Las restauraciones indirectas deben ser fijadas por medio de agentes cementantes, para evitar su desprendimiento y mantener un adecuado sellado marginal. El objetivo de este trabajo fue evaluar la resistencia adhesiva a dentina de los cementos resinosos de autograbado. Materiales y métodos: Se utilizaron 75 superficies planas de dentina, distribuidos aleatoriamente en 5 grupos, según los medios cementantes; GRUPO 1: RelyxU100 (3M/ESPE), GRUPO 2: Bis Cem (Bisco), GRUPO 3: Max Cem (Kerr), GRUPO 4: SeT PP (SDI) y GRUPO 5: Relyx ARC (3M/ESPE), control. Se confeccionaros probetas cilíndricas de cerámica (IPS Empress 2 / Ivoclar-Vivadent) de 4,1 mm de diámetro, las que fueron fijadas a las superficies de dentina mediante los distintos cementos. Se aplicó una carga constante de 25N durante 1 minuto y se realizó la fotopolimerización. Después de 24 horas de almacenamiento en un medio con 100 % de humedad a 37° C, las probetas fueron sometidas a ensayo de resistencia adhesiva bajo tensiones de corte en máquina Instron, con una velocidad del cabezal de 1 mm/minuto; a los 7, 14 y 21 días. Los datos fueron analizados mediante ANOVA y test de Tukey, mostrando diferencias significativas entre los materiales (p < 0,0001), no así respecto al factor tiempo ni a su interacción. El mayor valor de resistencia adhesiva lo presentó RelyX ARC (Grupo control) (15,52MPa), De los cementos autograbantes, el mejor comportamiento se obtuvo en Relyx U100 (10,80MPa), seguido por BisCem (6,36MPa), MaxCem (5,45MPa) y SeTPP (3,17MPa): Los cementos autoadhesivos evaluados presentaron menor resistencia adhesiva que los cementos resinosos que requieren tratamiento previo del sustrato dentario (grupo control). Esto debería ser considerado cuando se realiza su selección clínica, sobre todo en preparaciones dentarias poco retentivas.

Palabras clave: *cementos dentales, cementos resinosos, adhesivo dental.*

INTRODUCTION

When teeth lose their structural, functional and/or aesthetic integrity, they may be restored by inserting plastic or rigid materials. When much of the dental structure is lost, the use of plastic materials has well-known limitations, and partial or total rigid crown restorations (metal or aesthetic) are indicated. Rigid restorations must be bonded to the dental structures by luting agents to prevent them from becoming detached and provide adequate marginal sealing, ensuring their permanence in the oral cavity[1].

Despite the advantages of rigid restorations (resistance to abrasion, resistance to fractures, polymerization control, adequate contour anatomy and contact relationship[2]), bonding them to the tooth structure is still a challenge because this type of restoration doubles the adhesive interfaces: one interface at the tooth surface and the other at the surface of the restoration.

Society's aesthetic demand has motivated dental product manufacturers to develop metal-free restorative materials such as porcelain or compound resin onlays, veneers and crowns[3]. The possibility of restoring to each tooth not only form and function but also visual harmony between the restoration and the remaining tooth, in addition to the characteristics of its integration, make aesthetic onlays a valid alternative to plastic or cast metal restorations when there is significant destruction of tooth tissues, exceeding one third of intercuspal distance[4]. The preparations made for these restorations are more conservative, and regarding the bonding materials, the basic aim is to increase bond strength to the different substrates, i.e. it involves not only selecting a material with good bonding properties, but also proper preparation of the substrates to be bonded[5].

Bonding rigid restorations requires different mechanisms to ensure retention, such as the creation of opposing surfaces that can make close contact in order to achieve friction. This contact needs to be improved by interposing a liquid which in turn can be hardened by means of a chemical or physical mechanism – the luting agent[6].

The retention of restorations increases when there are more opposing surfaces, closer contact between parts and better performance of the cement regarding its mechanical properties and potential to achieve bonding (understanding bonding as micro-mechanical and/or chemical bonds between the liquid and the parts placed in contact)[7]. Proper selection and use of luting agent are extremely important, as many of the advantages of this type of material will be lost if an inadequate bonding system is used[8].

Resin cements on their own are not adhesive – they need to be supplemented with treatments of both the tooth surface and the restoration surface[1]. Dental tissues need to be acid-conditioned before placing an adhesive system which will bond micro-mechanically to the tooth and chemically to the luting agent[2,9]. This acid conditioning may be achieved independently with 37% phosphoric acid[10] or by using sixth- or seventh-generation self-etch adhesive systems which contain acidic monomers. The non-dental surfaces also need to be conditioned for luting in order to create micro-retentions to favor micro-mechanical bonding to the luting agents[2,8].

Adhesive self-etch resin cements that do not require treatment of the tooth structure were introduced on the dental market a few years ago[8,9,11]. They include monomers with phosphoric acid groups (phosphorylated methacrylates) that demineralize dental tissue and enable bonding, thereby simplifying the technique and saving time in the adhesive procedure[2,12].

Progress in the field of research, technological development and the advent of a wide range of self-etch adhesive cements motivated the current study, of which the aim is to ascertain whether self-etch adhesive cements provide one of the desirable properties for luting agents. Thus, the objective of the present work was to study *in vitro* the behavior of four self-etch adhesive cements regarding adhesion to the dentin structure and to compare them to a resin cement with classical bonding.

Table 1: Experimental materials, manufacturer and batch.

Group	Experimental	Manufacturer Material	Batch
1	RELYX U100	3M ESPE. DENTAL PRODUCTS. USA	424360
2	BISCEM	BISCO INC. USA	1100002087
3	MAXCEM	KERR CORPORATION. USA	3498965
4	SET PP	SDI. AUSTRALIA	S0905891
5	RELYX ARC	3M ESPE DENTAL PRODUCTS. USA	N166655

MATERIALS AND METHODS

This study evaluated the bond strength behavior of four self-etch resin cements compared to a conventional resin cement (described as conventional because it requires prior adhesive protocol including acid conditioning of tooth tissues before application). Seventy-five experimental specimens consisting of a ceramic test cylinder cemented to a dentin surface were prepared. Fifteen specimens were randomly assigned to each of the following groups: Group 1: RelyX U100 (3M Espe), Group 2: BisCem (Bisco), Group 3: MaxCem (Kerr), Group 4: seT PP (SDI), Group 5: RelyX ARC (3M Espe). The first four groups are self-etch resin cements, while Group 5 represents conventional cement and is the control group (Table 1).

The experimental specimens were prepared from 75 recently extracted non-carious human teeth (obtained from the Department of Surgery 2, School of Dentistry, Córdoba National University, with prior informed consent from patients). The teeth were stored in distilled water at 37 ºC until they were processed experimentally.

The occlusal surfaces of third molars were cut horizontally with a low-speed saw (Isomet Low Speed Saw. Buehler. USA. Department of Dental Biology, School of Dentistry, Córdoba National University), with teeth mounted on an *ad hoc* positioning device (Fig.1). The cut surfaces were polished using rotating discs of decreasing grain size under constant water cooling (metallographic polisher; A.B.O,) until flat, polished surfaces were obtained approximately 2 mm beyond the dentin-enamel junction. Once the surfaces were polished, the teeth were mounted in plastic shapers containing self-curing acrylic resin (Fig. 2) so that they could be attached to an *ad hoc* device for bond strength testing (Fig. 3).

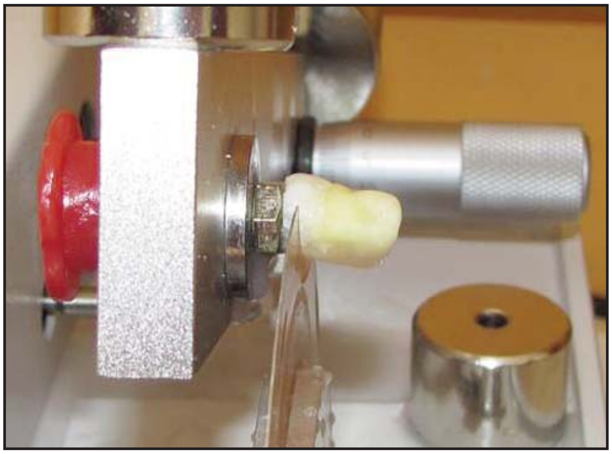

Fig. 1: Cross section of specimens.

Fig. 2: Specimens embedded in acrylic resin to be mounted on the universal testing machine.

The dentin surfaces in Groups 1 to 4, representing self-etch cements, received no prior treatment, but were simply washed with pressurized water and dried with cotton swabs, preserving dentin humidity. The dentin surfaces in Group 5 (control) were conditioned with 35% phosphoric acid gel (Scotch Bond Etchant, 3M Co.) for 15 seconds, washed with pressurized water for 15 seconds and dried for 5 seconds, after which the adhesive system (Single Bond 2, 3M Co) was applied, following the manufacturer's instructions.

Ceramic test cylinders (IPS Empress 2, Ivoclar-Vivadent) 4.1 millimeters in diameter were prepared (Eduardo Ruderman Dental Prosthetics Laboratory) (Fig. 4), to bond to the dentin surfaces using the different cements. The ceramic surfaces to be bonded were treated following manufacturer's instructions: etching with 10% hydrofluoric gel for 1 minute and silanization for 5 minutes, to complete evaporation (Monobond S, Vivadent.).

Fig. 3: Device for mounting on the universal testing machine.

Then the test cylinders were attached to the dentin surfaces using the different experimental self-etch resin cements (Groups 1 to 4) or the dual conventional cement (Group 5, control), strictly following the instructions of their respective manufacturers. The cement for each experimental group was placed on both the dentin surface and the ceramic surface. The dentin substrate was covered with an adhesive paper with a perforation of the same diameter as the ceramic test cylinders in order to limit the adhesion area (Fig. 5) and a constant 25N load was applied using a device for standardizing loads (Fig. 6). Any excess material was removed using a microbrush, and specimens were light-cured with a 540 mW/cm^2 halogen lamp (XL3000, 3M ESPE. USA) for 30 seconds each on vestibular and lingual aspects.

Once the ceramic test cylinders were attached, each specimen was stored in distilled water and kept at a constant temperature of 37 ºC until it was tested (Fig. 7). Specimens were tested mechanically under shear strain using a device specifically created for such purpose, mounted on a universal testing machine (Instron Corporation; Department of Dental Materials, School of Dentistry, Buenos Aires University), at 1mm/min crosshead speed.

In order to ascertain whether bond strength changed over time, the 15 specimens in each group were randomly assigned to subgroups of 5 specimens each to be tested at 7, 14 and 21 days.

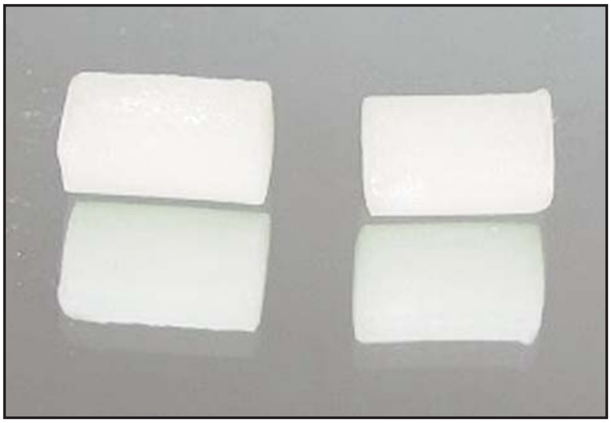

Fig. 4: Ceramic test cylinders made from IPS Empress 2 (Ivoclar Vivadent). (Ruderman Dental Prosthetics Laboratory).

Fig. 5: Adhesive paper with a perforation of the same diameter as the ceramic test cylinders covering tooth surface to limit the adhesion area.

Fig. 6: Ad-hoc standardizing device for cementing force, applying a constant 25N load.

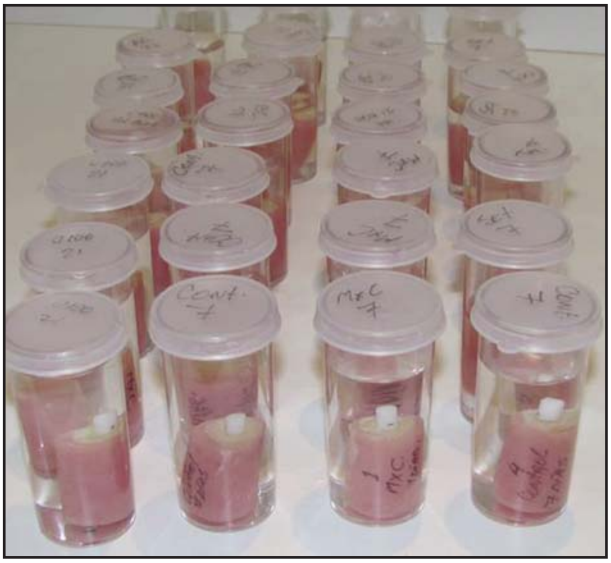

Fig. 7: Specimens immersed in distilled water until experimental processing.

Shear bond strength (MPa) was calculated from the relationship between shear load (N) and test cylinder surface area (mm). Data were recorded and analysis of variance and multiple comparison tests were performed to determine statistical significance.

RESULTS

Table 2 shows the mean values for bond strength in MegaPascals (MPa), standard deviation and confidence interval for each experimental material. RelyX ARC (control group) had the highest shear bond strength (15.52 MPa). The best behavior among self-adhesive cements was for RelyX U100 (10.08 MPa), with BisCem in second place (6.36 MPa) and MaxCem in third place (5.45 MPa), while SeT PP had the lowest bond strength (3.17 MPa).

Analysis of Variance (Table 3) showed no statistically significant difference (p< 0.0001) among the study materials. No difference in bond strength was found regarding time or material/time interaction.

Tukey's multiple comparisons test (Table 4) showed significant differences between materials, except for MaxCem and BisCem, which did not differ from each other, though they did differ from the rest.

The adhesive surfaces were analyzed after detachment of the ceramic test cylinders by observation under stereomicroscope at x20 magnification. Failure mode in bonding for all self-etch cements studied was adhesive, i.e., between cement and dentin.

Fig.8 shows the behavior of the different cements evaluated from the standpoint of bond strength at the three study times.

Table 2: Means, standard deviation and confidence interval (95%).

Group	Material	Means	S.D.	LL (95%)	LSD (95%)
1	RelyX U100	10.80	2.37	9.48	12.11
2	BisCem	6.36	1.70	5.42	7.30
3	MaxCem	5.45	1.36	4.69	6.20
4	seT PP	3.17	0.73	2.76	3.57
5	RelyX ARC	15.52	2.70	14.02	17.01

Table 3: Analysis of Variance.

Sources of variation	Sums of Squares	Degrees of Freedom	Mean Squares	F	p-value
Model	1479.51	14	105.68	28.35	<0.0001
Material	1448.27	4	362.07	97.12	<0.0001
time	7.21	2	3.60	0.97	0.3861
Material *time	24.03	8	3.00	0.81	0.6000
Error	223.67	60	3.73		
Total	1703.18	74			

Table 4: Multiple Comparisons Test.

Material	Means	n	S.E.
seT PP	3.17	15	0.50 A
MaxCem	5.45	15	0.50 B
BisCem	6.36	15	0.50 B
RelyX U100	10.80	15	0.50 C
RelyX ARC	15.52	15	0.50 D

Means with the same letter do not differ significantly(p<= 0.05)
Test:Tukey Alpha=0.05 LSD=1.98263
Error: 3.7279 gl: 60

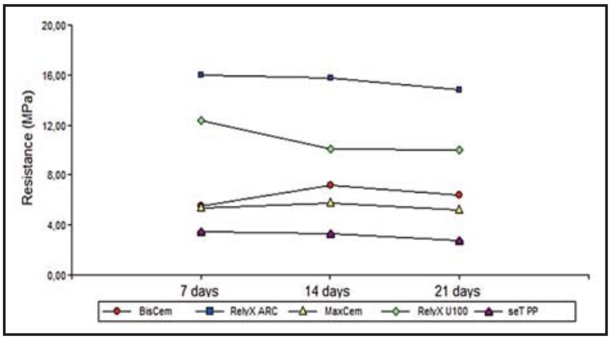

Fig. 8: Shear Bond Strength according to Material and Time.

DISCUSSION

The main advantage of self-adhesive cements is their quick and relatively simple application technique, which is one of the most desirable characteristics in dental materials because it reduces clinical steps, thereby saving operational time[11]. Conventional resin cements requiring multiple steps for application involve a complex, sensitive technique which may affect the bonding efficacy between the restoration and the tooth[13].

This study found significant differences in bond strength to dentin among the different experimental luting agents. Self-adhesive resin cements had significantly lower bond strength values than conventional, multi-step systems represented by the control group (RelyX ARC).

The low bond strength recorded for these cements is probably related to their low capacity for demineralizing and infiltrating the dentin substrate[11]. Despite their low initial pH, their greater viscosity may explain why a true hybrid layer is not formed when they are applied to dentin[14], since the quality of the dentin-cement interface is closely related to the extent of monomer infiltration into the demineralized dentinal collagen[15,16]. This was confirmed in our study by observation under stereomicroscope, which showed that the bond failure mode was adhesive, i.e. between the cement and the dentin surface.

To promote efficient micro-mechanical bonding to dentin collagen fibers, these cements should be capable of conditioning the dental substrate in a relatively short time, which requires optimal wetting properties to ensure rapid interaction with dentin[17,18]. As a general rule, polymer bonding depends on the surface energy and wetting capacity of the adhesive or luting agent to the substrate. Phosphoric acid etching produces an area with greater surface energy because it removes the smear layer and increases surface roughness, providing greater wettability[19,20]. This may explain the higher bonding values of conventional resin cements, represented in this study by RelyX ARC (control group).

Some studies have reported better bonding values for self-conditioning cements with prior treatment of dentin with phosphoric acid. They suggest that the increase in dentin water content after acid etching[21] may help create better ionization of acid monomers in the luting agents, enabling better conditioning of the surface and better polymer bonding[22-25]. Nevertheless, other studies have reported lower bond strength values when acid etching is performed prior to applying self-etch cements[2,9]. The difficulties in standardizing work methodologies, such as thickness of the smear layer, application of weight for cementing or preparation of test specimens make it impossible to compare the results reported by different authors.

The aim of this study was to evaluate four self-etch cements used according to their manufacturer´s instructions, which is what dentists will ultimately do during their work. The results found in this and other studies suggest that dentists who choose these systems because they save time and require simple techniques should take into account their lower bond strengths to dental tissues, and avoid using them for preparations which have little or no retention. The lower bond strength may be partly or wholly attributed to the self-etch cements' ability to interact chemically with the hydroxyapatite in the dentin, rather than to micro-mechanical adhesion, since they only act superficially with dentin[26-28].

Of the experimental materials evaluated, the lowest bond strength to dentin was found for MaxCem and seT PP. According to the manufacturers, the ability to self-etch is due to the presence of different acidic monomers in the formula of the cementing agents such as GPDM un MaxCem, and phosphorylated ester methacrylates in seT PP, whose acidic potential and concentration would be insufficient to promote dentin self-conditioning[29], in comparison to the phosphoric acid esters present in RelyX U 100, which presented higher bond strength. Another factor to consider is that once these cements polymerize, pH should change to a more neutral value. The study published in 2007 by Han[17] showed that 48 hours after polymerization, only RelyX U100 presented a neutral pH value (7), whereas pH for MaxCem was 3.6. A low pH value lasting for some time may have deleterious impact on bonding strength to dentin[17,30]

Finally, the bond to ceramic was not analyzed in the current study, but it is worth noting that bonding effectiveness has been reported by several authors, [5,9,31-36] who recommend treatment of ceramic surfaces by application of hydrofluoric acid and silane to ensure proper cement-ceramic adhesion, regardless of the cement used. In our experience, this is reflected by the fact that bond failures in all cases were between resin cements and dentin, with no instance of cement-ceramic failure.

Based on the results of this study and within the limitations of an *in vitro* study, the following may be concluded:

The self-conditioning cements evaluated showed values for bonding to dentin which are significantly lower than those for the conventional resin cement which represented the control group.

When this type of cement is selected, and considering the low bonding values found in this study, it is important to consider the characteristics of tooth preparation and the mechanical requirements of the restoration in order to prevent potential issues. If adhesive safety is required in procedures with preparations with low retention, resin cements with prior adhesive protocol would be the method of choice.

The authors declare they have no conflict of interest with any of the companies manufacturing the cements used in this study.

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